

Creep behavior and microstructure stability of precipitation-strengthened A2+B2 refractory high entropy alloys

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Kurzfassung

Refraktäre Hochentropielegierungen (RHEAs), die durch geordnete B2-Ausscheidungen verfestigt werden, gelten als vielversprechende Kandidaten für zukünftige Hochtemperatur-Konstruktionswerkstoffe. Dennoch sind ihr Kriechverhalten sowie ihre mikrostrukturelle Stabilität bislang unzureichend untersucht und verstanden. Insbesondere fehlen systematische Untersuchungen, die die Kriechverformung direkt mit der Gefügeentwicklung in ausscheidungsverfestigten A2+B2-RHEAs verknüpfen. Diese Arbeit trägt dazu bei, diese Forschungslücke durch eine umfassende experimentelle Untersuchung der polykristallinen Legierung 27,3Ta-27,3Mo-27,3Ti-8Cr-10Al (at.%) zu schließen.

Trotz ihres polykristallinen Gefüges und ihrer nicht dichtest gepackten Kristallstruktur zeigt die untersuchte Legierung minimale Kriechraten, die im Temperaturbereich oberhalb von 1000 °C und bei Spannungen zwischen 75 und 125 MPa mit denen der modernen einkristallinen Ni-Basis-Superlegierung CMSX-4 vergleichbar sind. Bemerkenswert ist, dass diese Leistungsdaten sehr nahe an der Solvustemperatur der Ausscheidungen ($\approx 0,98 T_{\text{solv}}$) beobachtet werden, im Vergleich zu $\approx 0,85 T_{\text{solv}}$ für CMSX-4. Gegenüber anderen einphasigen A2- und B2-RHEAs wird in der untersuchten Legierung eine um drei bis vier Größenordnungen niedrigere Kriechrate beobachtet.

Detaillierte mikrostrukturelle Analysen nach der Kriechbeanspruchung zeigen erstmals in einer A2+B2-RHEA eine Floßbildung senkrecht zur Belastungsrichtung in Körnern mit $\langle 100 \rangle$ nahe der Druckrichtung. Eine Gefügeabbildung aus unterschiedlichen Richtungen verdeutlicht die tatsächliche räumliche Morphologie der B2-Ausscheidungen, die durch lateral kontinuierliche, jedoch vertikal getrennte, plattenartige Strukturen gekennzeichnet ist. Darüber hinaus werden bei 1030 °C im Spannungsbereich zwischen 100 und 150 MPa folgende grundlegende Kriechprozesse beobachtet: Versetzungsklettern bei niedrigen Spannungen sowie Quergleiten von Schraubenversetzungen bei hohen Spannungen. Im mittleren Spannungsbereich werden lokal kletterkontrollierte Verformung, Schraubenversetzungen mit Sprüngen sowie das Schneiden von B2-Ausscheidungen durch stark gekoppelte Versetzungspaare beobachtet. Unter allen untersuchten Kriechbedingungen bleiben die kohärenten A2/B2-Grenzflächen erhalten.

Abschließend werden die Auswirkungen Re- und Ru-Zusätze auf Phasenstabilität, Umwandlungsverhalten und elementare Partitionierung mit dem Ziel einer Steigerung der Solvustemperatur systematisch untersucht. Beide Elemente erhöhen die Solvustemperatur der Ausscheidungen signifikant und verbessern damit die thermische Stabilität der verfestigenden Phase. Unterschiede im Partitionierungsverhalten von Re und Ru liefern eine mechanistische Grundlage für die Optimierung der Legierungszusammensetzung. Insgesamt etabliert diese Arbeit einen konsistenten experimentellen Rahmen zum Verständnis von Kriechverformung und mikrostruktureller Stabilität in A2+B2-RHEAs und definiert klare thermodynamische sowie mikrostrukturelle Zielgrößen für die Entwicklung zukünftiger kriechbeständiger, ausscheidungsverfestigter, refraktärer Legierungen.

Abstract

Refractory high-entropy alloys (RHEAs) strengthened by B2-ordered precipitates have emerged as promising candidates for next-generation high-temperature structural materials. However, their creep behavior and microstructural stability remain insufficiently investigated and understood. Systematic studies directly linking creep deformation to microstructural evolution in precipitation-strengthened A2+B2 RHEAs are still scarce. The present thesis addresses this gap through the first comprehensive experimental investigation of the polycrystalline alloy 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al (at.%).

Despite its polycrystalline microstructure and non-close-packed crystal structure, the alloy exhibits minimum creep rates comparable to those of the state-of-the-art, single-crystal Ni-based superalloy CMSX-4 in the temperature range beyond 1000 °C and at stresses between 75 and 125 MPa. Notably, this performance is achieved very close to the precipitate solvus temperature T_{solv} ($\approx 0.98 T_{\text{solv}}$), in contrast to $\approx 0.85 T_{\text{solv}}$ for CMSX-4. Compared to single-phase A2 and B2 RHEAs, the creep rate is reduced by three to four orders of magnitude. Detailed post-creep microstructural analyses reveal, for the first time in an A2+B2 RHEA, rafting perpendicular to the loading axis in grains with $\langle 100 \rangle$ orientation close to the compression axis. Multi-side imaging of the raft microstructure reveals laterally continuous, separated plate-like structures oriented perpendicular to the loading axis. Furthermore, the following elementary creep deformation processes are observed at 1030 °C and stresses between 100 and 150 MPa: dislocation climb at low stresses and cross-slip of screw dislocations at high stresses. In the intermediate stress region, climb-controlled deformation, jogged screw dislocations, and the cutting of B2 precipitates by strongly coupled dislocation pairs are locally observed. Fully coherent A2/B2 interfaces are retained under all investigated creep conditions.

Finally, the effects of Re and Ru additions on phase stability, transformation behavior, and elemental partitioning are systematically examined with respect to their effects on solvus temperature. Both elements significantly increase the precipitate solvus temperature, thereby extending the thermal stability of the strengthening phase. Distinct partitioning characteristics of Re and Ru are identified, providing a mechanistic basis for optimizing alloy composition. Overall, this work establishes a coherent experimental framework for understanding creep deformation and microstructural stability in A2+B2 RHEAs and defines clear thermodynamic and microstructural targets for the development of creep-resistant, precipitation-strengthened, refractory alloys.

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Nomenclature

Abbreviations

Abbreviation	Definition
APB	Antiphase Boundary
APT	Atom Probe Tomography
BCC	Body-Centered Cubic
BF	Bright Field
BN	Boron Nitride
CALPHAD	Calculation of Phase Diagrams
CGHE	Carrier Gas Hot Extraction
DSC	Differential Scanning Calorimetry
DF	Dark Field
EDS	Energy Dispersive X-ray Spectroscopy
EDM	Electrical Discharge Machining
FCC	Face-Centered Cubic
FFT	Fast Fourier Transform
FIB	Focused Ion Beam
HAGB	High-Angle Grain Boundary
HCR	High Creep Resistance
HEA	High Entropy Alloy
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
ICD	In-Column Detector
IFFT	Inverse Fast Fourier Transform
IPF	Inverse Pole Figure
IQ	Image Quality
KAM	Kernel Average Misorientation
LAGB	Low-Angle Grain Boundary

LBR	Lagneborg-Bergman-Reppich
LCR	Low Creep Resistance
LSW	Lifshitz-Slyozov-Wagner
PX	Polycrystalline
PLB	Power-Law Breakdown
RHEAs	Refractory High-Entropy Alloys
RT	Room Temperature
SEM	Scanning Electron Microscopy
SEM-BSE	Scanning Electron Microscopy using Backscattered Electrons
SEM-EBSD	Scanning Electron Microscopy with Electron Backscatter Diffraction
STEM	Scanning Transmission Electron Microscopy
STEM-HAADF	Scanning Transmission Electron Microscopy using High-Angle Annular Dark-Field imaging
SGB	Subgrain Boundary
SX	Single Crystal
TEM	Transmission Electron Microscopy
TEM-BF	Bright-Field Transmission Electron Microscopy
TEM-DF	Dark-Field Transmission Electron Microscopy
TEM-SAED	Selected Area Electron Diffraction in Transmission Electron Microscopy
XRD	X-ray Diffraction
ZA	Zone Axis

Symbols

Creep and Thermodynamic Variables

Symbol	Definition	Unit
T	Absolute temperature	K or °C
T_c	Critical temperature	K or °C
T_m	Melting temperature	K or °C
T_{solv}	Solvus temperature	K or °C
T_{ord}	Ordering temperature	K or °C
T_{solidus}	Solidus temperature	K or °C
ΔG_{mix}	Gibbs free energy of mixing	kJ/mol
ΔH_{mix}	Enthalpy of mixing	kJ/mol
ΔS_{mix}	Entropy of mixing	kJ/(mol·K)
S_{conf}	Configurational entropy	kJ/(mol·K)
R	Gas constant	kJ/(mol·K)
x_i	Atomic fraction of element i	–
i	Element index	–
ε	Creep strain	– or %
$\dot{\varepsilon}$	Creep rate	s ⁻¹
$\dot{\varepsilon}_{\text{min}}$	Minimum creep rate	s ⁻¹
$\dot{\varepsilon}_{\text{steady}}$	Steady-state creep rate	s ⁻¹
σ	Applied stress	MPa
σ_{eff}	Effective stress	MPa
σ_p	Threshold stress	MPa
σ_{A2}	Stress contribution associated with the A2 matrix	MPa
σ_{B2}	Stress contribution associated with B2 precipitates	MPa
σ_t	True stress	MPa
σ_e	Engineering stress	MPa

ε_e	Engineering strain	– or %
n	Stress exponent	–
n_{app}	Apparent stress exponent	–
n_m	Matrix-controlled stress exponent	–
n^*	Reference stress exponent in the sinh formulation	–
Q	Activation energy	kJ/mol
Q_{app}	Apparent activation energy	kJ/mol
Q_{A2}	Activation energy of the A2 matrix	kJ/mol
A	Material constant in creep law	–
A_1	Material constant in the effective-stress formulation	–
B	Material constant in the sinh-type formulation	–
V	Activation volume	m ³
k	Boltzmann constant	J/K
D_0	Diffusion pre-exponential factor	m ² /s
d	Grain size	μm
p	Grain-size exponent	–
G	Shear modulus	GPa
b	Burgers vector	nm
τ_{Orowan}	Orowan shear stress	MPa
λ	Interparticle spacing / channel width	nm
γ_{APB}	Antiphase boundary energy	J/m ²
Φ	Mechanism-dependent strengthening function	–

Microstructural and Experimental Variables

Symbol	Definition	Unit
r	Precipitate radius	nm
r_0	Initial precipitate radius	nm
a_p	Major axis of a precipitate	nm
b_p	Minor axis of a precipitate	nm
$\bar{a}_p$	Mean major axis of precipitates	nm
$\bar{b}_p$	Mean minor axis of precipitates	nm
r_{eq}	Equivalent particle radius	nm
$\bar{r}_{eq}$	Mean equivalent particle radius	nm
f	Precipitate volume fraction	– or %
κ	Coarsening-rate constant	nm ³ /h
N_A	Number density of precipitates	μm ⁻²
$N_{\text{particles}}$	Number of particles identified in an image	–
A_{image}	Analyzed image area	μm ²
C	Circularity	–
V_m^{B2}	Volume fraction of the B2 phase	%
l	Characteristic precipitate size used in the Thornton–Thompson parameter	nm
L	Thornton–Thompson parameter	–
γ	Interfacial energy	J/m ²
C_{44}	Elastic stiffness coefficient	GPa
δ	Lattice misfit	%
a	Lattice parameter (general)	nm
a_{A2}	Lattice parameter of the A2 matrix	nm
a_{B2}	Lattice parameter of the B2 precipitates	nm
a_{matrix}	Lattice parameter of the matrix phase	nm

$a_{\text{precipitate}}$	Lattice parameter of the precipitate phase	nm
d_{hkl}	Interplanar spacing of the (hkl) planes	nm
θ	Bragg angle	°
λ	X-ray wavelength	nm
g	Diffraction vector	–
ρ	Density	g/cm ³
ρ_{water}	Density of water	g/cm ³
F_{air}	Measured force in air	N
F_{water}	Measured force in water	N
C_i^{A2}	Atomic fraction of element i in the A2 matrix	–
C_i^{B2}	Atomic fraction of element i in the B2 precipitates	–
k_i	Partition coefficient	–
ΔC_i	Concentration-difference parameter	–
D_i^{A2}	Diffusion coefficient of the rate-controlling species in the A2 matrix	m ² /s
dH/dT	Temperature derivative of enthalpy in DSC analysis	J/(K·mol)

1 Introduction

Demands for higher thermal efficiency and lower emissions in modern gas turbine engines continue to drive the development of structural materials capable of sustaining extreme thermal and mechanical loads [1]. Further increases in turbine inlet temperature require materials that retain high creep resistance and microstructural stability during prolonged exposure. At present, single-crystal Ni-based superalloys represent the benchmark owing to their unique two-phase microstructure consisting of a disordered face-centered cubic matrix (FCC, Strukturbericht designation A1, denoted γ) and coherent, thermally stable, ordered precipitates with an $L1_2$ structure (Strukturbericht designation $L1_2$, denoted γ'), which provide effective and sustained precipitation strengthening [2–6]. However, the maximum service temperature of Ni-based superalloys is ultimately limited to around 1050 °C by the γ' solvus temperature and the alloy solidus temperature. Near the solvus temperature, the γ' volume fraction rapidly decreases and substantial coarsening severely degrades creep resistance. This limitation motivates the search for alternative alloy systems with higher solvus and solidus temperatures.

Refractory high-entropy alloys (RHEAs) are promising candidates for next-generation, high-temperature structural materials because of their high solidus temperatures and retained mechanical strength at elevated temperatures, arising from compositions based on refractory elements such as Nb, Mo, Ta, and W [7,8]. Early HEAs research focused on the concept of entropy-stabilized single-phase solid solutions [9,10]. Guided by this concept, the equiatomic $\text{Nb}_{25}\text{Mo}_{25}\text{Ta}_{25}\text{W}_{25}$ alloy forms a single-phase BCC solid solution and retains a compressive strength of ~405 MPa at 1600 °C, underscoring the remarkable high-temperature performance [11]. However, subsequent experimental studies have shown that configurational entropy alone is often insufficient to suppress phase separation or intermetallic formation in RHEAs [12–14]. As a result, current alloy development increasingly emphasizes the intentional design of beneficial multiphase microstructures rather than the exclusive pursuit of single-phase solid solutions.

This shift is particularly important for creep-resistant RHEAs. Most available high-temperature creep studies concern single-phase A2 alloys, whose creep resistance in the temperature range of 980–1100 °C remains substantially below that of state-of-the-art Ni-based superalloy CMSX-4 [15,16]. Their limited performance has been associated with the absence of precipitation strengthening, the polycrystalline condition, relatively fast diffusion in the non-close-packed A2 crystal structure, and microstructural instability during prolonged exposure [16]. These observations indicate that coherent and thermally stable precipitates

are likely essential for achieving high creep resistance in refractory alloys, analogous to the role of γ' in Ni-based superalloys.

Among the proposed multiphase RHEA concepts, many systems still suffer from fundamental microstructural limitations. In several reported alloys, the ordered B2 phase forms the matrix or dominates the phase fraction [17–20], which fundamentally limits ductility and reduces the effectiveness of precipitation strengthening. In other cases, the multiphase microstructure develops by spinodal decomposition, producing diffuse interfaces and less controllable characteristic length scales [21,22]. Such microstructures do not provide an ideal basis for sustained precipitation strengthening under creep conditions.

An alternative and more promising design strategy is based on the formation of coherent and ordered B2 precipitates within a disordered A2 matrix through classical nucleation and growth [16,23,24]. This two-phase architecture is conceptually analogous to the $\gamma + \gamma'$ microstructure of Ni-based superalloys and offers the possibility of combining a strong refractory matrix with coherent ordered precipitates. In addition, nucleation-controlled precipitation provides direct access to key strengthening parameters such as precipitate size, spacing, and coherency.

A representative alloy of this design strategy is 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al (at.%), hereafter referred to as TMT-8Cr-10Al, developed by Laube et al. [13,25]. After homogenization and furnace cooling, this alloy forms discrete, coherent, ordered B2 precipitates (approximately 10–30 nm in diameter) within a disordered A2 matrix through diffusion-controlled nucleation and growth. Compared with the much coarser γ' precipitates (about 500 nm) in CMSX-4 [13,25,26], the B2 precipitates in TMT-8Cr-10Al are significantly finer, as indicated by the different scale bars in Figure 1a and Figure 1b. Combined with a comparatively low coarsening rate, this nanoscale distribution provides pronounced resistance to microstructural degradation during prolonged exposure [25,27]. This alloy exhibits a B2 solvus temperature of approximately 1050 °C based on DSC measurements [13]. After long-term annealing at 1000 °C for 1000 h, a stable two-phase microstructure is preserved, with precipitates remaining in the nanometer size range [25], indicating high thermal stability compared with many reported RHEAs [13,24,28,29]. TMT-8Cr-10Al therefore represents a particularly instructive model system for investigating creep behavior and microstructural evolution in precipitation-strengthened A2+B2 RHEAs.

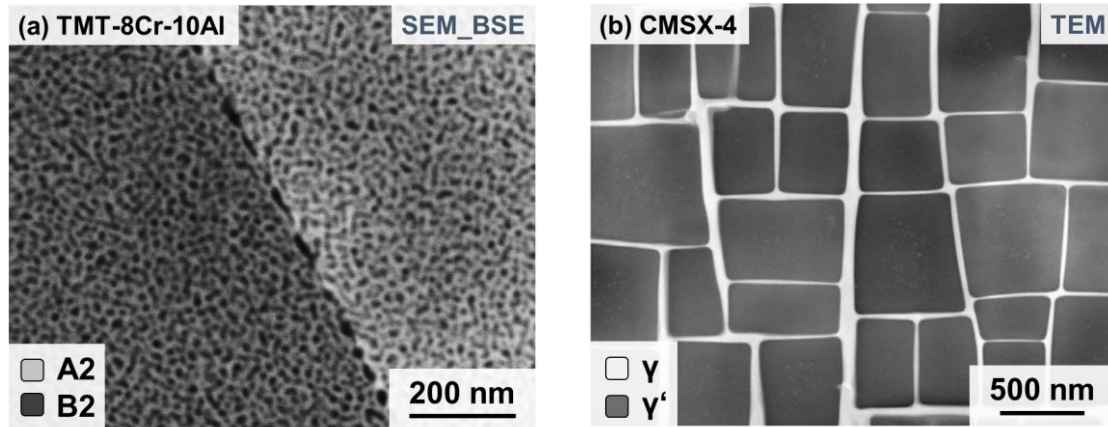


Figure 1. Comparison of two precipitation-strengthened microstructures. (a) SEM-BSE micrograph of the homogenized 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al, showing a disordered A2 matrix with ordered B2 precipitates. (b) TEM micrograph of the homogenized $\gamma+\gamma'$ CMSX-4 Ni-based superalloy, showing a disordered γ matrix with ordered γ' precipitates [26].

Despite this promising microstructural concept, the creep behavior of two-phase A2+B2 RHEAs has not yet been systematically investigated. Important open questions remain concerning their creep response near the precipitate solvus temperature, the associated microstructural evolution during creep, and the quantitative contribution of precipitation strengthening. Addressing these questions constitutes the first major objective of this thesis. To support a quantitative interpretation of the creep data, the Lagneborg-Bergman-Reppich (LBR) approach, originally developed for Ni- and Co-based superalloys [30–32], is applied here for the first time to an A2+B2 RHEA. This provides a framework for separating matrix and precipitate contributions to the overall creep resistance and enables a direct comparison with conventional precipitation-strengthened superalloys.

A second objective of this work is to improve the high-temperature phase stability of the A2+B2 microstructure. Although TMT-8Cr-10Al remains stable close to its solvus temperature, further increases in operating temperature require both a higher solvus temperature and slower precipitate coarsening. Recent studies indicate that Ru and Re act as strong B2 stabilizers in refractory binary systems, particularly when combined with elements from groups IV and V, as suggested by computational predictions [33,34]. Experimental results further confirm that Ru-stabilized B2 precipitates remain stable up to 1700 °C, whereas Re-stabilized B2 precipitates are stable up to 1550 °C [34–36]. Their effect on the phase stability and precipitate evolution of the TMT-8Cr-10Al alloy system is therefore investigated here with the aim of identifying alloying strategies for improved high-temperature stability.

To address these challenges, this thesis specifically focuses on the following research questions:

A. Creep behavior and microstructural evolution

1. How does the creep behavior of a precipitation-strengthened A2+B2 RHEA compare with that of state-of-the-art, single-crystal Ni-based superalloys and polycrystalline single-phase RHEAs?
2. How does the A2+B2 microstructure evolve during creep deformation, and which deformation processes are dominant under different stress conditions?

B. Quantification of precipitation strengthening

3. What is the quantitative contribution of precipitates to the overall creep resistance, as determined using the LBR approach, and how does it compare with that in other precipitation-strengthened alloys?

C. Alloying strategies for enhanced solvus temperature and phase stability

4. How do Ru and Re additions affect the solvus temperature and precipitate coarsening behavior?
5. How do Ru and Re partition between the A2 matrix and B2 precipitates, and how does this partitioning influence high-temperature microstructural stability?

By addressing these questions, this work aims to (i) establish a fundamental understanding of creep behavior in precipitation-strengthened A2+B2 RHEAs, (ii) provide a quantitative evaluation of precipitation strengthening in this alloy system, and (iii) identify alloying strategies that support the design of next-generation high-temperature materials.

2 Fundamentals

2.1 Refractory High Entropy Alloys (RHEAs)

2.1.1 Concept and Design Principles

The concept of high-entropy alloys (HEAs), introduced by Yeh et al. [9] and Cantor et al. [37] in 2004, proposes that random atomic mixing among multiple principal elements increases configurational entropy and thereby stabilizes single-phase solid solutions. Although this idea initiated a paradigm shift away from traditional alloy design based on single base elements, subsequent thermodynamic analyses and experimental studies have shown that configurational entropy rarely dominates phase stability on its own [38]. Instead, the phase constitution of HEAs generally results from the competition between entropy and enthalpy contributions to the Gibbs free energy of mixing, given by:

$$\Delta G_{\text{mix}} = \Delta H_{\text{mix}} - T \Delta S_{\text{mix}} \quad (2.1)$$

where ΔG_{mix} is the Gibbs free energy of mixing, ΔH_{mix} is the enthalpy of mixing, T is the absolute temperature, and ΔS_{mix} is the entropy of mixing.

For an ideal random solid solution, the entropy of mixing corresponds to the configurational entropy, S_{conf} , which can be expressed as:

$$S_{\text{conf}} = -R \sum_i x_i \ln x_i \quad (2.2)$$

where R is the gas constant and x_i is the atomic fraction of element i .

Although S_{conf} increases with the number of constituent elements, the enthalpy contribution, ΔH_{mix} , often plays a dominant role in determining the phase constitution. Strong enthalpy contributions can promote chemical ordering or phase separation, particularly when significant atomic size or chemical mismatches exist among constituent elements [38,39]. Consequently, modern HEA design has moved beyond the original entropy-centered concept towards a broader microstructure-based approach that places particular emphasis on phase stability, ordering tendencies, and high-temperature structural performance.

Among various HEA families, refractory high-entropy alloys (RHEAs), typically composed of high-melting-point transition metals such as Nb, Mo, Ta, W, and V, have emerged as promising candidates for high-temperature applications due to their high solidus temperatures, slow diffusion kinetics, and substantial solid-solution strengthening up to 1600 °C [8,11,21,22]. However, refractory elements generally form volatile or non-protective oxides at high

temperatures, which limits their environmental stability [40]. To address this issue, passivating elements such as Cr and Al are commonly added to promote the formation of protective scales and thereby enhance oxidation resistance [41,42]. In addition to its environmental benefit, Al plays an important chemical role in RHEAs. Its large negative ΔH_{mix} with refractory transition metals promotes chemical ordering and can drive the formation of an ordered B2 phase [13,43,44]. Depending on the Al content and overall chemistry, this ordering tendency can produce either an ordered B2 matrix containing a disordered A2 phase or an A2 matrix strengthened by an ordered B2 phase [12,13,25]. In particular cases [13,25,43,45–48], the B2 phase may form as coherent precipitates embedded in the A2 matrix.

This A2+B2 two-phase microstructural configuration closely parallels the well-established $\gamma + \gamma'$ architecture of Ni-based superalloys. The 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al (at.%), referred to as TMT-8Cr-10Al in this study, belongs to this class of A2+B2 RHEAs. This microstructural architecture forms the basis for the creep behavior and microstructural evolution discussed in the following chapters.

2.1.2 A2+B2 Microstructure in RHEAs

(1) A2 and B2 Crystal Structures

The phase constitution of RHEAs is predominantly based on the disordered body-centered cubic (BCC) A2 crystal structure and one of its ordered derivatives, the B2 structure. Depending on alloy composition, additional ordered variants, such as L2₁, or more complex intermetallic phases may also occur. In the present work, the focus is placed on A2+B2 two-phase microstructures.

The crystallographic relationship between A2 and B2 structures is schematically illustrated in Figure 2. The A2 phase exhibits a BCC Bravais lattice with space group $\text{Im}\bar{3}\text{m}$, in which the constituent elements statistically occupy the equivalent 2a Wyckoff position (Figure 2a). In contrast, the B2 phase adopts the ordered CsCl-type structure with space group $\text{Pm}\bar{3}\text{m}$. Although the B2 structure is frequently described as BCC because of its close structural relationship to the parent BCC framework, it is strictly a primitive cubic Bravais lattice with a two-atom basis occupying the 1a and 1b Wyckoff positions (Figure 2b). The difference between A2 and B2 can therefore be understood as chemical ordering on a BCC-derived framework. In the disordered A2 phase, the corner and body-centered sites are statistically equivalent, whereas in the ordered B2 phase, different atomic species preferentially occupy the 1a and 1b sublattices, resulting in long-range chemical order.

At the microstructural level, Al-containing RHEAs typically exhibit a characteristic compositional partitioning between A2 and B2 phases. As discussed in Section 2.1.1, this behavior is driven by strong negative mixing enthalpies between Al and refractory transition

metals, which promotes chemical ordering and stabilizes the formation of A2+B2 two-phase microstructures [13,24,28,49,50].

The TMT-8Cr-10Al alloy investigated in this study follows this characteristic partitioning behavior. Previous studies have shown that it forms an A2 matrix, primarily enriched in Ta and Mo, together with coherent B2 precipitates enriched in Al and Ti, while Cr remains uniformly distributed between the two phases [13,25]. Owing to their nearly identical lattice parameters and close crystallographic relationship, A2 and B2 phases can form coherent interfaces with low interfacial energy, which contributes to the long-term thermal stability of the A2+B2 two-phase microstructure at high temperatures [25].

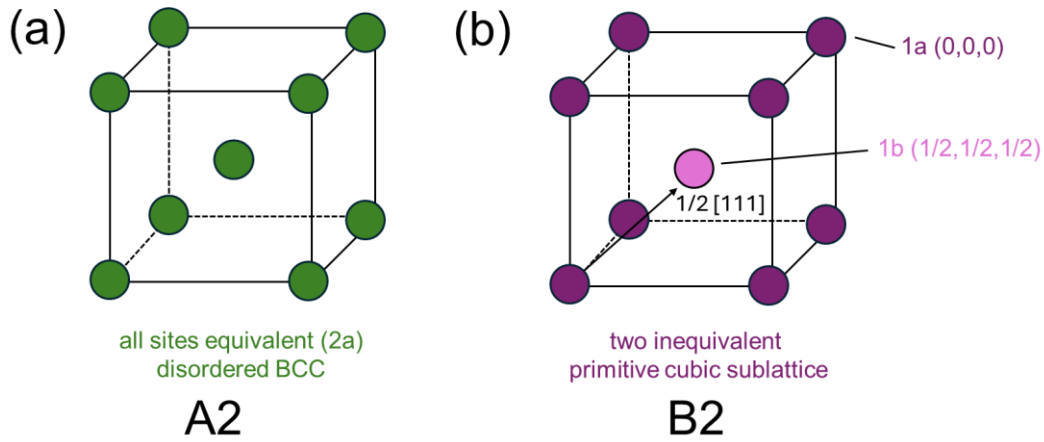


Figure 2. Schematic illustration of the crystal structures of (a) the disordered A2 phase and (b) the ordered B2 phase. In the A2 structure, all constituent atoms occupy the equivalent 2a Wyckoff positions of the BCC lattice. In the B2 structure, chemical ordering produces two inequivalent primitive cubic sublattices: the 1a sublattice at (0,0,0) and the 1b sublattice at (1/2,1/2,1/2). The 1b sublattice is displaced by $1/2[111]$ relative to the 1a sublattice, as indicated by the arrow in (b).

(2) Deformation relevance of chemical ordering in A2+B2 microstructure

The different degrees of chemical ordering in A2 and B2 phases have direct implications for dislocation motion. In the disordered A2 phase, plastic deformation is generally associated with $1/2\langle 111 \rangle$ dislocations gliding on multiple possible BCC slip planes (e.g., {110}, {112}, {123}) [51,52]. These slip directions correspond to the shortest lattice translations in the BCC lattice and therefore minimize the Burgers vector length and the associated dislocation line energy. As generally established for BCC metals and alloys, screw dislocations are particularly important for plastic deformation. Their non-planar core structure gives rise to a high Peierls barrier. This barrier represents the lattice-friction resistance against dislocation glide [53,54]. At low and intermediate temperatures, screw dislocations therefore do not move easily as

straight dislocation lines. Instead, their motion proceeds by thermally activated kink-pair nucleation and propagation, where local segments of the screw dislocation first overcome the Peierls barrier and the resulting kinks subsequently migrate along the dislocation line. This behavior accounts for the pronounced temperature and strain-rate dependence of plastic flow in BCC metals and alloys.

In the ordered B2 phase, dislocation motion is additionally constrained by long-range chemical order. Since the 1a and 1b sublattices are inequivalent, only Burgers vectors corresponding to lattice translations of the ordered B2 structure preserve the sublattice occupation. Burgers vectors such as $\langle 111 \rangle$ or $\langle 100 \rangle$ therefore maintain the ordered arrangement. By contrast, a $1/2\langle 111 \rangle$ displacement exchanges the two sublattices, generates an antiphase boundary (APB), and introduces an additional energetic penalty proportional to the antiphase boundary energy (γ_{APB}) [55–58]. To reduce this energy penalty, plastic deformation in many B2 phases often proceeds via superdislocations consisting of two coupled $1/2\langle 111 \rangle$ partials separated by an APB ribbon. The leading partial creates the APB, whereas the trailing partial restores the ordered arrangement. The value of γ_{APB} affects the separation and coupled motion of these partials. A higher γ_{APB} increases the energetic penalty for independent motion of the leading partial, reduces the allowable separation between the partials, and promotes strongly coupled motion. The mobility of such superdislocations also depends on temperature, alloy composition, and defect structure. Owing to the additional constraints imposed by long-range order, B2 phases can exhibit higher resistance to dislocation motion than their disordered A2 counterparts. However, this should not be generalized as an intrinsic lack of ductility, since the mechanical response of monolithic B2 compounds varies substantially with composition, defect structure, and active slip modes [59,60]. For instance, B2 alloys such as YCu have been reported to remain ductile even at cryogenic temperatures [61].

These elementary glide behaviors provide the crystallographic basis for understanding deformation in A2 and B2 phases. Under high-temperature creep conditions, they must be considered together with diffusion-assisted processes such as dislocation climb, recovery, jog motion of screw dislocations, and climb-assisted bypass of precipitates. More generally, studies on coherent precipitate-strengthened systems, particularly $\gamma+\gamma'$ Ni-based superalloys, have shown that the mechanical response cannot be inferred from the intrinsic strength of the individual phases alone. Coherency, lattice misfit, precipitate morphology, and interfacial stress fields also strongly affect dislocation-precipitate interactions [62–64]. In the present A2+B2 microstructure, these considerations provide a useful framework, while the specific deformation response is additionally governed by B2 chemical ordering, APB energy, precipitate size and spacing, and the temperature-dependent mobility of dislocations. This framework is used in the later discussion of dislocation-precipitate interactions and high-temperature creep behavior.

2.1.3 Thermodynamics of the A2+B2 Microstructure

(1) Thermodynamic Basis of Chemical Decomposition

The formation of an A2+B2 microstructure is governed not only by crystallographic ordering, but also by the thermodynamic tendency of a supersaturated solid solution to decompose into compositionally distinct regions. This is important for the present work because the decomposition pathway influences the morphology, interface character, precipitate spacing, and thermal stability of the precipitate phase. These microstructural features later affect coarsening, rafting, and dislocation-precipitate interactions during high-temperature creep.

Figure 3 schematically illustrates the thermodynamic basis for chemical decomposition within a miscibility gap. In this simplified representation, a homogeneous A2 solid solution separates into two compositionally distinct disordered regions of the same structural type, denoted as A2' and A2'', i.e., $A2 \rightarrow A2' + A2''$. This decomposition is driven by a reduction in Gibbs free energy. As shown in Figure 3, above the critical temperature T_c , the free energy curve exhibits a single minimum, corresponding to a stable homogeneous solid solution. Below T_c of the miscibility gap, the free energy curve develops a double-well shape, indicating that the homogeneous A2 solid solution becomes thermodynamically unstable or metastable with respect to compositional fluctuations. Under these conditions, the system separates into two phases with distinct compositions corresponding to the minima of the free energy curve. The extent of this driving force increases with decreasing temperature within the miscibility gap. Depending on the alloy composition and its position within the miscibility gap, this decomposition may proceed via different pathways, which are discussed in the following subsection.

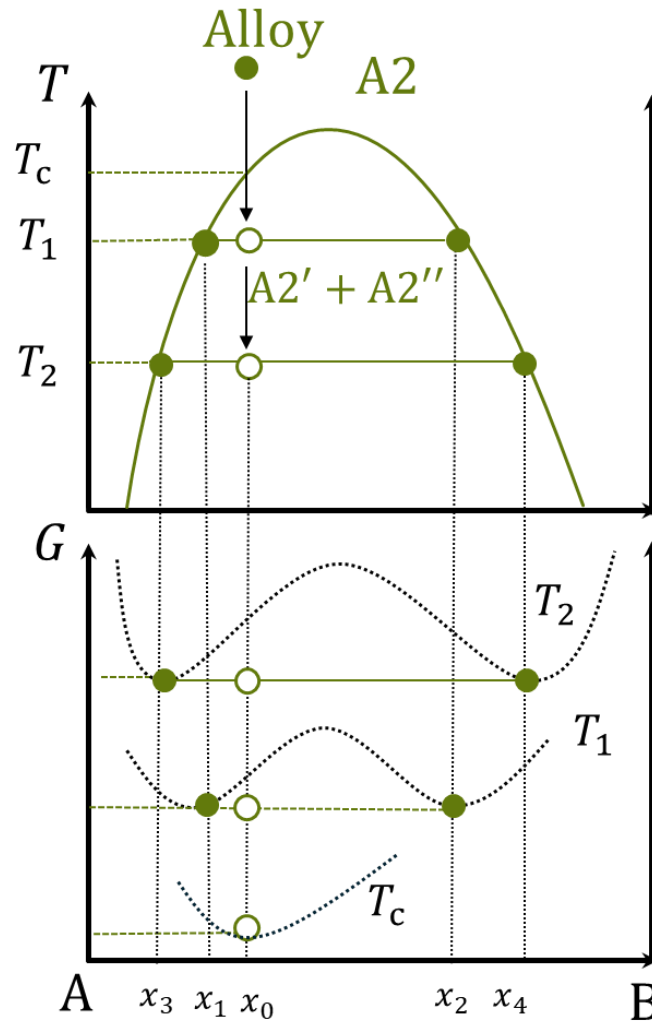


Figure 3. Schematic representation of chemical decomposition within a miscibility gap, corresponding to the decomposition $A2 \rightarrow A2' + A2''$. The upper panel shows a simplified temperature-composition diagram with a critical temperature T_c . The lower panel shows the corresponding Gibbs free energy curves at different temperatures. The development of a double-well shape below T_c provides the thermodynamic driving force for phase separation.

(2) Phase Separation Pathways within the Miscibility Gap

The pathway by which a supersaturated A2 solid solution decomposes is important because it determines whether the resulting microstructure develops through continuous composition modulation or through discrete precipitate formation. These two cases lead to different interface sharpness, precipitate morphology, and coarsening behavior, all of which are relevant for the stability of A2+B2 microstructures at high temperatures.

Depending on the position of the alloy composition within the miscibility gap, phase separation may proceed either by spinodal decomposition or by classical nucleation and growth, as illustrated schematically in Figure 4. The binodal boundary defines the limit of the two-phase region, whereas the spinodal boundary lies inside the miscibility gap and separates the metastable region from the unstable region.

When quenched into the spinodal region (Case I), the homogeneous solid solution is thermodynamically unstable. In this case, phase separation proceeds via spinodal decomposition, which involves the continuous amplification of compositional fluctuations without the need for nucleation [65]. This process results in an interconnected microstructure of A_2' and A_2'' regions separated initially by diffuse interfaces.

In contrast, quenching into the metastable region between the binodal and spinodal boundaries (Case II), phase separation requires overcoming a nucleation barrier and therefore proceeds by classical nucleation and growth. The formation of the minority phase occurs as a precipitation process. For example, discrete A_2'' nuclei form within the supersaturated A_2 matrix and subsequently grow by diffusion, leading to isolated precipitates embedded in an A_2' matrix. These precipitates are typically associated with comparatively sharp interfaces.

The distinction between these two decomposition pathways is essential, as the resulting interface character and morphology strongly influence coarsening kinetics, interfacial stability, and the high-temperature microstructural evolution. At this stage, both pathways describe chemical partitioning only and do not imply long-range ordering into the B2 structure, which is addressed in the following subsection.

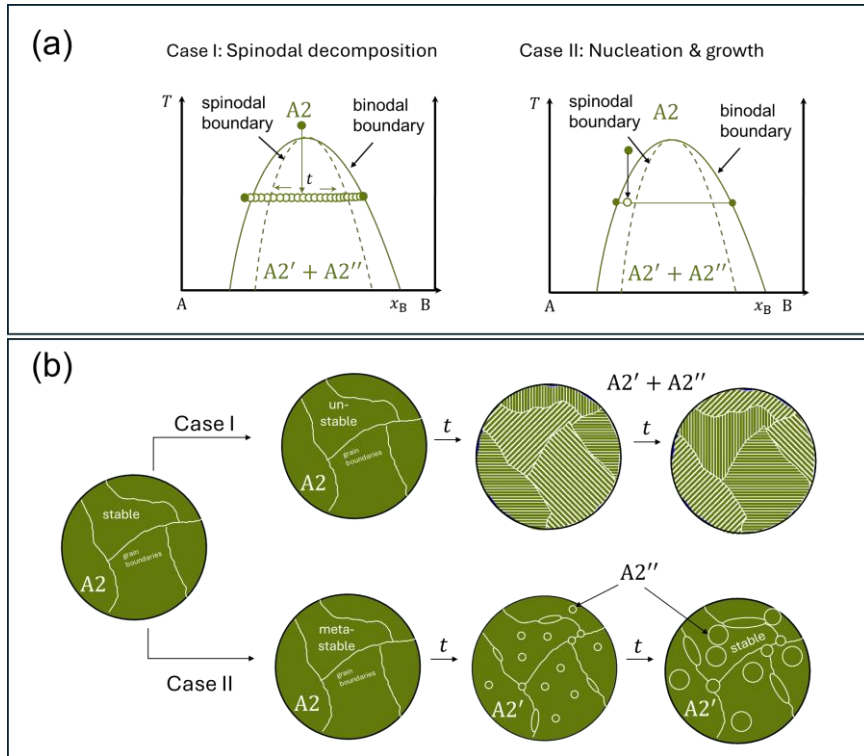


Figure 4. Schematic illustration of phase separation pathways in a supersaturated $A2$ solid solution within a miscibility gap. (a) Phase diagrams showing binodal (solid line) and spinodal (dashed line) boundaries. Decomposition inside the spinodal region (Case I) occurs by spinodal decomposition from an unstable $A2$ solid solution, whereas decomposition between the binodal and spinodal boundaries (Case II) proceeds by classical nucleation and growth from a metastable $A2$ solid solution. (b) Corresponding microstructural evolution. Spinodal decomposition (Case I) leads to an interconnected $A2' + A2''$ structure with diffuse interfaces, while nucleation and growth (Case II) results in discrete $A2''$ precipitates in an $A2'$ matrix with sharp interfaces.

(3) Ordering Pathways Toward the $A2+B2$ Microstructure

Chemical decomposition alone produces compositionally distinct $A2'$ and $A2''$ regions, but not necessarily an ordered $B2$ phase. The $A2+B2$ microstructure forms only when chemical partitioning is accompanied by long-range ordering of one decomposition product into the $B2$ structure. Thus, the development of an $A2+B2$ microstructure can be considered as a coupled process involving compositional decomposition and chemical ordering.

In systems where phase separation proceeds by nucleation and growth, ordering may occur either sequentially or concurrently with chemical partitioning, as illustrated schematically in Figure 5. In the first scenario (Case I), the supersaturated $A2$ solid solution first decomposes into two disordered phases, $A2 \rightarrow A2' + A2''$, followed by ordering of the precipitate-forming

$A2''$ phase into B2, $A2'' \rightarrow B2$, resulting in a final $A2' + B2$ microstructure. In the second scenario (Case II), compositional decomposition and ordering occur nearly concurrently, $A2 \rightarrow A2' + B2$, such that the ordered precipitates form directly during decomposition. The relative importance of these two pathways depends on the thermodynamic driving forces for decomposition and ordering, atomic mobility, alloy composition, and heat-treatment temperature. In both cases, the final $A2+B2$ microstructure is controlled by the coupling between chemical partitioning and long-range ordering.

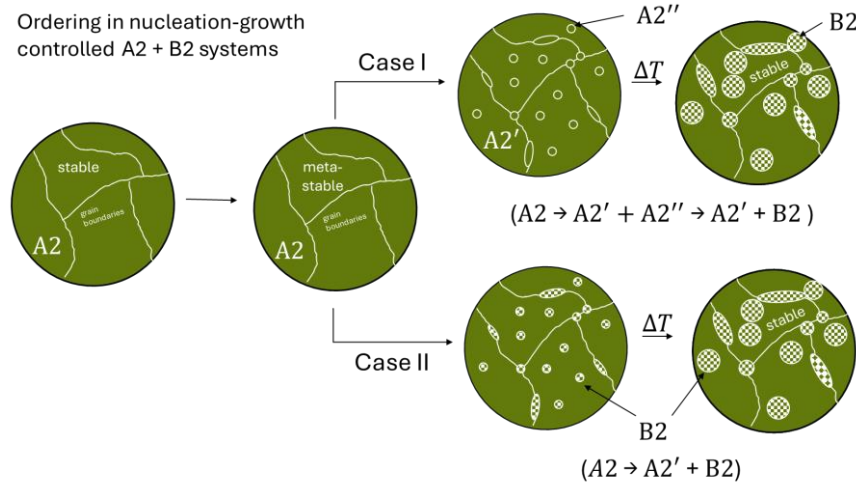


Figure 5. Schematic representation of two possible ordering pathways in nucleation-controlled $A2+B2$ systems. Case I: chemical phase separation first produces two disordered regions, $A2 \rightarrow A2' + A2''$, followed by ordering of the precipitate-forming region, $A2'' \rightarrow B2$, resulting in an $A2'+B2$ microstructure. Case II: compositional decomposition and B2 ordering occur concurrently, leading directly to $A2 \rightarrow A2' + B2$.

(4) Precipitate Solvus and Ordering Stability

For precipitation-strengthened $A2+B2$ alloys, the precipitate solvus temperature T_{solv} associated with chemical phase separation should be distinguished from the ordering temperature T_{ord} . The precipitate solvus temperature defines the upper temperature limit of the compositionally decomposed two-phase field. Above T_{solv} , the compositionally distinct precipitate phase dissolves into the matrix. Below T_{solv} , a compositionally distinct precipitate phase can persist, but it exhibits ordered B2 only if the temperature is also below T_{ord} .

This distinction is important because the strengthening effect of ordered precipitates depends on both precipitate stability and retention of long-range chemical order. More generally, the relative magnitudes of T_{solv} and T_{ord} define the temperature window over which both precipitate stability and order-related strengthening can be retained. This concept is particularly important for alloy design: raising T_{solv} alone is insufficient if T_{ord} remains lower

than the intended operating temperature. An optimized A2+B2 alloy should retain both a stable precipitate population and long-range B2 order under service conditions. Therefore, the solvus-ordering relationship provides a thermodynamic basis for interpreting the phase stability, microstructural evolution, and high-temperature deformation behavior discussed in the following chapters.

2.2 Creep and Microstructure Evolution in Superalloys

To interpret the creep behavior of the present A2+B2 RHEAs, a clear theoretical framework for high-temperature deformation and microstructural evolution is required. As introduced in Section 2.1, the mechanical response of these alloys is governed by coupled interactions between dislocations, the disordered A2 matrix, ordered B2 precipitates, and coherent A2/B2 interfaces. Although creep involves a variety of thermally activated processes, its macroscopic behavior is primarily controlled by dislocation motion, diffusion, and the evolution of the precipitate microstructure [66–69].

Ni-based superalloys provide the most established reference system for analyzing such behavior. In these alloy systems, creep deformation is strongly influenced by coherent precipitates, interfacial energy, precipitate coarsening, rafting, and dislocation-precipitate interactions. Although the crystal structure differs from that of A2+B2 RHEAs, the underlying concepts, such as effective stress, precipitate resistance, dislocation cutting, bypass, and microstructural stability under load, are directly relevant. The following subsections therefore introduce the essential principles relevant to precipitation-strengthened alloys, beginning with the empirical description of creep and progressing to the concepts of effective stress, dislocation-precipitate interaction, and precipitate evolution under load.

2.2.1 Classical Creep Law

Creep deformation is the time-dependent plastic strain that develops in materials subjected to a sustained mechanical load at elevated homologous temperatures, typically at $T > 0.4\text{--}0.5 T_m$, where T_m is the absolute melting temperature [70–72]. A typical creep curve, schematically shown in Figure 6a, is commonly divided into primary, secondary, and tertiary creep regimes. These regimes reflect the changing balance between strain hardening, dynamic recovery, microstructural evolution, and damage accumulation. During primary creep, the creep rate decreases because dislocation multiplication and dislocation interactions increase the resistance to further plastic flow. During secondary creep, the strain rate may become approximately constant when hardening and recovery processes are balanced. During tertiary creep, the strain rate accelerates due to damage accumulation, necking, cavitation, grain-boundary degradation, or microstructural instability, eventually leading to failure.

The minimum creep rate $\dot{\epsilon}_{\min}$ is the lowest measured strain rate, commonly determined during the secondary creep regime, which typically covers most of the deformation (and time) prior to rupture. It provides the most reproducible parameter for quantifying creep resistance and therefore forms the basis for quantitative analysis of high-temperature deformation [71]. However, $\dot{\epsilon}_{\min}$ must be distinguished from a steady-state creep rate $\dot{\epsilon}_{ss}$, as shown in Figure 6b. A steady-state regime implies that the strain rate remains approximately constant over a finite strain interval, because the relevant hardening and recovery processes are in mechanical balance. The $\dot{\epsilon}_{\min}$ may coincide with the $\dot{\epsilon}_{ss}$ if such an extended strain-rate plateau is present. However, it may also occur merely as a local minimum between primary deceleration and tertiary acceleration when no extended steady-state regime is established. Therefore, the identification and interpretation of $\dot{\epsilon}_{\min}$ require inspection of both the strain-time curve and the strain-rate evolution. Following conventional practice in high-temperature plasticity, a representative steady-state creep rate should be evaluated over a sufficiently large plastic-strain interval, commonly at least $\sim 1\text{--}2\%$ of plastic strain when experimentally accessible [73].

In precipitation-strengthened alloys, the distinction between $\dot{\epsilon}_{ss}$ and $\dot{\epsilon}_{\min}$ is particularly important. Even when the macroscopic strain rate appears nearly constant, the microstructure may continue to evolve by dislocation rearrangement, recovery, precipitate coarsening, rafting, or changes in the dislocation-precipitate interaction mode. Thus, the secondary regime should be interpreted as a mechanical steady state only if the strain-rate plateau is sufficiently extended, whereas $\dot{\epsilon}_{\min}$ should be treated as an experimentally defined minimum value if such a plateau is absent [74].

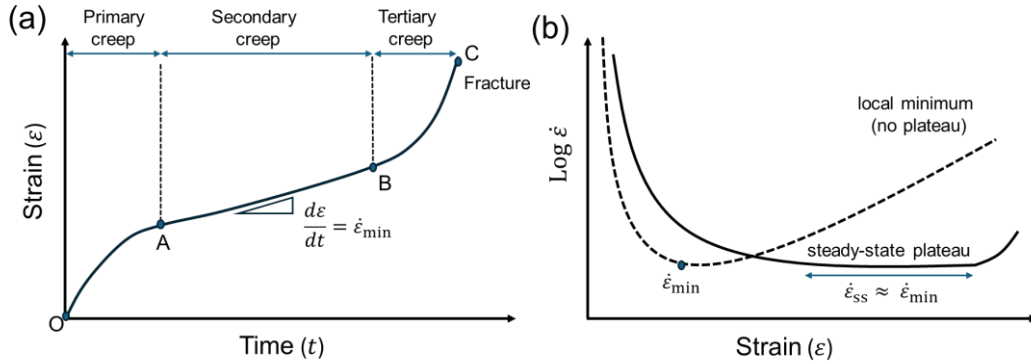


Figure 6. Schematic representation of creep deformation at elevated homologous temperatures. (a) Conventional creep strain-time curve showing primary, secondary, and tertiary creep regimes. (b) Corresponding strain-rate evolution plotted as $\log \dot{\epsilon}$ versus ϵ . A steady-state regime is characterized by an extended strain-rate plateau, whereas the $\dot{\epsilon}_{\min}$ may also occur as a local minimum between primary deceleration and tertiary acceleration without an extended steady state.

A widely used phenomenological description of $\dot{\epsilon}_{\min}$ is given by a power-law relation between applied stress and creep rate, often combined with an Arrhenius-type temperature dependence [71]:

$$\dot{\epsilon}_{\min} = A \sigma^{n_{\text{app}}} \exp\left(-\frac{Q_{\text{app}}}{RT}\right) \quad (2.3)$$

Here, A is the structure-dependent constant, σ is the applied stress, n_{app} is the apparent stress exponent, Q_{app} is the apparent activation energy, R is the gas constant, and T is the temperature. The term “apparent” is used because n_{app} and Q_{app} may include contributions from several concurrent processes, such as dislocation glide, climb, diffusion, recovery, and microstructural evolution in complex precipitation-strengthened alloys [75]. However, in simple metals dominated by lattice self-diffusion, Q_{app} may approach the activation energy for self-diffusion [76].

This phenomenological expression does not, by itself, specify the underlying deformation mechanism. The values of n_{app} and Q_{app} provide useful indicators of stress and temperature sensitivity, but they must be interpreted together with microstructural observations and mechanistic creep models [71]. This is particularly important for precipitation-strengthened alloys, where threshold-stress effects, precipitate evolution, and changes in dislocation-precipitate interaction can produce high n_{app} that do not correspond to a single mechanism in a single-phase material.

A more mechanistic normalization is given by the Mukherjee-Bird-Dorn (MBD) relation [74]:

$$\dot{\epsilon}_{\min} = A \frac{D_0 G b}{RT} \left(\frac{b}{d}\right)^p \left(\frac{\sigma}{G}\right)^{n_{\text{app}}} \exp\left(-\frac{Q_{\text{app}}}{RT}\right) \quad (2.4)$$

Here, A is the Dorn constant, and D_0 is the diffusion pre-exponential factor. G represents the shear modulus, b is the Burgers vector, d denotes the grain size, and p is the grain size exponent.

In this formulation, n_{app} and p provide useful but not unique mechanistic indications. Diffusion creep is usually associated with low stress exponents and a strong grain-size dependence. Nabarro-Herring creep is associated with lattice diffusion and approximately $p = 2$, whereas Coble creep is associated with grain-boundary diffusion and approximately $p = 3$. Dislocation creep generally exhibits higher stress exponents, often in the range of $n_{\text{app}} \approx 3-5$, while even higher n_{app} may arise from threshold-stress effects, precipitate strengthening, or a transition toward power-law breakdown. Therefore, the parameters in Eq. (2.4) should be interpreted together with microstructural observations rather than as unique identifiers of a single creep mechanism. In the present work, the creep specimens exhibit very large grains, such that only a few grain boundaries are contained within an

individual gauge section. Grain-boundary-mediated contributions are therefore expected to be of minor importance, and the following discussion focuses primarily on matrix-, precipitate-, and dislocation-controlled creep deformation.

2.2.2 Effective Stress and Threshold Stress

In precipitation-strengthened alloys, dislocations interact with precipitates, coherent or semi-coherent interfaces, and the associated elastic and chemical resistance fields. These interactions reduce the stress effectively available for dislocation motion in the matrix and thereby decrease the creep rate at a given applied stress. This concept is commonly expressed by introducing an effective stress, σ_{eff} , which represents the part of the applied stress that actively drives the relevant deformation process within the matrix [30,71]:

$$\sigma_{\text{eff}} = \sigma - \sigma_p \quad (2.5)$$

where σ is the applied stress and σ_p represents an internal resistance term associated with dislocation-precipitate interactions. In this formulation, σ_p should not be interpreted as a literal portion of the applied stress carried by the precipitates. Rather, it represents the mechanical resistance arising from dislocation-precipitate interactions, such as coherency stresses, back stresses, precipitate cutting resistance, or bypass resistance.

The applicability of this description depends on the active deformation path and the topology of the two-phase microstructure. It is most directly applicable when creep strain is mainly accommodated by dislocation motion in a continuous matrix phase, as in many classical precipitation-strengthened superalloys. In complex A2+B2 RHEAs, and particularly in alloys with inverted or strongly interconnected phase morphologies, this assumption must be examined carefully. Therefore, the effective-stress concept is used here as a framework for separating matrix-related deformation resistance from precipitate-related internal resistance, but its physical interpretation must always be linked to the observed microstructure.

Incorporating the effective-stress concept leads to a modified matrix-controlled creep relation for precipitation-strengthened systems [30,77]:

$$\dot{\epsilon}_{\text{min}} = A_1 (\sigma - \sigma_p)^{n_m} \exp\left(-\frac{Q_m}{RT}\right) \quad (2.6)$$

where A_1 is a material-dependent constant, n_m denotes the matrix-controlled stress exponent, and Q_m is the apparent activation energy associated with the matrix-controlled creep process. This formulation assumes that plastic strain is primarily accommodated by the matrix, while the precipitates reduce the effective stress available for matrix deformation. It should be noted that the value of n_m may vary with stress depending on the dominant deformation mechanism in the matrix phase.

The physical meaning of σ_p is illustrated schematically in Figure 7 using a Norton-type representation. In a double-logarithmic plot of $\dot{\epsilon}_{\min}$ versus σ , the matrix creep behavior follows a linear relation characterized by the intrinsic slope n_m . In precipitation-strengthened alloys, the presence of σ_p effectively shifts the stress scale required for creep deformation, resulting in a higher σ at a given creep rate. Consequently, the n_{app} obtained using σ becomes larger than the matrix-controlled exponent, i.e., $n_{app} > n_m$.

In the conventional interpretation, σ_p is often treated as an apparent threshold stress, i.e. an additional stress that must be overcome before a given creep rate can be achieved in a precipitation-strengthened alloy. This is an idealized description and should not be understood as a strict lower bound below which plastic flow is impossible. In more advanced descriptions, such as the Lagneborg-Bergman-Reppich (LBR) framework, σ_p is interpreted as an effective internal resistance or back-stress contribution associated with dislocation-precipitate interactions. In this case, σ_p is not necessarily constant but may vary with applied stress and approaches zero as the applied stress tends to zero. Consequently, creep deformation is not strictly prohibited below a fixed threshold stress but instead becomes progressively slower with decreasing applied stress. This distinction is important for the interpretation of experimental creep data and for the consistent application of mechanistic models. This concept of σ_p was originally developed in Ni-based superalloys and has since been applied to a wide range of precipitation-strengthened alloy systems [30,77,78].

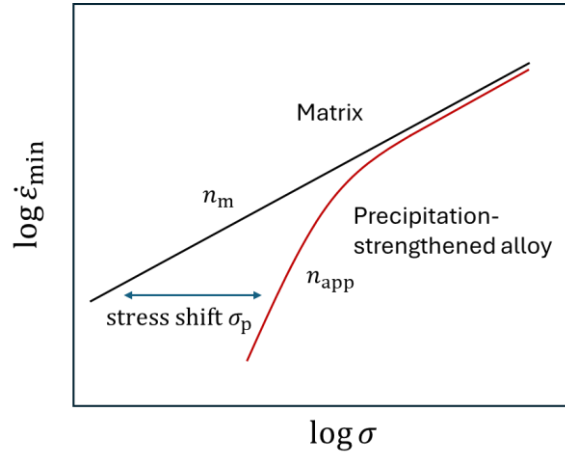


Figure 7. Schematic log-log representation of $\dot{\epsilon}_{\min}$ versus σ illustrating the σ_{eff} concept. The threshold stress σ_p produces a horizontal stress shift between the matrix and the precipitation-strengthened alloy. The slope of the matrix curve defines n_m , while the slope obtained for the strengthened alloy using the applied stress corresponds to the apparent stress exponent n_{app} .

2.2.3 Threshold Stress Estimation

Quantifying σ_p is essential for separating the intrinsic matrix-controlled creep behavior from the strengthening contribution of coherent precipitates. As introduced in Section 2.2.2, σ_p represents an effective internal resistance associated with dislocation-precipitate interactions. In classical precipitation-strengthened systems, two related approaches are commonly used for estimating σ_p : the original Lagneborg-Bergman (LB) method and its subsequent refinement, known as the Lagneborg-Bergman-Reppich (LBR) method [30,77].

In its original formulation, the LB method determines σ_p by comparing the creep response of a precipitation-strengthened alloy with that of a corresponding matrix alloy at the same temperature and $\dot{\epsilon}_{\min}$. For classical Ni-based superalloys, the reference matrix alloy ideally reproduces the chemical composition of the matrix phase, ensuring that the extracted σ_p primarily reflects precipitate-induced resistance rather than compositional differences. Under the assumption that the intrinsic matrix stress exponent n_m remains constant, Eq. (2.6) can be rearranged into the following form at a fixed temperature [32]:

$$\dot{\epsilon}_{\min}^{1/n_m} = A_2^{1/n_m} (\sigma - \sigma_p) \quad (2.7)$$

This formulation allows σ_p to be estimated graphically by linear back-extrapolation of transformed creep data to the stress axis, as illustrated schematically in Figure 8a. However, this approach relies on the assumption that n_m remains constant, which is often not fulfilled in practice. As a result, n_m and σ_p cannot generally be treated as constant simultaneously, limiting the reliability of this method.

To overcome this limitation, the LBR method was proposed by Reppich and co-workers [32]. In this approach, σ_p is determined as the horizontal stress difference between the precipitation-strengthened alloy and the corresponding matrix alloy at identical creep rates and temperatures. Unlike the LB method, the LBR approach does not require a constant stress exponent and provides a more physically consistent description when the matrix creep response varies with applied stress. The concept is illustrated schematically in Figure 8b.

An alternative and widely used approach is based on the logarithmic form of the effective stress relation at fixed temperature:

$$\ln(\dot{\epsilon}_{\min}) = \ln A_2 + n_m \ln (\sigma - \sigma_p) \quad (2.8)$$

Here, σ_p is iteratively adjusted until the data collapse onto a straight line in the double-logarithmic representation. The value of σ_p that restores linearity indicates that the effective stress has been correctly identified. This approach minimizes uncertainties associated with extrapolation and is particularly advantageous when a corresponding matrix alloy is not

available or when the stress exponent varies with applied stress, as is often the case in complex alloy systems.

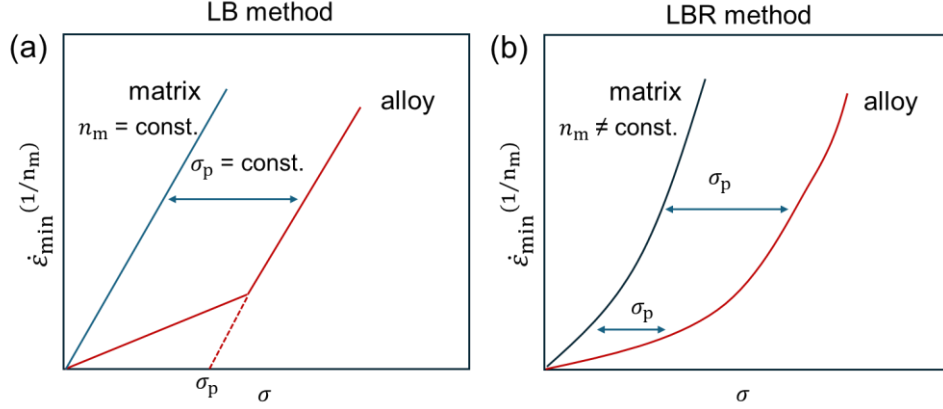


Figure 8. Schematic comparison of the LB and LBR methods for determining the threshold stress σ_p . (a) LB method assuming constant n_m and σ_p , yielding a linear representation in transformed coordinates. (b) LBR method using the matrix creep response as a physical reference, where σ_p is obtained as the horizontal stress difference at equal creep rates.

Correcting for σ_p is essential for meaningful mechanistic interpretation. When creep rates are analyzed directly using the applied stress, precipitation-strengthened alloys often exhibit very high n_{app} that do not correspond to any creep mechanism or model developed for single-phase materials. Normalization by the effective stress recovers matrix-controlled exponents characteristic of dislocation glide or climb, thereby distinguishing intrinsic deformation behavior from precipitate-induced resistance [77,79]. The applicability of this LBR method requires that a stable precipitate population persists during steady-state deformation and that creep rates of both matrix and multiphase alloy are available over a sufficiently broad range of stresses [79,80].

As stated above, the simplest threshold-stress treatments assume a constant matrix stress exponent. However, in many alloy systems, the apparent stress exponent varies with applied stress, particularly near the transition towards power-law breakdown. To account for this behavior, a sinh-type formulation is often employed [77]:

$$\dot{\epsilon}_{min} = B \sigma^{n^*-1} \sinh\left(\frac{V\sigma}{2kT}\right) \quad (2.9)$$

where n^* is a reference stress exponent, V is the activation volume, k is the Boltzmann constant, and T is the test temperature. This formulation smoothly transitions from a power-law at low stresses to an exponential dependence at high stresses. At low stresses, $\sinh(x) \approx x$, and the equation reduces to the conventional Norton-type power law. At high

stresses, $\sinh(x) \approx \frac{1}{2}e^x$, capturing the rapid increase in creep rate characteristic of the power-law breakdown (PLB) regime. Thus, the sinh-type model unifies classical Norton creep and PLB behavior within a single framework. Importantly, it facilitates the consistent application of the LBR method to systems where the stress exponent varies with applied stress.

From Eq. (2.9), the stress-dependent matrix creep exponent $n(\sigma)$ can be derived as [77]:

$$n(\sigma) = \frac{d \ln \dot{\epsilon}_{\min}}{d \ln \sigma} = (n^* - 1) + \left(\frac{V\sigma}{2kT} \right) \coth \left(\frac{V\sigma}{2kT} \right) \quad (2.10)$$

This formulation provides a consistent description of creep behavior over a wide stress range and is particularly useful when applying threshold stress approaches to systems in which the stress exponent is not constant.

2.2.4 Dislocation-Precipitate Interactions

In precipitation-strengthened alloys, the creep response is strongly affected by the interaction between mobile dislocations and precipitates within the matrix. As introduced in Section 2.2.2, such interactions can be described in terms of a precipitate-related internal resistance σ_p , which reduces the effective stress available for dislocation motion. In coarse-grained or near-single-crystalline precipitation-strengthened alloys, grain-boundary contributions to creep deformation are negligible, and plastic deformation is typically dominated by dislocation-controlled processes within the grains [71]. Under such conditions, the interaction between moving dislocations and precipitates becomes the primary factor governing creep resistance.

The magnitude of the precipitate-related resistance depends on several coupled microstructural and thermodynamic parameters, including precipitate size r , precipitate volume fraction f , interparticle spacing or channel width λ , interface coherency, lattice misfit δ , shear modulus G , and, for ordered precipitates, the APB energy γ_{APB} [71,77,79,80]. These factors collectively determine the effective resistance encountered by dislocations during plastic deformation and thereby directly influence the magnitude of σ_p .

In general terms, the precipitate-related resistance can be expressed as [80]:

$$\sigma_p = \alpha \frac{Gb}{\lambda} \Phi(r, f, \gamma_{\text{APB}}, \delta, T) \quad (2.11)$$

where G is the shear modulus, b is the Burgers vector, and Φ represents a mechanism-dependent function that accounts for the operative dislocation-precipitate interaction mechanism. The factor $\frac{Gb}{\lambda}$ reflects the Orowan bypassing stress scale, while Φ accounts for additional effects such as ordering, coherency, precipitate size, and temperature-dependent mobility. Depending on the balance among these parameters, dislocations may either cut the

precipitates, bypass them by looping, or circumvent them by climb-assisted motion. These mechanisms determine both the magnitude and the temperature dependence of σ_p [71,77,79].

At relatively low temperatures or for fine and coherent precipitates, dislocation cutting may occur if the applied stress is sufficient to overcome order- and coherency-related resistance [62,80]. In ordered systems, such as $\gamma+\gamma'$ superalloys or B2-strengthened alloys, dislocation cutting is additionally constrained by long-range chemical order. A leading partial dislocation cutting an ordered precipitate can create an APB, whereas a following partial can restore the ordered arrangement. The resistance against this process depends on γ_{APB} , precipitate size, precipitate volume fraction, and dislocation line tension [71].

For strongly coupled dislocation pairs cutting ordered precipitates, the critical cutting stress can be estimated using the H  ther-Reppich model [81]:

$$\tau_{HR} = \frac{1}{2} \cdot 0.83 \frac{T_L w f^{\frac{1}{2}}}{b r} \left[\frac{\pi^2 r \gamma_{APB}}{4 w T_L} - 1 \right]^{\frac{1}{2}} \cdot \left\{ 0.94 \left[1 + (C_{SL} f^{\frac{1}{2}})/2 \right] \right\} \quad (2.12)$$

where τ_{HR} is the critical shear stress for precipitate cutting, T_L is the dislocation line tension, w is an adjustable parameter in the H  ther-Reppich description of order strengthening by strong pair coupling, f is the precipitate volume fraction, b is the Burgers vector, r is the mean precipitate radius, and C_{SL} is the empirical constant introduced in the Schwarz-Labusch correction.

The dislocation line tension can be approximated as:

$$T_L \approx \frac{1}{2} G b^2 \quad (2.13)$$

Equations (2.12) and (2.13) show that cutting of ordered precipitates becomes easier when the shear modulus, line tension, precipitate size, or APB energy decreases. This is particularly relevant at elevated temperatures, where the shear modulus decreases. Furthermore, the γ_{APB} decreases with a reduced degree of long-range order. A reduction in γ_{APB} lowers the energetic penalty associated with APB formation and therefore decreases the critical stress required for coupled dislocation-pair cutting.

As the precipitate size increases, the elastic strain energy associated with coherency increases if the precipitates remain coherent. Above a critical size, this energy may be partly relieved by a transition to semi-coherent interfaces, for example through the formation of misfit dislocations. Under these conditions, precipitate cutting becomes less favorable, and bypass mechanisms such as Orowan looping may dominate. The corresponding Orowan stress can be approximated by [82,83]:

$$\tau_{\text{Orowan}} \approx 0.4 \frac{Gb}{\lambda} \quad (2.14)$$

where λ is the relevant interparticle spacing or effective channel width between precipitates. In a rafted microstructure, λ should be interpreted as the effective channel width along the dislocation path rather than as a general average particle spacing. Eq. (2.14) shows that a smaller channel width increases the stress required for glide-controlled bypass.

At higher homologous temperatures, diffusion-assisted dislocation climb becomes increasingly important, enabling dislocations to bypass precipitates without loop formation. Under this condition, σ_p generally decreases with increasing temperature and assumes only a fraction of $\frac{Gb}{\lambda}$, because the enhanced diffusion facilitates recovery and bypass processes. This transition in deformation mechanism is also reflected in the apparent stress exponent n_{app} , which decreases from high values ($n_{\text{app}} > 8$) in the cutting-dominated regime to moderate values ($n_{\text{app}} \approx 4-5$) when climb-assisted creep becomes rate-controlling [78,80].

Although these mechanisms are often described in terms of specific temperature regimes, their relative contributions are controlled by the combined influence of microstructural, mechanical, and thermodynamic parameters. Temperature primarily affects diffusion and recovery kinetics, whereas precipitate size, spacing, and γ_{APB} determine the resistance to dislocation motion. Consequently, the creep behavior of precipitation-strengthened alloys should be regarded as arising from the coupled effects of multiple parameters governing dislocation motion and precipitate stability.

In two-phase superalloys containing coherent, ordered precipitates, the stability and distribution of precipitates play a central role in controlling creep resistance. A uniform and stable precipitate distribution delays the transition from cutting to bypassing, thereby maintaining a higher σ_p over extended deformation durations [71]. These relationships provide the conceptual basis for correlating precipitate size, spacing, and stability with threshold stress and creep resistance in the precipitation-strengthened alloys discussed in the following chapters.

2.2.5 Coarsening and Rafting during Creep

The creep behavior of precipitation-strengthened alloys is closely linked to the stability and evolution of their two-phase microstructures. As discussed in Sections 2.2.2 and 2.2.4, the resistance to creep deformation is governed by dislocation-precipitate interactions and is therefore sensitive to changes in precipitate size, morphology, and spatial distribution. During high-temperature exposure, these microstructural features evolve continuously, leading to a corresponding change in σ_p . These microstructural changes occur primarily through two

fundamental processes: diffusion-controlled coarsening under elevated-temperature exposure and stress-assisted directional coarsening, commonly referred to as rafting, under mechanical loading. Both processes determine the microstructural state that governs dislocation motion during creep.

(1) Diffusion-Controlled Coarsening (Ostwald Ripening)

In the absence of significant external stress, precipitate evolution is dominated by diffusion-driven coarsening, commonly referred to as Ostwald ripening. In this process, solute atoms diffuse from smaller particles to larger ones to reduce the total interfacial energy between precipitate and matrix. As a result, the average precipitate size increases, while the precipitate number density decreases and the average interparticle spacing λ generally increases [84].

The kinetics of precipitate coarsening are often described by a generalized power-law relation:

$$\bar{r}^m - \bar{r}_0^m = \kappa t \quad (2.15)$$

where $\bar{r}$ is the average precipitate radius after ageing exposure time t , $\bar{r}_0$ is the initial average radius at the beginning of the coarsening regime, m is the coarsening exponent, and κ is the coarsening rate constant determined by diffusion and thermodynamic parameters. The exponent m depends on the rate-controlling coarsening mechanism and is expected to remain time-independent if this mechanism does not change.

For bulk-diffusion-controlled coarsening, the classical Lifshitz-Slyozov-Wagner theory (LSW) predicts $m = 3$ [85]. Eq. (2.15) then reduces to the cubic coarsening law:

$$\bar{r}^3 - \bar{r}_0^3 = \kappa t \quad (2.16)$$

This bulk-diffusion-controlled case is used for the coarsening analysis in the present study. For a multicomponent system, the LSW-type coarsening rate constant κ can be approximated as [85]:

$$\kappa = \frac{8 \gamma V_m^p D_i^m}{9RT \sum_i^N (c_i^p - c_i^m)^2 / c_i^m} \quad (2.17)$$

where γ is the interfacial energy, V_m^p is the molar volume of the precipitate, D_i^m is the diffusion coefficient of component i in the matrix, R is the gas constant, T is the absolute temperature, and c_i^p and c_i^m are the concentrations of component i in the precipitate and matrix, respectively. This expression shows that coarsening is promoted by high interfacial energy and fast diffusion, whereas strong chemical partitioning between matrix and precipitate reduces the coarsening rate.

The classical LSW theory is based on idealized assumptions, including diffusion-controlled growth and spherical precipitate morphology. However, in many precipitation-strengthened alloys, precipitates deviate from an ideal spherical morphology and may exhibit elongated or cuboidal shapes. In such cases, an equivalent particle radius can be introduced as a practical characteristic length scale based on volume conservation [86]:

$$r_{eq} = (a_p b_p^2)^{1/3} \quad (2.18)$$

where a_p and b_p denote the major and minor axes of the precipitates, respectively. This definition corresponds to the radius of a sphere with the same volume as the ellipsoidal particle. For a population of non-spherical precipitates, the mean equivalent radius, $\bar{r}_{eq}$, used in the coarsening analysis is obtained by averaging over all measured particles. Although this approximation does not explicitly account for shape anisotropy in the coarsening kinetics, it provides a practical measure for evaluating average precipitate growth in systems with non-spherical precipitates.

With prolonged high-temperature exposure, the formation of larger and more widely spaced precipitates generally reduces the resistance to dislocation motion. Consequently, σ_p tends to decrease with increasing exposure time, leading to a gradual degradation of creep resistance. Diffusion-controlled coarsening therefore provides the baseline microstructural evolution against which creep-induced directional changes must be interpreted.

(2) Directional Coarsening (Rafting)

When a uniaxial load is applied during creep, the external stress interacts with the internal misfit stress field associated with coherent precipitates. This superposition produces an anisotropic elastic strain-energy distribution, which can bias solute diffusion and drive precipitates to grow preferentially along directions that minimize the total elastic strain energy. This stress-assisted morphological evolution is referred to as directional coarsening or rafting. Rafting is well established in Ni- and Co-based superalloys and has been studied particularly extensively in $\langle 001 \rangle$ -oriented single crystals, where regular raft morphologies are readily formed and analyzed [26,87–93]. However, rafting is not restricted to the $\langle 001 \rangle$ orientation. It may also occur under other loading orientations, such as $\langle 110 \rangle$ or $\langle 111 \rangle$, although the resulting morphology and apparent degree of rafting can differ substantially. In polycrystalline alloys, the local rafting response is therefore expected to vary from grain to grain depending on crystallographic orientation, stress state, lattice misfit, elastic anisotropy, and precipitate morphology [88,89,94,95].

The direction of rafting is commonly discussed in terms of the lattice misfit between precipitate and matrix:

$$\delta = \frac{2(a_p - a_m)}{a_p + a_m} \quad (2.19)$$

where a_p and a_m denote the lattice parameters of the precipitate and matrix phases, respectively. For a positive lattice misfit ($\delta > 0$, the case in the alloy system under consideration in this work), the precipitate has a larger lattice parameter than the matrix. Under coherent conditions, elastic compatibility imposes compressive stresses within the precipitates and tensile stresses in the surrounding matrix (Figure 9a). In the absence of external loading, this misfit-induced stress field is spatially symmetric, resulting in an isotropic elastic strain energy distribution and thus isotropic coarsening.

Upon application of uniaxial compressive stress (as performed in the experimental part of this thesis, Section 3.3.1), the externally applied stress superimposes on the internal misfit stress (Figure 9b). Along the loading axis, the compressive stresses reinforce each other, increasing the elastic strain energy in this direction, whereas perpendicular to the loading axis, the elastic energy is reduced. The system lowers its total elastic energy by redistributing solutes, leading to precipitate elongation perpendicular to the loading direction. This is referred to as N-type rafting (Figure 9c). Conversely, when the applied stress and misfit strain act in the same direction along a given direction, elongation occurs parallel to the stress axis, resulting in P-type rafting [96].

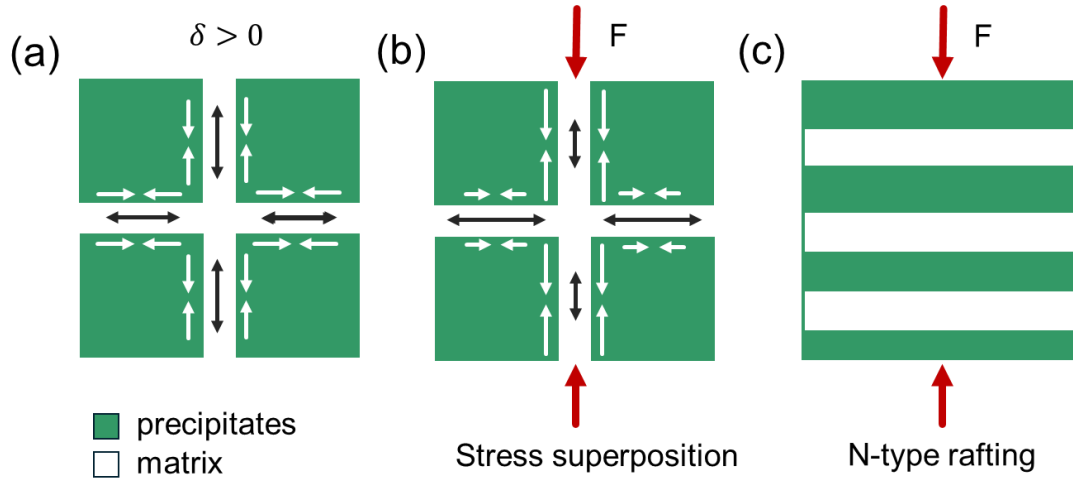


Figure 9. Schematic illustration of stress-assisted rafting for coherent precipitates with positive lattice misfit, $\delta > 0$, under compressive loading. (a) Symmetric misfit-induced internal stress field around coherent precipitates in the absence of an external load. (b) Superposition of misfit strain and external compressive stress, resulting in an anisotropic elastic strain energy distribution. (c) Formation of N-type rafts, where precipitates elongate perpendicular to the loading direction to reduce the total elastic energy.

Directional coarsening alters the topology of the precipitate network and thereby modifies dislocation motion pathways. Depending on the alignment of precipitates relative to the loading direction, rafting may either enhance or reduce creep resistance. As demonstrated by Saito and co-workers [97], the degree of raft perfection is a key factor governing high-temperature creep performance. A well-developed and continuous raft microstructure can effectively impede dislocation motion and stabilize σ_p under high-temperature, low-stress conditions, whereas irregular or discontinuous rafting may accelerate deformation.

Diffusion-controlled coarsening and stress-assisted rafting represent two fundamental modes of precipitate evolution during creep. Their combined influence governs the temporal evolution of σ_p and thus determines the long-term creep performance of precipitation-strengthened alloys. These concepts provide the basis for interpreting the microstructural evolution observed in the investigated A2+B2 alloy system.

3 Experimental Methods

3.1 Materials Preparation

3.1.1 Alloy Composition and Melting

To investigate the high-temperature creep behavior of an A2+B2 two-phase RHEA, a base alloy with the nominal composition of 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al (at.%), hereafter referred to as TMT-8Cr-10Al, was synthesized [13,98]. To enhance the precipitate T_{solv} and thermal stability, Re and Ru were subsequently introduced into the TMT-8Cr-10Al base alloy at concentrations of 1.5, 2.5, and 5 at.% each. To maintain the overall compositional balance, the additions of Re and Ru were compensated by equiatomic reductions of Ta, Mo, and Ti. Based on the type of alloying element, the modified compositions were classified into two series: Re-modified and Ru-modified alloys. The resulting compositions are designated as 1.5Re, 2.5Re, and 5Re, as well as 1.5Ru, 2.5Ru, and 5Ru, according to their nominal Re or Ru contents in atomic percent. The nominal compositions of all alloys investigated in this work are summarized in Table 1.

All alloys were produced by repetitive arc melting in an inert Ar atmosphere, using a water-cooled copper crucible in an AM/0.5 arc melting furnace (Edmund Bühler GmbH, Germany). Approximately 150 g of each alloy was produced. The raw elements Ta, Mo, Ti, Cr, Al, Re, and Ru were sourced from chemPUR GmbH (Germany), with purities of 99.9%, 99.95%, 99.8%, 99.99%, 99.99%, 99.9% and 99.9%, respectively. The elements were accurately weighed according to the designed compositions and placed into the copper crucible. Prior to melting, the chamber underwent three evacuation and high-purity Ar ($\geq 99.998\%$, ARCALTM Prime) backfilling cycles, reducing the pressure to approximately 5×10^{-2} mbar, followed by flooding with Ar to ambient pressure. After the cycles, a high vacuum of 4×10^{-5} mbar was achieved to minimize O contamination. During the melting operation, the Ar pressure was maintained at 600 mbar to provide an inert environment. A small Zr piece was melted prior to each melting step to act as an O getter. Each alloy button was fully melted, allowed to cool for 2 minutes, then flipped and re-melted at least five times to promote chemical homogeneity. Due to the large variation in melting and boiling points among the alloying elements (e.g., Ta melting at ~ 3020 °C, while Cr and Al boil below this temperature), special attention was given to process control measures to minimize elemental evaporation and ensure complete melting. The final chemical compositions of the investigated alloys and the concentrations of O and N were determined by inductively coupled plasma-optical emission spectroscopy (ICP-OES) and carrier gas hot extraction (CGHE), respectively, and are summarized in Table 1.

Table 1. Nominal target compositions, experimentally measured compositions, and interstitial impurity contents of the alloys investigated in this work. The concentrations of O and N were determined by CGHE.

Abbreviation	Target Composition	Experimental Composition / at.%							Impurities / at-ppm	
		Ta	Mo	Ti	Cr	Al	Re	Ru	O	N
TMT-8Cr-10Al	27.3Ta-									
	27.3Mo-27.3Ti-8Cr-10Al	26.0	29.8	27.4	7.3	9.4	–	–	2240 ± 560	330 ± 100
1.5Re	26.8Ta-									
	26.8Mo-26.8Ti-8Cr-10Al-1.5Re	28.9	27.9	28.4	6.0	7.4	1.4	–	1370 ± 114	545 ± 331
2.5Re	26.5Ta-									
	26.5Mo-26.5Ti-8Cr-10Al-2.5Re	28.9	27.5	28.1	5.7	7.2	2.6	–	1322 ± 140	306 ± 70
5Re	25.7Ta-									
	25.7Mo-25.7Ti-8Cr-10Al-5Re	27.0	25.1	27.4	6.9	8.5	5.2	–	1646 ± 633	405 ± 433
1.5Ru	26.8Ta-									
	26.8Mo-26.8Ti-8Cr-10Al-1.5Ru	29.1	27.8	27.4	6.5	7.9	–	1.3	1911 ± 119	381 ± 102
2.5Ru	26.5Ta-									
	26.5Mo-26.5Ti-8Cr-10Al-2.5Ru	28.2	27.7	27.8	6.1	7.6	–	2.6	1008 ± 24	136 ± 10
5Ru	25.7Ta-									
	25.7Mo-25.7Ti-8Cr-10Al-5Ru	28.9	27.8	26.0	5.5	6.7	–	5.0	417 ± 0	142 ± 20

An additional monolithic A2 reference alloy was prepared to represent the matrix phase of the A2+B2 TMT-8Cr-10Al microstructure. The target composition was derived from the A2 matrix composition obtained by APT after aging the TMT-8Cr-10Al alloy at 1000 °C for 1000 h [25]. The alloy was produced using the same arc melting procedure described above. The nominal composition of the monolithic A2 alloy is summarized in Table 2.

Table 2. Desired and experimental composition of the monolithic A2 alloy (at.%).

Abbreviation	Target Composition					Experimental Composition / at.%				
	Ta	Mo	Ti	Cr	Al	Ta	Mo	Ti	Cr	Al
Monolithic A2	30.4	31.6	25.1	7.1	5.7	33.2	32.9	25.5	4.9	3.5

3.1.2 Heat Treatment

Two distinct heat treatment procedures were applied to prepare the specific microstructural conditions for subsequent experiments.

To eliminate dendritic segregation in the as-cast condition and promote chemical homogeneity prior to mechanical testing and microstructural characterization, the as-cast alloy buttons were homogenized at 1600 °C for 72 h [13,25] in a HTRT 70-600/18 resistance tube furnace (Carbolite Gero GmbH & Co. KG, Germany). The heating and cooling rates were 100 K/h. Prior to heat treatment, the alloy samples were wrapped in Ta foil and placed in Al₂O₃ crucibles containing Ti sponge, which served as an oxygen getter under flowing inert gas conditions. To prevent potential chemical reactions between the alloy and the crucible walls, Mo foil was inserted between the samples and the Al₂O₃ crucible surface. The furnace chamber was evacuated and backfilled with high-purity Ar ($\geq 99.998\%$) three times to minimize oxygen contamination. A continuous low-flow Ar purge (≤ 1 mL/min) was maintained throughout the homogenization process.

For subsequent isothermal aging and quenching experiments, small cuboidal specimens were cut from the homogenized alloy using electrical discharge machining (EDM). These samples were again wrapped in Ta foil and sealed in fused-silica glass ampoules under vacuum. To absorb residual O, Ti sponge was placed around and above the specimens inside the ampoules. The sealed ampoules were then annealed in a L3/S27 resistance furnace from Nabertherm (Germany) at various temperatures and durations depending on the specific experimental objective. After heat treatment, rapid quenching was performed by breaking the ampoules in water. The limited oxidation of the Ti getter after heat treatment indicated that the samples remained well protected from oxidation during the process.

3.1.3 Density Determination

The density of the alloys was determined using the Archimedes principle, commonly used for small or irregularly shaped samples. A Mettler-Toledo ME204T precision balance equipped with a density measurement was used for this purpose. The samples tested were extracted from the homogenized condition of the investigated alloys. Each specimen piece had a mass of around 0.8 g, suitable for precision measurement. To ensure measurement precision, each specimen was polished with P320, P600, and P1000 SiC papers to remove oxides, followed by ultrasonic cleaning in isopropanol for 10 min to remove surface contaminants.

Measurements were performed in air and in distilled water at room temperature, and the density was calculated according to the Archimedes principle using the following equation:

$$\rho = \frac{F_{\text{air}}}{F_{\text{air}} - F_{\text{water}}} \times \rho_{\text{water}} \quad (3.1)$$

where F_{air} and F_{water} are the measured forces acting on the sample in air and when fully immersed in distilled water, and ρ_{water} is the density of water at the measurement temperature.

3.2 Microstructural Characterization

3.2.1 Sample Preparation

Samples were prepared either from homogenized bulk alloys or directly from as-cast ingots, depending on the specific requirements of the corresponding tests and characterization methods.

Compression creep specimens were extracted from homogenized material using EDM to ensure geometric precision and consistency, with final dimensions of $(5 \times 3 \times 3) \text{ mm}^3$. All six surfaces were ground with SiC abrasive papers up to P2500 grit to remove surface oxides and provide smooth surfaces suitable for high-temperature mechanical testing. Differential scanning calorimetry (DSC) specimens were also prepared by EDM, shaped into discs with diameters of 5–6 mm and a thickness of $\sim 1 \text{ mm}$. All surfaces were ground to P2500 grit, and one surface was further polished to P4000 grit to ensure good thermal contact with the DSC crucible. Specimens for microstructural analysis, chemical composition determination, and X-ray diffraction (XRD) had no strict geometric requirement and were cut from either the as-cast or homogenized alloys using a precision diamond wire saw (Model 7500, Well Diamantdrahtsäge GmbH, Mannheim, Germany).

Samples for scanning electron microscopy (SEM) and electron backscatter diffraction (SEM-EBSD) were prepared using the following standardized metallographic steps: (1) Samples were hot mounted in EpoMet F resin (Buehler, USA). (2) Grinding was performed under a

central load using SiC abrasive papers up to P4000 grit, with a load of 20 N per sample, at 150 rpm in counter rotation. (3) Polishing was conducted sequentially using 3 and 1 μm diamond suspensions on MD-DAC cloths (Struers, Germany), each for 5 min at 20 N. (4) A final chemical-mechanical polish followed, using OPS-S NonDry colloidal silica suspension (pH 9.8) on an MD-Chem cloth (Struers, Germany) for 15 min. (5) The polished samples were subjected to vibratory polishing for more than 16 h using a Buehler VibroMet 2 polisher, with MicroCloth and OPS-S suspension.

XRD samples were ground with SiC papers up to P2500 grit to eliminate surface oxidation and ensure sufficient planarity. For chemical composition analysis, including CGHE and ICP-OES, small representative specimens were extracted from the alloy buttons and sequentially ground with P600 and P1000 SiC papers. All prepared specimens, regardless of the test type, were ultrasonically cleaned in ethanol prior to use to eliminate surface contaminants and residues.

3.2.2 Scanning Electron Microscopy (SEM)

SEM was employed to characterize the microstructural evolution of the RHEAs studied across different processing conditions, including as-cast, homogenized, and post-creep conditions. Multiple SEM systems were utilized to accommodate both low- and high-magnification observations and to obtain complementary compositional and crystallographic data.

For general microstructural characterization at lower magnification, a Zeiss EVO50 was used. Imaging was conducted using both secondary electron (SEM-SE) and backscattered electron (SEM-BSE) detectors to capture surface morphology and atomic number contrast, respectively. The accelerating voltage was adjusted between 10 and 20 kV depending on the resolution and contrast requirements. Compositional analysis was conducted via energy-dispersive X-ray spectroscopy (SEM-EDS). For each sample, SEM-EDS spectra were collected from five randomly selected regions. ZAF corrections (atomic number, absorption, and fluorescence) were applied using Aztec software (Oxford Instruments, UK), and the results were averaged. To improve reliability, calibration was performed using reference data obtained by ICP-OES on homogenized alloy samples. For detailed microstructural analysis and confirmation of the nanoscale two-phase morphology, high-magnification SEM imaging was conducted using a Zeiss LEO 1530 (Zeiss, Germany) under similar accelerating voltages (10–20 kV). This setup allowed clear identification of the fine precipitate microstructures, interfacial features, and deformation-induced morphological changes relevant to the creep behavior of the RHEAs investigated.

3.2.3 Electron Backscatter Diffraction (SEM-EBSD)

SEM-EBSD was employed to characterize the crystallographic orientation and deformation-induced microstructural evolution of the base TMT-8Cr-10Al alloy under creep conditions.

SEM-EBSD measurements were conducted using a Zeiss Auriga 60 SEM, equipped with a field emission gun and dedicated analytical detectors. An accelerating voltage of 20 kV was applied, with samples tilted at 70° and positioned at a working distance of 16–20 mm during data acquisition. The diffraction patterns were recorded using a DigiView camera (EDAX, USA), and data collection was performed with OIM Data Collection software. Post-processing was carried out in OIM Analysis, where standard noise-reduction procedures were applied. These included the neighbor confidence index (CI) correlation method (CI threshold < 0.05) and grain dilation using a grain tolerance angle of 5°, to enhance the reliability and completeness of orientation maps. To analyze crystallographic orientation-dependent creep deformation behavior, grain orientations corresponding to the three corners of the standard inverse pole figure (IPF) triangle for the loading axis, namely [001], [101], and [111], were examined. Rafting behavior was analyzed with respect to the loading axis, providing insight into anisotropic deformation characteristics within the polycrystalline alloy.

3.2.4 Transmission Electron Microscopy (TEM)

Transmission electron microscopy (TEM) was used to identify the A2 matrix and B2 precipitates and their crystallographic relationships, as well as to characterize dislocation behavior in crept specimens.

Electron-transparent lamellae were prepared by focused ion beam (FIB) milling using dual-beam systems (Zeiss Auriga and FEI Strata 400S). A protective Pt layer (~200 nm) was deposited prior to milling. Site-specific lift-outs were performed using a micromanipulator and mounted on Cu half grids. Final thinning was carried out at decreasing ion beam currents (500 pA to 20 pA) and voltages (30 kV to 5 kV), resulting in lamella thicknesses below 100 nm.

TEM investigations were conducted using Thermo Fisher Themis Z and Themis 300 microscopes operated at 300 kV. Conventional TEM and scanning transmission electron microscopy (STEM) modes were employed. Bright-field (TEM-BF), dark-field (TEM-DF), and selected area electron diffraction (TEM-SAED) were used for phase identification and crystallographic analysis. High-resolution transmission electron microscopy (HRTEM) and STEM imaging were applied where necessary. STEM investigations were performed using a high-angle annular dark-field (HAADF) detector. Elemental mapping was carried out by STEM-EDS when necessary.

The relative difference in lattice plane spacings between the A2 matrix and B2 precipitates under constrained conditions was determined from atomic-resolution STEM-HAADF images. Interplanar spacings were measured from filtered images obtained by fast Fourier transform (FFT) analysis. Lattice parameters for both A2 and B2 phases were averaged from multiple regions to improve statistical reliability. The lattice misfit δ is generally defined as the relative difference between the unconstrained lattice parameters of the two phases and was then

calculated from these measured lattice parameters using Eq. (2.19) given in Section 2.2.5. It should be noted that the TEM-based measurements performed in the present work provide the relative difference in lattice plane spacings under constrained conditions within the bulk microstructure. Therefore, the experimentally determined values may deviate from the true unconstrained lattice misfit value as defined above.

TEM lamellae from crept specimens tested at 1030 °C under 100, 125 and 150 MPa were prepared using the same FIB procedure, with lift-outs taken from grains in which the [001] crystallographic direction was approximately parallel to the loading axis as identified by SEM-EBSD. Milling parameters were adapted accordingly, with final cleaning performed at 2 kV. Diffraction-contrast imaging was carried out using a Thermo Fisher Tecnai F30 operated at 300 kV. TEM-SAED, TEM-DF, and two-beam TEM-BF imaging were used for precipitate and dislocation analysis. Atomic-resolution STEM-HAADF imaging was performed using a probe-corrected Titan Themis operated at 300 kV.

3.2.5 Three-Dimensional Representation of B2 Precipitates

Selected creep specimens of TMT-8Cr-10Al and annealed 5Re and 5Ru alloys were examined to assess the three-dimensional morphology of B2 precipitates. For the crept condition, a grain oriented close to $\langle 100 \rangle$ was selected based on EBSD analysis. A site-specific micropillar was prepared from the selected grain by FIB milling using a Thermo Fisher Helios G4 dual-beam system.

The micropillar was oriented to expose the top, lateral, and front surfaces of the same grain, allowing a comprehensive assessment of precipitate morphology along different crystallographic directions. During FIB preparation, a protective Pt layer ($\sim 1 \mu\text{m}$ thick) was first deposited to prevent ion-induced damage. Subsequent coarse milling was carried out at 30 kV with a beam current of 21 nA, followed by a final polishing step using a current of 2.4 nA to minimize surface artefacts. All micrographs were recorded using an in-column BSE detector, while operating in immersion mode. Pseudo-3D representation of the precipitate network was then achieved by correlating the microstructural features observed on the top, lateral, and front surfaces, providing spatial information on the rafting morphology and its connectivity within the A2+B2 microstructure.

3.2.6 Atom Probe Tomography (APT)

Three-dimensional atom probe tomography (APT) was employed to investigate the elemental partitioning behavior of Re- and Ru-modified alloys based on TMT-8Cr-10Al.

Needle-shaped specimens were fabricated using dual-beam FIB systems (FEI Strata and Zeiss Auriga). The regions of interest were site-specifically lifted out and mounted onto Si micro-posts via Pt deposition. Tip sharpening was carried out by annular milling under gradually

decreasing Ga⁺ ion beam currents (from 500 pA down to 20 pA), resulting in apex radii below 50 nm. To reduce Ga implantation and surface amorphization, a final low-energy polishing step (5 kV, 50 pA) was applied. APT measurements were performed using a LEAP 4000X HR instrument (Cameca, USA) operated in laser-pulsed mode with a UV laser ($\lambda = 355$ nm, 60 pJ) at pulse frequencies of 100–200 kHz. Samples were maintained at cryogenic temperatures (~ 50 K) to suppress thermal diffusion and enable controlled field evaporation. The detection rate was set to $\sim 0.3\%$ atoms per pulse to avoid multiple-hit events and maintain analytical fidelity. Detected ion data, including time-of-flight, lateral coordinates, and sequence number, were collected for each event. Data reconstruction and processing were conducted using IVAS 3.8.8 and AP Suite 6 software (Cameca). The software applies trajectory correction algorithms and spatial calibration based on tip geometry and evaporation sequence, allowing reconstruction of a 3D atomistic model of the analyzed volume.

3.2.7 X-ray Diffraction (XRD)

X-ray diffraction (XRD) was performed to identify the phase constitution and determine the lattice parameters of the studied alloys. All measurements were carried out using a Bruker D2 Phaser diffractometer operating in Bragg-Brentano geometry with a Cu K α radiation source ($\lambda = 1.5406$ Å). The 2θ scanning range was set between 10° and 145° , with a step size of 0.01° and an accumulated detection time of 384 s per step. To ensure uniform exposure and reduce texture effects, the samples were continuously rotated during the measurement. The diffraction peaks were indexed according to the A2 and B2 crystal structures. Peak assignment and phase identification were conducted using standard powder diffraction databases. Bragg's law [99] was applied to calculate the lattice parameter a :

$$2d_{hkl} \sin(\theta) = \lambda \quad (3.2)$$

For cubic systems [99]:

$$d_{hkl} = a / \sqrt{h^2 + k^2 + l^2} \quad (3.3)$$

Only peaks satisfying the structure factor conditions for BCC and B2 lattices ($h + k + l = \text{even}$) were considered valid. When $K_{\alpha 1}$ and $K_{\alpha 2}$ peak separation occurred, only the $K_{\alpha 1}$ peak was used to ensure accuracy in peak position and lattice parameter calculation.

To compensate for systematic errors such as sample displacement or surface irregularity, a correction function $f_{\text{corr}}(\theta)$ was applied following the method described in the literature [100]:

$$f_{\text{corr}}(\theta) = 0.5 \times [\cot^2(\theta) + \cos(\theta) \cdot \cot(\theta)] \quad (3.4)$$

Lattice parameters calculated from individual peaks were weighted using $f_{corr}(\theta)$ and linearly extrapolated to $f_{corr}(\theta) = 0$ (equivalent to $\theta = 90^\circ$). The extrapolated value was taken as the final lattice parameter of the alloy.

While XRD provided useful insight into phase presence and crystallographic changes, it is important to note that interfacial lattice misfit between A2 and B2 phases cannot be reliably determined via the applied XRD method due to its limited spatial resolution.

3.2.8 Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) was used to investigate the thermal behavior of the designed A2+B2 RHEAs. All experiments were conducted using a high-sensitivity DSC 404 F1 Pegasus system (Netzsch-Gerätebau GmbH, Germany) operating in dynamic heat flux mode. The measurements were performed with the sample and reference crucibles thermally coupled inside a single furnace. Samples were placed in Pt crucibles with Al_2O_3 inserts, covered with perforated Pt lids to permit controlled gas exchange. To prevent oxidation and ensure stable thermal conditions, a constant flow of ultra-high-purity argon (99.9999%) was maintained throughout the measurement. Heating rates of 10, 20, and 40 K/min were applied to assess the thermal response of the B2 phase. To ensure accurate and reproducible measurements, the DSC system was calibrated using standard melting point references (Al, In, Ag, Au), and sapphire was used as the enthalpy calibration material, in accordance with DIN 51007:2019. Prior to each test, crucibles, lids, and Al_2O_3 inlays were ultrasonically cleaned in isopropanol and annealed at 1200 °C under inert conditions to remove residual contaminants. The thermal signals were processed using proprietary Netzsch software, including baseline correction, signal smoothing, and peak analysis.

3.2.9 Image Analysis

To quantify the morphological characteristics and spatial distribution of B2 precipitates after long-term annealing treatments, a systematic image analysis procedure was conducted based on SEM-BSE micrographs using ImageJ software (NIH, USA) [101]. SEM-BSE images were converted to 8-bit grayscale format and segmented by thresholding based on the contrast between the B2 precipitates and the A2 matrix. The resulting binary images were analyzed using the “Analyze Particles” function to extract precipitate statistics.

For each analyzed condition, the projected area A , perimeter P , and the major and minor axes of each precipitate, a_p and b_p , were determined. Precipitate shape was quantified using the circularity index:

$$C = \frac{4\pi A}{P^2} \quad (3.5)$$

A value of $C = 1$ corresponds to a perfect circle, while lower values indicate elongated or irregular particle shapes.

Different characteristic size parameters were used depending on the precipitate morphology. For approximately equiaxed and well-separated precipitates, the area-equivalent radius was calculated from the projected area:

$$r_{eq,A} = \sqrt{\frac{A}{\pi}} \quad (3.6)$$

For anisotropic but still separated precipitates, the major and minor particle axes, a_p and b_p , were measured directly. In the Re-modified alloys, where the precipitates remained sufficiently separated, the equivalent particle radius was calculated based on Eq. (2.18). In the Ru-modified alloys, prolonged aging led to partial interconnection of the precipitates, so that the full particle dimensions could no longer be determined reliably. In these cases, the minor axis b_p , corresponding to the precipitate thickness, was used as the most robust characteristic length scale.

The B2 area fraction was determined from the ratio of the segmented B2 area to the total analyzed image area. For approximately isotropic and randomly distributed precipitates, this value was used as an estimate of the precipitate volume fraction. For strongly anisotropic or partially interconnected morphologies, it was treated as a two-dimensional area fraction.

The two-dimensional number density of precipitates was calculated as:

$$N_A = \frac{N_p}{A_{image}} \quad (3.7)$$

where N_p is the total number of segmented precipitates and A_{image} is the analyzed image area. Because both equiaxed and anisotropic, partially interconnected precipitate morphologies are present in this work, stereological conversion to a three-dimensional number density was not generally applied. The number density is therefore reported as a two-dimensional quantity and used for relative comparisons between different conditions.

For conditions in which the precipitate size approached the resolution limit of SEM-BSE imaging, the segmentation procedure was highly sensitive to image contrast and threshold selection. In these cases, the uncertainty was estimated by varying the threshold parameters within a reasonable range. The resulting error bars therefore reflect both the statistical variation of the analysis and the uncertainty associated with threshold selection.

3.3 Mechanical Testing

3.3.1 Compression Creep Testing

To evaluate the creep resistance, uniaxial compression creep tests were performed in the temperature range of 1000–1060 °C under constant compressive stress levels between 75 and 175 MPa. All test specimens were prepared from homogenized alloy buttons via EDM and cut into cuboidal shapes ($3 \times 3 \times 5 \text{ mm}^3$) to ensure dimensional consistency. The surfaces in contact with the compression dies were mechanically ground up to P2500 grit to reduce frictional effects and remove oxide layers. Additionally, a BN spray coating was applied to the contacting surfaces to further minimize interfacial friction during loading. Creep testing was carried out on a Zwick Z100 SW universal testing machine, equipped with a Maytec MSRA-81/P/RS high-temperature vacuum furnace. The tests were performed under high vacuum conditions (typically $< 10^{-4}$ mbar) to ensure sufficiently low oxide partial pressure during high-temperature exposure.

The system was closed-loop digitally controlled, ensuring a constant true compressive stress during each test. The constant true compressive stress (σ_t) was calculated from the engineering stress σ_e and engineering strain ε_e according to:

$$\sigma_t = \sigma_e(1 - \varepsilon_e) \quad (3.8)$$

where σ_e and ε_e are taken as positive under the compressive loading.

For all tests, temperature gradients along the sample length were verified to be less than 1 K at testing temperatures, ensuring thermal uniformity. The strain-time data were used to extract $\dot{\varepsilon}_{\min}$. The stress and temperature dependence of creep behavior was analyzed in terms of apparent stress exponent (n_{app}) and apparent activation energy (Q_{app}), discussed in later sections. The validity of results across multiple systems and sample shapes was confirmed by consistent $\dot{\varepsilon}_{\min}$ values, with observed deviations well within the sample-to-sample variation.

4 Creep Behavior of the A2+B2 TMT-8Cr-10Al

This chapter establishes the creep behavior of the A2+B2 TMT-8Cr-10Al alloy in relation to precipitate stability and microstructural evolution. The analysis begins with the determination of the precipitate solvus temperature, followed by the characterization of the initial microstructure, creep response, rafting behavior, and B2 strengthening contribution. Parts of the results presented in Chapter 4 have been previously published in Ref. [98]. TEM experiments used for lattice misfit determination were carried out in collaboration with Dr. Aparajita Pramanik and Prof. Ankur Chauhan (Indian Institute of Science, Bengaluru, India).

4.1 Thermal Stability of A2+B2 TMT-8Cr-10Al

To interpret the high-temperature creep behavior of TMT-8Cr-10Al, the precipitate solvus temperature T_{solv} of the A2+B2 two-phase microstructure must be determined. As discussed in Section 2.1.3, this stability limit is defined by the precipitate T_{solv} , which marks the transition from a two-phase microstructure to a single-phase A2 solid solution.

Previous estimates of this stability limit were mainly derived from DSC measurements, which indicated an apparent B2 ordering temperature of about 1050 °C [13]. However, such calorimetric data do not provide direct information on the temperature required for complete precipitate dissolution under prolonged thermal exposure. Therefore, T_{solv} is determined in the present work based on microstructural observations after long-term annealing at different temperatures, followed by quenching and SEM-BSE characterization. Annealing temperatures between 1040 and 1070 °C were selected to bracket the expected T_{solv} range and to capture the progressive reduction in precipitate volume fraction up to complete dissolution. Each sample was held isothermally for 95 h, matching the thermal exposure applied in the subsequent creep experiments (see Section 4.3.1).

Figures 10a–d present the SEM-BSE microstructures of specimens annealed at 1040, 1050, 1060, and 1070 °C for 95 h. At 1040 and 1050 °C, well-defined precipitates are observed in the A2 matrix, indicating that both temperatures are below T_{solv} . At 1060 °C, the precipitate fraction is significantly reduced, but residual precipitates are still detected, demonstrating that this temperature remains below T_{solv} , although the thermodynamic driving force for phase separation is significantly reduced. In contrast, no precipitate contrast is detected after annealing at 1070 °C, consistent with complete dissolution of the precipitates into the A2 matrix. The quantitative evolution of the precipitate volume fraction shown in Figure 10e, as obtained from image-based area fraction measurements, supports these observations. Based on these results, T_{solv} is estimated to lie between 1060 and 1070 °C. For the following

discussion, the midpoint of the experimentally resolved dissolution interval is defined as T_{solv} , yielding a value of $(1065 \pm 5) ^\circ\text{C}$.

The value of T_{solv} is approximately 10–20 K higher than the apparent B2 T_{ord} obtained from DSC measurements [13], as schematically illustrated in Figure 10f. This difference indicates that the calorimetric signal reflects the onset of ordering-related transformations, whereas complete dissolution of precipitates under prolonged exposure requires higher temperatures and diffusion-controlled processes. The T_{solv} determined here therefore provides the reference temperature for assessing precipitate stability during creep.

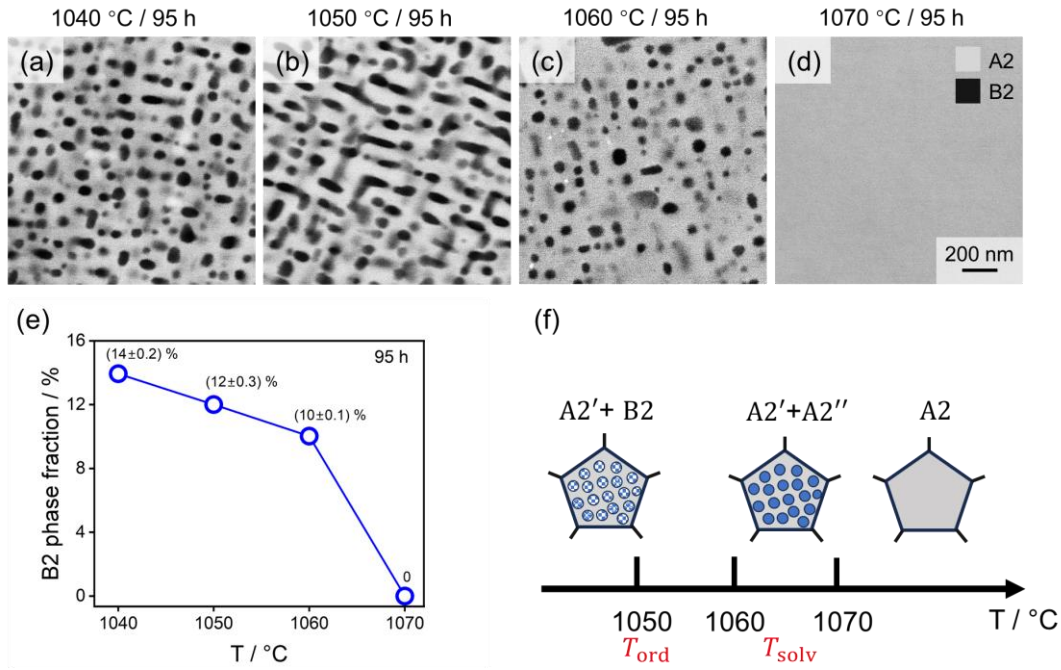


Figure 10. Determination of the precipitate solvus temperature, T_{solv} , of TMT-8Cr-10Al. (a–d) SEM-BSE micrographs after annealing at 1040, 1050, 1060, and 1070 °C for 95 h followed by water quenching, showing the progressive dissolution of the precipitates with increasing temperature. (e) Quantitative evolution of the volume fraction of precipitates, determined from image-based area fraction measurements of the corresponding microstructures in (a–d). (f) Schematic illustration of the difference between T_{ord} and T_{solv} in the TMT-8Cr-10Al alloy.

4.2 Initial Microstructure for Creep Investigation

4.2.1 TMT-8Cr-10Al

The initial microstructure for creep investigation is shown in Figure 11. The EBSD-IPF map (Figure 11a) reveals a random distribution of grain orientations, indicating the absence of a pronounced crystallographic texture. The alloy exhibits a coarse-grained morphology with an

average grain size exceeding 200 μm , which is comparable to the specimen dimensions used for creep testing (see Section 3.3.1).

The low-magnification SEM-BSE micrograph in Figure 11b confirms the coarse-grained microstructure and shows a limited number of pores, which are attributed to the Kirkendall effect during homogenization of the dendritic cast structure [102]. At higher magnification, as shown in Figure 11c, a two-phase microstructure is clearly observed, consisting of a continuous A2 matrix and dispersed B2 precipitates. The observed phase contrast between A2 and B2 is consistent with the compositional partitioning reported previously [13,25,98]. In addition, a small number of dark particles, marked by white arrows in Figure 11c, are observed at grain boundaries. These particles, with a volume fraction below 0.1%, were previously identified as Ti-rich nitrides originating from N contamination during arc melting and homogenization [25].

This initial microstructural condition provides the basis for the creep behavior discussed in the following sections. The coarse-grained microstructure implies that grain boundary-mediated deformation processes are not expected to significantly contribute under the present creep conditions, consistent with the framework introduced in Section 2.2.1.

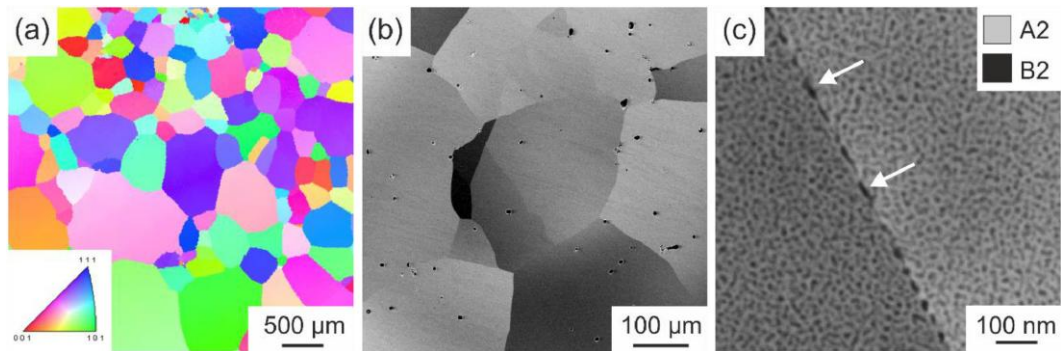


Figure 11. Initial microstructure of TMT-8Cr-10Al after homogenization. (a) EBSD-IPF map showing a coarse-grained structure with random grain orientations. (b) SEM-BSE micrograph illustrating the grain morphology and the presence of pores. (c) High-magnification SEM-BSE image showing the A2 matrix and B2 precipitates. Adapted from Ref. [98].

4.2.2 Monolithic A2 Reference Alloy

The monolithic A2 reference alloy was produced by arc melting followed by homogenization, as described in Section 3.1. The alloy was designed to represent the matrix phase of the A2+B2 microstructure and serves as a reference for evaluating the precipitation strengthening contribution to creep resistance.

The initial homogenized and furnace-cooled microstructure is shown in Figure 12. The low-magnification SEM-BSE image in Figure 12a reveals a coarse-grained microstructure with

equiaxed grains and a limited number of pores. At higher magnification, as shown in Figure 12b, a homogeneous contrast is observed without any indication of second-phase particles or ordered domains, suggesting a single-phase microstructure.

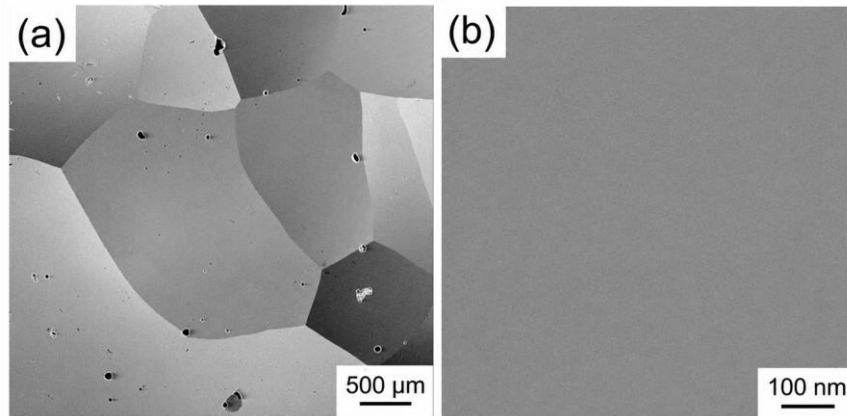


Figure 12. Initial microstructure of the monolithic A2 reference alloy after homogenization at 1600 °C for 72 h followed by furnace cooling. (a) Low-magnification SEM-BSE image showing a coarse-grained microstructure with equiaxed grains and a small number of pores. (b) High-magnification SEM-BSE image indicating a homogeneous contrast without observable second phases.

The phase constitution was further examined by XRD, as shown in Figure 13a. All diffraction peaks can be indexed to a BCC crystal structure. No superlattice peaks associated with the ordered B2 phase are detected within the resolution limit of the measurement. However, it should be noted that excluding B2 order and confirming disordered A2 by XRD alone is challenging due to the weak intensity of superlattice peaks [44].

To further assess the possible presence of ordering reactions, DSC measurements were performed on the monolithic A2 alloy at three different heating rates, as shown in Figure 13b. The DSC curves exhibit no distinct λ -shaped thermal peak over the investigated temperature range. The λ -shaped features are typically associated with order-disorder transformations, as observed for B2 ordering in related alloy systems [13]. The absence of a λ -shaped peak therefore indicates that no long-range chemical ordering occurs within the investigated temperature range. The DSC curves exhibit a broad thermal feature. Its magnitude appears to exceed the Dulong-Petit limit of 3R for the lattice heat capacity, which represents the classical high-temperature limit of the lattice heat capacity per mole of atoms. This indicates that the broad feature should not be interpreted as a simple equilibrium heat-capacity anomaly associated with long-range ordering. It may instead arise from baseline effects, relaxation processes, or other non-ideal thermal contributions during heating. Since no distinct λ -shaped peak is observed, this broad feature is not used to define an ordering temperature. Together

with the XRD results, this provides strong evidence that the monolithic A2 alloy remains disordered and exhibits a single-phase A2 structure.

The composition measured by SEM-EDS is summarized in Table 2 and is consistent with the matrix composition of the A2+B2 TMT-8Cr-10Al alloy previously determined by APT [25]. The monolithic A2 alloy therefore provides a representative approximation of the matrix chemistry for the creep comparison in Section 4.6.

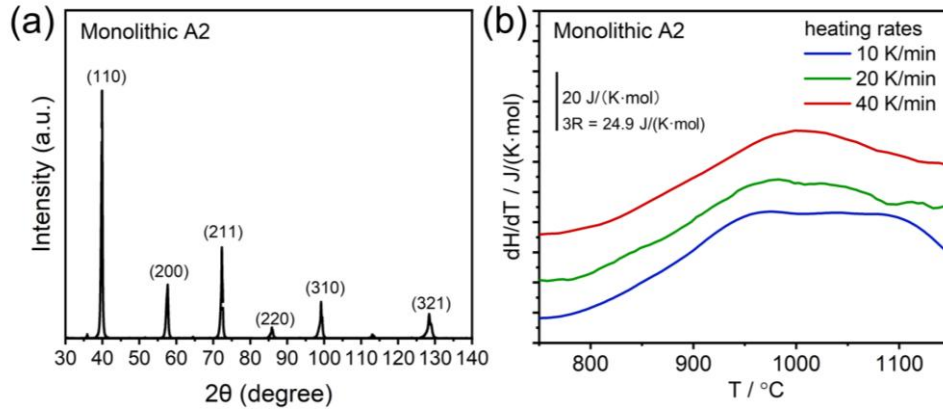


Figure 13. (a) X-ray diffraction pattern of the homogenized and furnace-cooled monolithic A2 reference alloy. (b) DSC curves of the monolithic A2 alloy tested at heating rates of 10, 20, and 40 K/min. The curves are vertically shifted for clarity. The vertical scale bar indicates 20 J/(K·mol), and the Dulong-Petit value is $3R = 24.9 \text{ J/(K·mol)}$. No λ -shaped thermal peak associated with an order-disorder transformation is observed, indicating the absence of B2 ordering over the investigated temperature range.

4.3 High-Temperature Creep Behavior of TMT-8Cr-10Al

4.3.1 Creep Performance at 1000–1060 °C

To evaluate the high-temperature deformation behavior of the TMT-8Cr-10Al alloy, creep tests at 1000 °C and 125 MPa are first considered as a reference condition below T_{solv} . This temperature corresponds to approximately $0.95 T_{\text{ord}}$ and $0.94 T_{\text{solv}}$ and lies within the regime where the A2+B2 two-phase microstructure remains thermally stable even after long-time exposure [25].

As shown in Figure 14a, the strain-time curves at 1000 °C and 125 MPa exhibit a well-defined transient creep regime followed by a gradual stabilization of the strain rate. Although minor variations are observed in the transient regime, the strain rate decreases with increasing strain, indicating strain hardening associated with dislocation-mediated deformation. After the initial transient regime, the interrupted tests converge to similar strain rates of

approximately $2.5 \times 10^{-9} \text{ s}^{-1}$. This consistent response across interrupted tests confirms the reproducibility of creep behavior under these conditions.

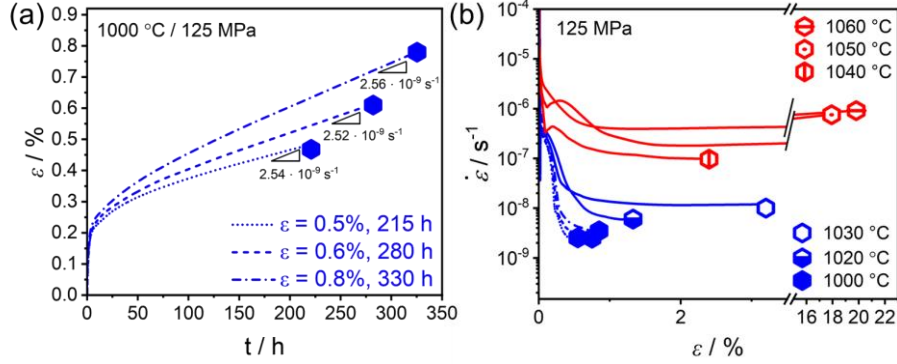


Figure 14. Creep behavior of TMT-8Cr-10Al at 125 MPa. (a) Creep strain ϵ as a function of time t at 1000 °C for interrupted tests with final strains of 0.5, 0.6, and 0.8%. (b) Logarithmic plot of $\dot{\epsilon}$ vs. ϵ at different temperatures. Open symbols indicate the endpoints of individual tests. The minimum creep rate $\dot{\epsilon}_{\min}$ is used as the characteristic creep-rate parameter throughout the analysis. Adapted from Ref. [98].

The temperature dependence of creep behavior is summarized in Figure 14b. The interrupted tests at 1000 °C are shown as blue dashed/dotted lines, while continuous creep tests at 1020, 1030, 1040, 1050, and 1060 °C are plotted as solid lines with open hexagons to indicate the endpoints of the individual tests. As summarized in Table 3, $\dot{\epsilon}_{\min}$ increases with increasing temperature and exhibits a pronounced, nearly an order-of-magnitude increase between 1030 and 1040 °C, despite a temperature difference of only 10 K.

Based on this behavior, two temperature regimes can be distinguished. For $T \leq 1030 \text{ °C}$, $\dot{\epsilon}_{\min}$ remains in the range of 10^{-9} - 10^{-8} s^{-1} , corresponding to a high creep resistance (HCR) regime. For $T \geq 1040 \text{ °C}$, $\dot{\epsilon}_{\min}$ increases to 10^{-7} s^{-1} or higher, corresponding to a low creep resistance (LCR) regime. This transition occurs as the temperature approaches T_{solv} , where a reduction in precipitate stability leads to decreased precipitate strengthening, as discussed in Sections 2.1.3 and 2.2.5.

In the following analysis, $\dot{\epsilon}_{\min}$ is used as the characteristic creep-rate parameter for all creep tests. As discussed in Section 2.2.1, $\dot{\epsilon}_{\min}$ and $\dot{\epsilon}_{\text{ss}}$ are conceptually different. In the present creep curves, however, the strain rate generally reaches a minimum and then remains nearly constant over a measurable strain interval. Under these conditions, $\dot{\epsilon}_{\min}$ is regarded as representative of $\dot{\epsilon}_{\text{ss}}$ within the experimental resolution. The tests at 1000 °C represent a special case because the extremely low creep rates limit the experimentally accessible strain range, as accumulating approximately 5% plastic strain would require test durations approaching 200 days at a typical creep rate of $\sim 2.5 \times 10^{-9} \text{ s}^{-1}$. A formal steady-state regime

over a large plastic-strain interval cannot be demonstrated within reasonable testing times. Nevertheless, the three interrupted tests converge to very similar endpoint strain rates despite different interruption strains and test durations. These values are therefore considered reliable estimates of $\dot{\epsilon}_{\min}$, although a fully developed steady-state regime is not explicitly claimed for the 1000 °C tests.

Table 3. $\dot{\epsilon}_{\min}$ of TMT-8Cr-10Al measured at 125 MPa for different temperatures. Values represent averages of repeated tests where available; otherwise, representative values from individual tests are reported.

σ / MPa	125					
T / °C	1000	1020	1030	1040	1050	1060
$\dot{\epsilon}_{\min}$ / s ⁻¹	$2.5 \cdot 10^{-9}$	$7.1 \cdot 10^{-9}$	$1.9 \cdot 10^{-8}$	$9.6 \cdot 10^{-8}$	$1.6 \cdot 10^{-7}$	$4.1 \cdot 10^{-7}$

4.3.2 Apparent Stress Exponent and Activation Energy

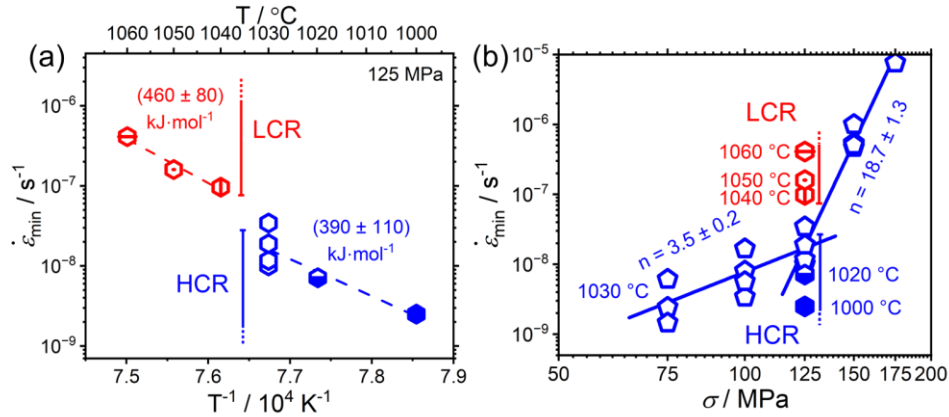


Figure 15. Temperature and stress dependence of the $\dot{\epsilon}_{\min}$ of TMT-8Cr-10Al. (a) Arrhenius plot for 125 MPa. HCR data correspond to temperatures of 1000, 1020 and 1030 °C, and LCR to 1040, 1050 and 1060 °C. (b) Norton plot. Adapted from Ref. [98].

Figure 15a shows the Arrhenius plot obtained from $\dot{\epsilon}_{\min}$ at an applied stress of 125 MPa. The Q_{app} value was determined using the standard power-law creep formalism introduced in Section 2.2.1. Two distinct linear temperature regimes are observed, corresponding to the HCR and LCR regimes defined in Section 4.3.1. Linear fitting yields $Q_{\text{app}} = (390 \pm 110) \text{ kJ/mol}$ for the HCR regime ($T \leq 1030 \text{ °C}$) and $Q_{\text{app}} = (460 \pm 80) \text{ kJ/mol}$ for the LCR regime ($T \geq 1040 \text{ °C}$). As discussed in Section 2.2.1, Q_{app} does not necessarily represent a single elementary diffusion process, especially in complex multi-component single-phase or multi-phase materials [75]. In the present case, TMT-8Cr-10Al contains five principal elements and

exhibits a two-phase A2+B2 microstructure. The Q_{app} value should be interpreted as an effective parameter reflecting the combined contribution of multiple thermally activated processes, including matrix deformation and concurrent microstructural evolution.

The n_{app} values were determined from creep tests conducted at 1030 °C under varying applied stresses of 75, 100, 125, 150 and 175 MPa. Figure 15b shows the corresponding Norton plot based on $\dot{\epsilon}_{min}$. The low stress regime between 75 and 125 MPa yields $n_{app} = (3.5 \pm 0.2)$, whereas the high-stress regime between 125 and 175 MPa yields $n_{app} = (18.7 \pm 1.3)$. As mentioned in Sections 2.2.2 and 2.2.3, such a strong increase in the n_{app} value is commonly reported for precipitation-strengthened alloys when the applied stress is used directly instead of the effective stress. The lower value is consistent with creep controlled mainly by dislocation climb in the matrix, whereas the higher value at elevated stress indicates a change in creep response with increasing stress. The present data therefore suggest that the applied stress does not directly represent the effective driving stress for creep in TMT-8Cr-10Al and the creep behavior at 1030 °C cannot be described by a single stress exponent over the investigated stress range. Its interpretation requires consideration of the effective stress concept introduced in Section 2.2.2, together with the dislocation-precipitate interactions and microstructural evolution discussed in Sections 2.2.4 and 2.2.5.

In contrast to single-crystalline $\gamma+\gamma'$ Ni-based superalloys, where creep behavior is often strongly influenced by crystallographic orientation, channel geometry, rafting, and interfacial dislocation processes, BCC-based refractory alloys may exhibit a different degree of orientation sensitivity. The crystallographic slip modes reported for BCC and B2 refractory alloys provide useful context for this comparison. As mentioned in Section 2.1.2, plastic deformation in BCC alloys is primarily accommodated by $1/2\langle 111 \rangle$ dislocations gliding on $\{110\}$, $\{112\}$, and $\{123\}$ planes. At ambient temperature, similar deformation behavior has been reported for several RHEA systems with different phase constitutions. For example, deformation in the BCC+B2 alloy $AlMo_{0.5}NbTa_{0.5}TiZr$ is governed by dislocation-mediated plasticity, while TEM studies on B2-ordered refractory alloys have identified $\langle 111 \rangle$ as the dominant slip direction, with slip occurring mainly on $\{110\}$ planes and additionally on $\{112\}$ planes in specific orientations [103,104]. These observations indicate that multiple slip modes can be activated in BCC-based refractory alloys at ambient temperature, which may contribute to a more homogeneous plastic response and reduced crystallographic anisotropy. Similar dislocation-mediated deformation mechanisms have also been reported at elevated temperatures in BCC+B2 refractory complex concentrated alloys [105], indicating that the crystallographic framework for slip remains relevant beyond ambient-temperature deformation. Although the present creep experiments were conducted at 1030 °C, the relatively uniform creep response observed here is consistent with this expectation and with the experimentally reported high-temperature creep behavior of single-phase BCC RHEAs

such as HfNbTaTiZr [15,16]. The consistency of repeated tests at 1030 °C across the full stress range further confirms the reproducibility and reliability of the measured creep data.

4.3.3 Comparison with CMSX-4 and Single-Phase RHEAs

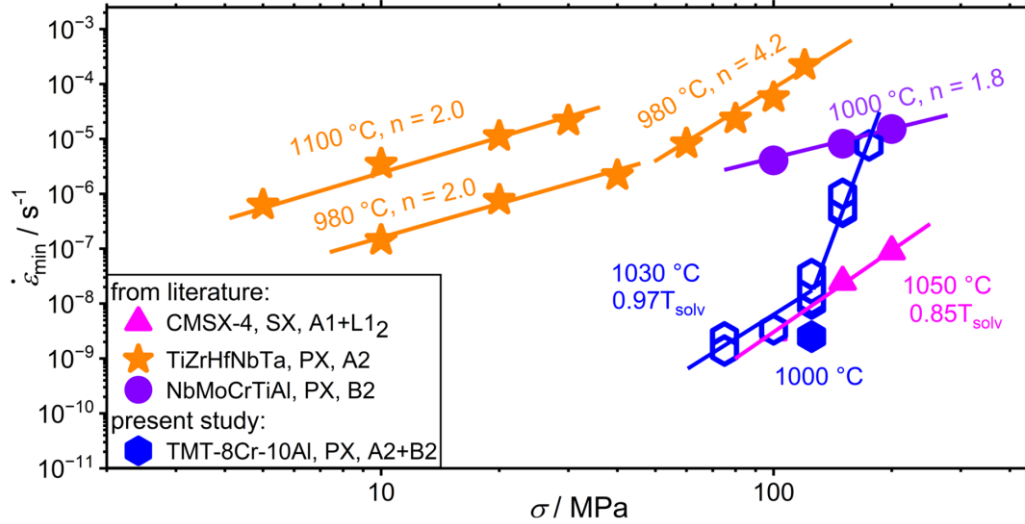


Figure 16. Comparison of creep rate vs. applied stress for the investigated PX, A2+B2 TMT-8Cr-10Al alloy and PX, single-phase B2 NbMoCrTiAl alloy [106], PX, single-phase A2 TiZrHfNbTa [16] as well as for the SX Ni-based superalloy CMSX-4 with A1+L₁₂ microstructure [107,108]. The values for TMT-8Cr-10Al and CMSX-4 are additionally annotated by their testing temperatures relative to the precipitate solvus temperature, T_{solv} , where applicable. Adapted from Ref. [98].

Figure 16 presents creep results for polycrystalline (PX) TMT-8Cr-10Al, which features an A2 matrix with coherent B2 precipitates, in comparison to the single-crystal (SX) Ni-based superalloy CMSX-4 (containing A1 matrix and L₁₂ precipitates), as well as two PX single-phase refractory alloys, namely A2-type TiZrHfNbTa and B2-type NbMoCrTiAl. The $\dot{\epsilon}_{\text{min}}$ values obtained for PX TMT-8Cr-10Al at 1030 °C under applied stresses of 75–125 MPa are comparable to those of SX Ni-based superalloy CMSX-4 tested at 1050 °C.

For a meaningful comparison with CMSX-4, the test temperature must be considered relative to both the precipitate T_{solv} and the solidus temperature T_{solidus} . As discussed in Sections 2.1.3 and 2.2.5, the ratio T/T_{solv} reflects the thermodynamic stability of the strengthening phase, whereas T/T_{solidus} is more closely related to the general importance of diffusion-controlled processes during creep. For TMT-8Cr-10Al, testing at 1030 °C corresponds to approximately $0.97 T_{\text{solv}}$, assuming a precipitate T_{solv} of 1065 °C. In contrast, CMSX-4 tested at 1050 °C corresponds to only about $0.85 T_{\text{solv}}$, based on its L₁₂ T_{solv} of

approximately 1280 °C [109]. This indicates that TMT-8Cr-10Al is tested closer to the dissolution threshold of its strengthening phase. A comparison relative to T_{solidus} leads to the opposite trend. The T_{solidus} of CMSX-4 is reported to be approximately 1339 °C [110], corresponding to a testing condition of about 0.82 T_{solidus} at 1050 °C. For TMT-8Cr-10Al, a T_{solidus} of 2282 °C was estimated based on CALPHAD calculations using Pandat software (CompuTherm LLC) with the RHEA database, resulting in a testing condition of about 0.51 T_{solidus} at 1030 °C. The comparable creep rates observed for the two alloys therefore reflect the combined influence of precipitate stability and diffusion-controlled processes.

When compared to single-phase RHEAs, such as PX A2-type TiZrHfNbTa and B2-type NbMoCrTiAl, the difference in creep resistance is more pronounced. At comparable stress levels and temperature ranges, the $\dot{\epsilon}_{\text{min}}$ of TMT-8Cr-10Al is approximately four orders of magnitude lower than those of the single-phase alloys. As outlined in Sections 2.2.2 to 2.2.5, this difference is consistent with the strong strengthening effect of a stable two-phase microstructure containing coherent precipitates. In addition, the reduced creep resistance of the TiZrHfNbTa and NbMoCrTiAl alloys is associated with microstructural decomposition during creep, transforming from a solid solution into a multiphase intermetallic structure [106]. The significantly lower creep rates observed for TMT-8Cr-10Al compared to the single-phase alloys highlight the role of the two-phase microstructure in reducing the creep rate relative to single-phase solid-solution alloys.

4.4 Lattice Misfit between A2 Matrix and B2 Precipitates

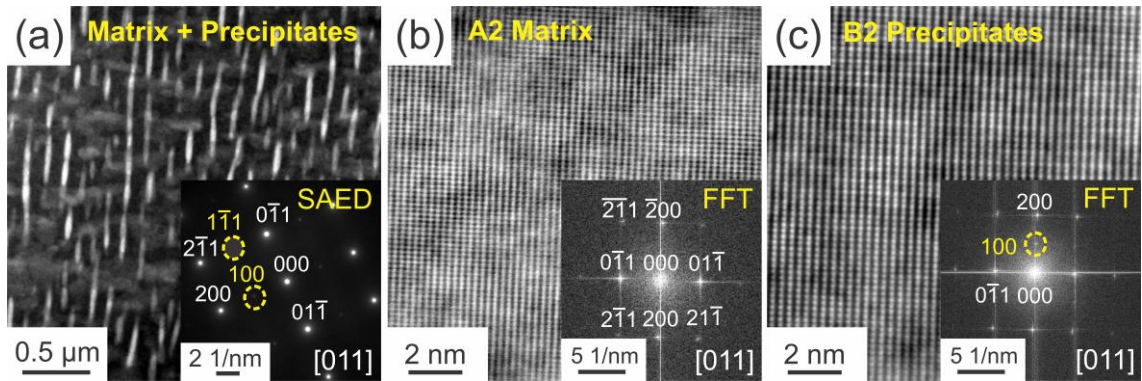


Figure 17. TEM results on TMT-8Cr-10Al alloy after heat treatment at 1000 °C for 1000 h. (a) TEM-DF micrograph showing bright contrast B2 precipitates uniformly distributed throughout the matrix. The micrograph was acquired using a superlattice reflection in the corresponding TEM-SAED pattern along a $\langle 011 \rangle$ zone axis from the same region (inset). (b) Atomic-resolution STEM-HAADF micrograph of the matrix with its corresponding FFT in the inset. (c) Atomic-resolution STEM-HAADF micrograph of a B2 precipitate with its corresponding FFT in the inset. Adapted from Ref. [98].

As discussed in Section 2.2.5, the rafting behavior of coherent two-phase microstructures is governed by the sign of the lattice misfit (positive or negative), together with the loading mode (tension or compression). Determining the sign of the lattice misfit between the A2 matrix and B2 precipitates is therefore essential for interpreting the creep-induced rafting behavior of TMT-8Cr-10Al. To this end, TEM analysis was conducted on a specimen aged at 1000 °C for 1000 h. This condition lies within the HCR range (1000–1030 °C) in Section 4.3.1 and represents a creep-relevant microstructural state with high thermal stability.

Figure 17a shows a TEM-DF image with bright B2 precipitates uniformly distributed within the A2 matrix. The inset TEM-SAED pattern, acquired along the $\langle 011 \rangle$ zone axis, exhibits weak but distinct $\{100\}$ superlattice spots, confirming the presence of long-range ordering in the B2 phase. The atomic-resolution STEM-HAADF images shown in Figures 17b–c further confirm the distinct crystal structures of the A2 matrix and B2 precipitates and indicate a coherent orientation relationship of $\{100\}_{A2} \parallel \{100\}_{B2}$ and $\langle 001 \rangle_{A2} \parallel \langle 001 \rangle_{B2}$. The lattice parameters were determined from FFT analysis at multiple locations, yielding average values of 0.323 ± 0.001 nm for a_{A2} and 0.325 ± 0.001 nm for a_{B2} . The lattice misfit, calculated according to Eq. (2.19), is $(+0.6 \pm 0.4) \%$. The positive sign indicates that the B2 precipitates possess a slightly larger lattice parameter than the surrounding A2 matrix.

Although localized strain fields or elastic relaxation may introduce some uncertainty into the absolute values, the consistently positive misfit obtained from multiple regions provides a reliable basis for interpreting the creep-induced morphological evolution discussed in the following section. In particular, the positive lattice misfit establishes the expected framework for assessing directional coarsening under compressive creep conditions, following the principles outlined in Section 2.2.5.

4.5 Microstructural Evolution of TMT-8Cr-10Al after Creep

4.5.1 B2 Precipitate Coarsening

The microstructural evolution of the B2 precipitates in TMT-8Cr-10Al was systematically investigated under creep conditions at 1000 °C and 125 MPa at different strain levels. Figures 18a–c show SEM-BSE micrographs after creep tests interrupted at total strains of 0.5, 0.6, and 0.8 %, corresponding to creep durations of 215, 280, and 330 h, respectively. To distinguish the effect of mechanical loading from purely thermal exposure, reference specimens were aged at 1000 °C for the same durations without applied stress, as shown in Figures 18d–f.

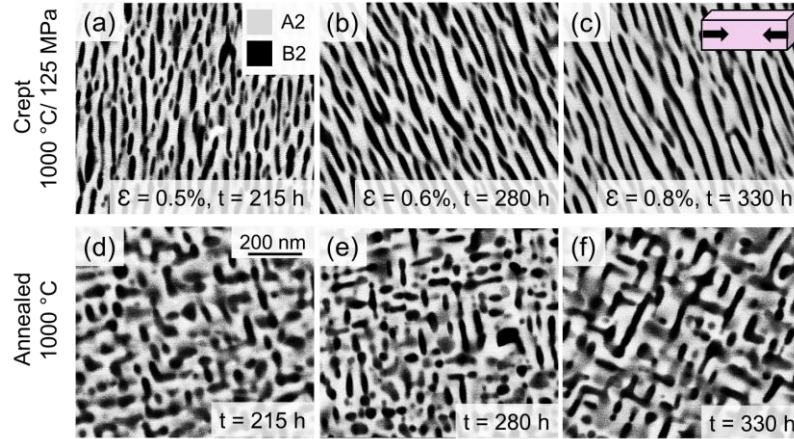


Figure 18. SEM-BSE micrographs showing the microstructure evolution of TMT-8Cr-10Al at 1000 °C. (a–c) Specimens crept at 125 MPa and interrupted at total strains of (a) 0.5%, (b) 0.6%, and (c) 0.8%, corresponding to durations of 215, 280, and 330 h, respectively. (d–f) Stress-free reference specimens aged at 1000 °C for (d) 215 h, (e) 280 h, and (f) 330 h. Adapted from Ref. [98].

In both the creep-exposed and thermally aged conditions, the B2 precipitates increase in size with time. However, the precipitates grow more rapidly under creep conditions and begin to show directional elongation. In contrast, the aged specimens show coarsening without a pronounced preferred orientation. As discussed in Section 2.2.5, this difference indicates that precipitate evolution during creep cannot be described solely by diffusion-controlled coarsening, but also includes a stress-assisted contribution to the morphological development.

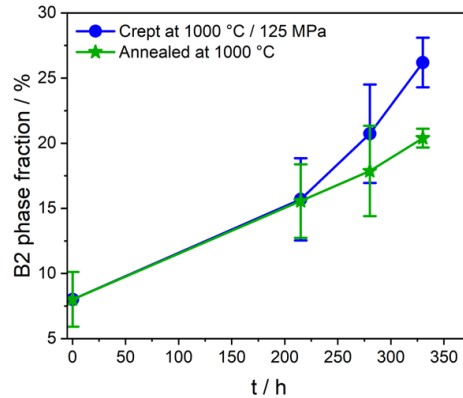


Figure 19. Apparent B2 phase fraction of TMT-8Cr-10Al at 1000 °C as a function of time during creep at 125 MPa and stress-free thermal annealing. The values were determined by image analysis of SEM-BSE micrographs. The error bars represent the combined uncertainty arising from image threshold selection and statistical variation of the segmentation procedure. Adapted from Ref. [98].

The B2 phase fraction was estimated from SEM-BSE micrographs using the image-analysis procedure described in Section 3.2.9, as summarized in Figure 19. An apparent increase in phase fraction with time is observed for both creep and aging conditions. However, it should be noted that the B2 precipitate size remains close to the resolution limit of SEM-BSE imaging over the investigated time range. As a result, the threshold-based segmentation is highly sensitive to image contrast and the selection of threshold parameters, which are not uniquely defined. To account for this, the uncertainty in the phase fraction was estimated by varying the threshold parameters within a reasonable range. The resulting error bars shown in Figure 19 therefore reflect both the statistical variation of the analysis and the uncertainty associated with threshold selection.

In addition, the analysis is based on two-dimensional projections of a three-dimensional microstructure. Overlap of precipitates along the thickness direction may lead to an overestimation of the phase fraction. Furthermore, differences in precipitate morphology between creep and aging conditions may affect the segmentation. The elongated precipitates formed under creep conditions are more likely to be detected as continuous regions, whereas smaller and more equiaxed precipitates in aged samples may be partially unresolved. Considering these limitations, the measured phase fractions and their apparent evolution should be considered semi-quantitative. Within the estimated uncertainty, no definitive trend in phase fraction evolution can be established.

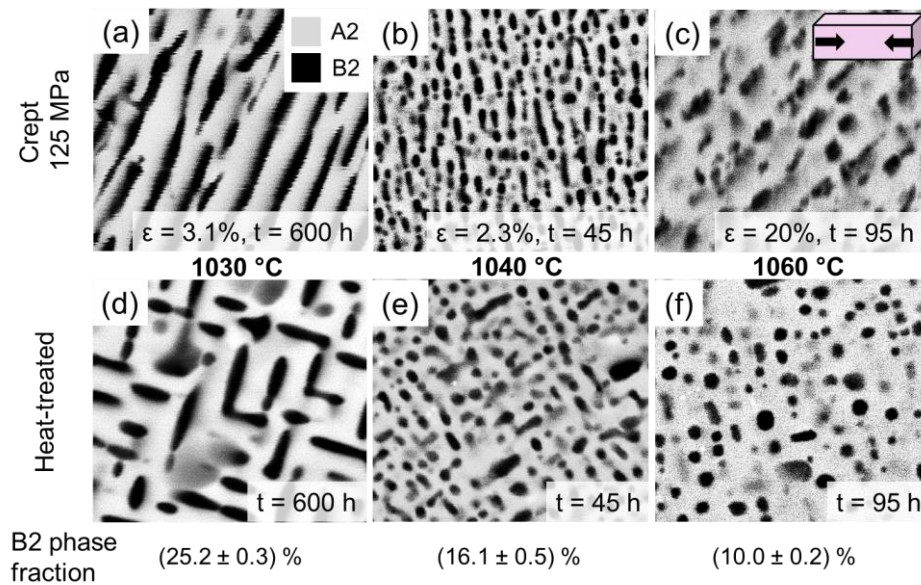


Figure 20. Microstructure evolution of TMT-8Cr-10Al after creep tests and heat treatments. (a) 1030 °C, 125 MPa, 3.1 %. (b) 1040 °C, 125 MPa, 2.3 %. (c) 1060 °C, 125 MPa, 20 %. (d) 1030 °C, 600 h. (e) 1040 °C, 45 h. (f) 1060 °C, 95 h and (g) 1070 °C, 95 h. The same magnification was used in all micrographs. Adapted from Ref. [98].

The effect of temperature on precipitate coarsening was further examined at 1030–1060 °C under a constant compressive stress of 125 MPa. Representative SEM-BSE micrographs of creep-deformed specimens are presented in Figures 20a–c, along with stress-free reference specimens aged for equivalent times and followed by water quenching shown in Figures 20d–f. As discussed in Sections 2.2.5 and 4.1, increasing temperature is expected to promote both coarsening and progressive destabilization of the B2 precipitates as the T_{solv} is approached. At 1030 °C, the creep-exposed microstructure remains precipitate-rich and shows elongated B2 domains, closely resembling the growth regime established at 1000 °C. At 1040 °C, the precipitates become notably finer and more fragmented. At 1060 °C, the B2 phase fraction is reduced to approximately 10%, and only a limited number of precipitates remain after 95 h of creep exposure. This evolution is consistent with the approach to the T_{solv} identified in Section 4.1, where the dissolution of B2 precipitates is expected near the solvus range of 1060–1070 °C.

It should be emphasized that the creep specimens were furnace cooled after testing, whereas the heat-treated reference specimens were quenched. As a result, some degree of re-nucleation or precipitate growth during cooling cannot be excluded for the creep-tested samples, potentially altering the final microstructural conditions. Nevertheless, the systematic reduction in B2 fraction with increasing temperature indicates that the observed trend is primarily governed by the intrinsic thermal stability of the precipitates rather than by post-test cooling effects.

4.5.2 Stress-Induced Rafting

The stress-free aged specimens shown in Figures 18d–f exhibit no pronounced preferred precipitate alignment, whereas the crept specimens in Figures 18a–c display a clear alignment of B2 precipitates relative to the loading direction. This demonstrates that the applied stress induces a directional morphological evolution of the precipitates. As discussed in Section 2.2.5, such behavior is characteristic of stress-assisted directional coarsening expected for a two-phase microstructure with a positive lattice misfit under compressive loading. In classical studies of single-crystal Ni-based superalloys [87,95,111–113], rafting is typically examined in specimens with a well-defined $\langle 100 \rangle$ orientation relative to the load axis. In the present polycrystalline alloy, however, the resulting rafting morphology is additionally influenced by the crystallographic orientation of each grain with respect to the applied loading axis.

To establish the correlation between grain orientation and rafting morphology, EBSD-IPF maps from SEM-EBSD were combined with SEM-BSE micrographs acquired from the same regions. Figures 21a–b show representative results for creep tests interrupted at total strains of 0.5 and 0.8 %, respectively. The selected grains, labeled 1–4, cover a range of crystallographic orientations. The corresponding precipitate morphologies shown in

Figures 21 a1–a4 and Figures 21 b1–b4 reveal a clear dependence of rafting direction on grain orientation.

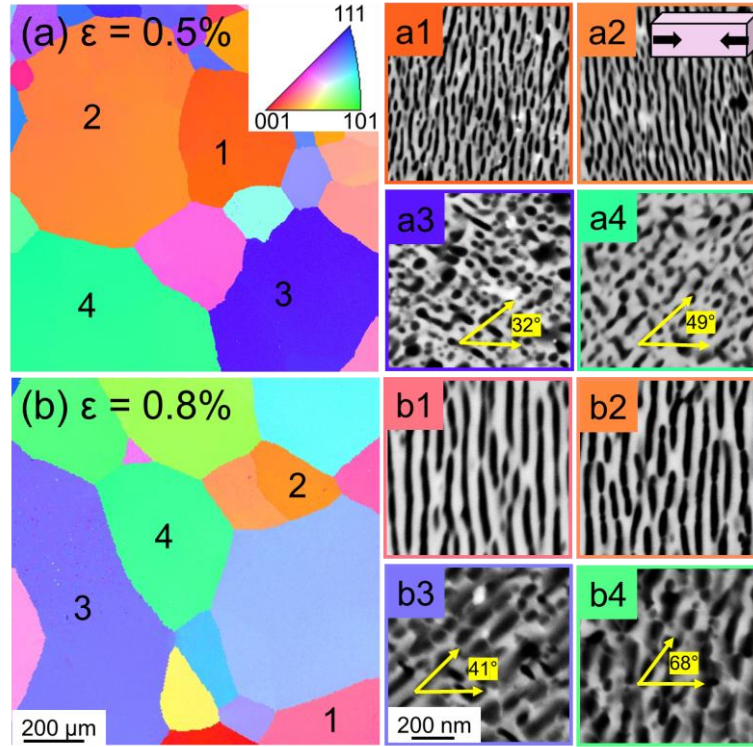


Figure 21. EBSD-IPF maps and corresponding B2 rafting directions in individual grains after creep at 1000 °C and 125 MPa to total strains of (a) 0.5% and (b) 0.8%. Grains labeled 1–4 illustrate the dependence of rafting morphology on crystallographic orientation relative to the loading axis. Adapted from Ref. [98].

Two important observations can be drawn from these results. First, the rafting direction is not unique, but varies systematically with grain orientation. In grains where a $\langle 100 \rangle$ axis is aligned approximately parallel to the compression direction, the precipitate rafts align perpendicular to the loading axis, consistent with the N-type rafting expected for a positive lattice misfit under compressive loading, as established in Section 4.4 and discussed in Section 2.2.5. Second, in grains deviating from $\langle 100 \rangle$ orientations, the rafting direction tilts away from a perfectly perpendicular alignment, leading to both positive and negative inclination angles of approximately 45–70° relative to the compression axis. Such behavior mirrors observations in polycrystalline Co-based alloys [94,95], where rafting perpendicular to stress is exclusive to $\langle 100 \rangle$ grains, while other orientations produce oblique raft patterns.

Overall, the rafting morphology in TMT-8Cr-10Al is governed by the combined effects of positive lattice misfit and grain orientation. Under compressive loading, N-type rafting

dominates in favorably oriented grains, whereas less favorable orientations produce inclined raft patterns. This grain-orientation dependence distinguishes the present polycrystalline alloy from oriented single crystals and provides the basis for selecting $\langle 100 \rangle$ -oriented grains for the quasi-three-dimensional analysis below and for the TEM analysis in Chapter 5.

4.5.3 Quasi-Three-Dimensional Representation of B2 Precipitates

While the SEM-BSE observations provide essential information on the post-creep morphology of the B2 precipitates, they are inherently limited to two-dimensional projections and therefore cannot fully resolve the spatial connectivity, thickness, or true geometry of the rafted structures. To obtain additional insight into the three-dimensional morphology, a specimen crept at 1030 °C and 150 MPa to a total strain of 7.8 % was selected, since this condition exhibits well-developed rafting.

A grain with a loading direction close to $\langle 100 \rangle$ was identified using SEM-EBSD, as shown in Figure 22a. Site-specific FIB milling was then performed on this grain (Figure 22b), and a micropillar was fabricated to expose clean and orthogonal observation planes. Three orthogonal perspectives, namely the top, lateral, and front faces, were imaged using an in-column detector (ICD), as shown in Figures 22c–e. Based on these orthogonal views, a quasi-3D representation of the B2 precipitate morphology was constructed (Figure 22f). It should be noted that this representation is derived from two-dimensional observations on three orthogonal surfaces and does not constitute a full 3D reconstruction.

The observations indicate that the B2 precipitates form a plate-like geometry with a pronounced planar aspect ratio and that they are aligned perpendicular to the loading direction. This is consistent with the N-type rafting tendency expected for compressive loading in a system with positive lattice misfit, as established in Section 4.4 and discussed in Section 2.2.5. On the top and lateral faces shown in Figure 22c and Figure 22d, the precipitates appear as elongated and nearly continuous plates, whereas on the front face shown in Figure 22e, their cross-sections are close to circular. This combination indicates that the precipitates are laterally extended in two directions while remaining comparatively thin in the third direction. These observations indicate a rafted B2 structure composed of plate-like precipitates.

To quantify this morphology, the projected major and minor dimensions of individual B2 precipitates were measured from the orthogonal ICD micrographs. The apparent aspect ratio was defined as the ratio between the projected major axis a_p and the projected minor axis b_p . The elongated precipitates observed on the top and lateral faces exhibit average apparent aspect ratios of 4.8 ± 2.4 and 5.1 ± 2.4 , respectively. When both faces are considered together, the average apparent aspect ratio is 4.9 ± 2.4 . In contrast, the precipitate cross-sections on the front face show a much lower apparent aspect ratio of 1.7 ± 0.8 . These values quantitatively

support the plate-like character of the B2 precipitates and underpin the morphological interpretation derived from the orthogonal views. The values should be regarded as apparent aspect ratios because they were obtained from two-dimensional projections of the orthogonal surfaces.

Furthermore, the apparent segmentation occasionally observed in 2D SEM-BSE images is a projection artifact rather than evidence of precipitate discontinuity. The cut-through features marked by red arrows in Figure 22e correspond to narrow gaps between adjacent rafted plates that are impinged and undergoing coagulation. The quasi-three-dimensional analysis thus supports the interpretation that creep induces a connected, plate-like raft morphology in $\langle 100 \rangle$ -oriented grains.

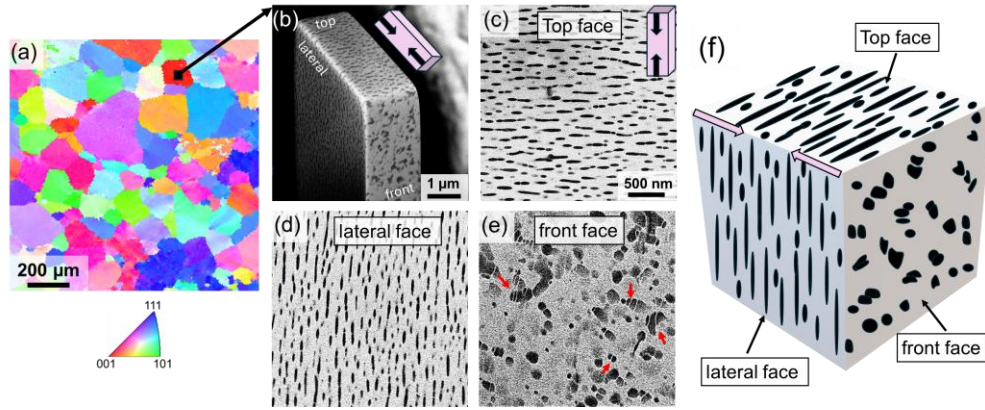


Figure 22. Quasi-three-dimensional representation of rafted B2 precipitates in TMT-8Cr-10Al after creep at 1030 °C and 150 MPa to a total strain of 7.8%. (a) EBSD-IPF map highlighting the selected $\langle 100 \rangle$ -oriented grain. (b) Site-specific FIB preparation of the micropillar. (c–e) In-column detector micrographs acquired from the top, lateral, and front faces, respectively. (f) Quasi-three-dimensional representation of the rafted B2 precipitate morphology based on the three orthogonal views. Adapted from Ref. [114].

4.6 Strengthening Contribution of B2 Precipitates

The LBR framework introduced in Section 2.2.3 is applied here for the first time to estimate the effective strengthening contribution of B2 precipitates to the creep resistance of A2+B2 TMT-8Cr-10Al. Consistent with the effective-stress and dislocation-precipitate interaction concepts introduced in Sections 2.2.2 and 2.2.4, plastic deformation is assumed to be primarily accommodated in the A2 matrix, while the B2 precipitates provide an additional resistance to dislocation motion. Under this assumption, the effective stress available for matrix-controlled creep in the A2+B2 alloy is:

$$\sigma_{\text{eff}} = \sigma - \sigma_{\text{B2}} \quad (4.1)$$

where σ_{eff} is the effective stress acting in the A2 matrix, and σ_{B2} represents the effective stress contribution associated with the B2 precipitates.

Using the effective-stress creep relation introduced in Eq. (2.6), the creep response can therefore be expressed locally as:

$$\dot{\epsilon}_{\text{min}} = B (\sigma - \sigma_{\text{B2}})^{n_{\text{A2}}} \exp\left(-\frac{Q_{\text{A2}}}{RT}\right) \quad (4.2)$$

where n_{A2} and Q_{A2} denote the apparent stress exponent and the apparent activation enthalpy of the A2 matrix, respectively, and B is a material constant.

The formulation introduced in Eq. (4.2) assumes a constant matrix stress exponent n_{A2} . However, the experimental results presented in Section 4.3 indicate a clear increase in the apparent stress exponent with applied stress, showing that the creep response cannot be described by a single constant stress exponent over the entire stress range. Therefore, the sinh-type formulation introduced in Eq. (2.9) is applied here to account for the stress dependence of the matrix creep response. Within this framework, the creep data are re-plotted in the form $\dot{\epsilon}_{\text{min}}^{1/n_{\text{A2}}(\sigma)}$ as a function of the applied stress σ , which enables a direct comparison between the A2 matrix response and the A2+B2 precipitation-strengthened alloys with the LBR framework.

A suitable matrix reference is required for LBR analysis. In traditional $\gamma+\gamma'$ Ni- or Co-based superalloys, this is ideally achieved by comparing the two-phase alloy with a single-phase matrix alloy matching the γ matrix composition. In the present study, two matrix-reference approaches are considered. The first approach uses the creep response of the nominal single-phase A2 condition at 1070 °C, extrapolated to 1030 °C. The second is the creep response of a monolithic A2 alloy with a composition representative of the A2 matrix in the two-phase microstructure, tested directly at 1030 °C. Together, these two approaches allow the robustness of the extracted strengthening contribution to be assessed.

The use of the single-phase A2 condition at 1070 °C is supported by the precipitate T_{solv} of (1065 ± 5) °C as determined experimentally in Section 4.1, which places 1070 °C within the single-phase A2 field. The measured creep response at 1070 °C can therefore be taken as an approximation of the matrix creep behavior. Its extrapolation to 1030 °C relies on two key assumptions. First, the chemical difference between the nominal alloy composition in the single-phase A2 condition (i.e., the A2 phase tested at 1070 °C) and the actual A2 matrix composition in the two-phase alloy does not significantly affect the minimum creep rate. Second, the apparent activation energy obtained from the temperature-dependent creep data at 125 MPa in the A2+B2 condition also holds for the single-phase A2 condition and allows for the extrapolation of creep data at 1030 °C from 1070 °C.

To estimate the A2 matrix response at 1030 °C, the data measured at 1070 °C were extrapolated using an Arrhenius-type temperature dependence [32]. This extrapolation assumes that the dominant deformation behavior and the apparent activation energy do not change fundamentally between 1030 and 1070 °C. To account for the uncertainty associated with this extrapolation, the two Q_{app} values in Section 4.3.2, $Q_{app} = (390 \pm 110)$ kJ/mol and $Q_{app} = (460 \pm 80)$ kJ/mol, were used to define the uncertainty range of the extrapolated A2 response shown in Figure 23a.

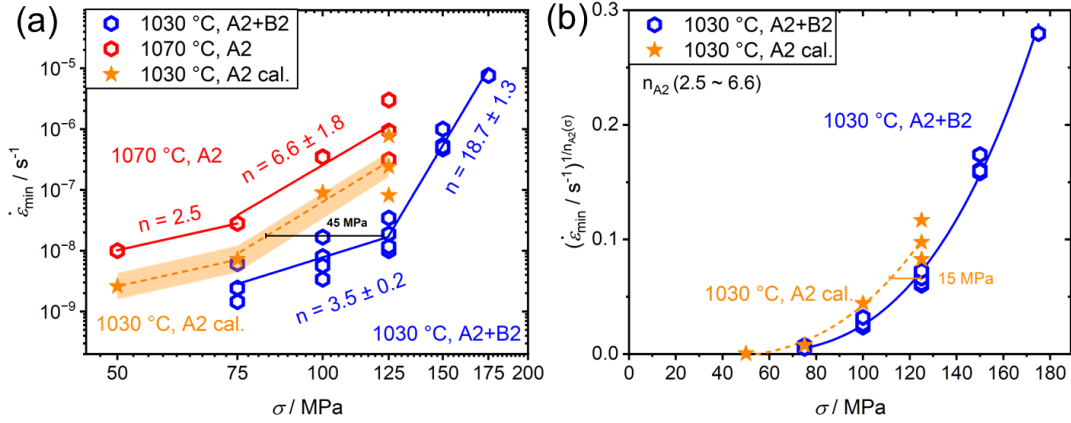


Figure 23. LBR-based estimate of the B2 strengthening contribution using the extrapolated A2 reference. (a) Norton plot of $\dot{\epsilon}_{min}$ as a function of σ for the extrapolated A2 reference and the A2+B2 alloy at 1030 °C. The A2 reference data were recalculated from 1070 °C to 1030 °C using the thermal activation data presented in Figure 15a. The shaded band indicates the uncertainty associated with this recalculation. At $\dot{\epsilon}_{min} = 10^{-8} s^{-1}$, the direct stress offset between the extrapolated A2 reference and the A2+B2 alloy is approximately 45 MPa. (b) LBR plot of $\dot{\epsilon}_{min}^{1/n_{A2}(\sigma)}$ as a function of σ . The horizontal offset between the extrapolated A2 reference and the A2+B2 alloy gives the LBR-derived B2 strengthening contribution σ_{B2} , which is approximately 15 MPa at $\dot{\epsilon}_{min} = 10^{-8} s^{-1}$. Adapted from Ref. [98].

Figure 23a compares the extrapolated A2 reference with the A2+B2 alloy at 1030 °C in a Norton plot. At a $\dot{\epsilon}_{min}$ of $10^{-8} s^{-1}$, the A2+B2 alloy requires an applied stress approximately 45 MPa higher than the extrapolated A2 reference. This value represents the direct stress offset at the selected creep rate. It should not be interpreted as the LBR-derived threshold stress, because it is obtained directly from the Norton plot of $\dot{\epsilon}_{min}$ versus σ .

To estimate the precipitate-related contribution in the LBR framework, the same data are shown in the LBR plot in Figure 23b. In this plot, $\dot{\epsilon}_{min}^{1/n_{A2}(\sigma)}$ is plotted as a function of σ . Here, $n_{A2}(\sigma)$ represents the local apparent stress exponent of the extrapolated A2 reference. It was approximated by interpolation between the apparent slopes of the A2 reference curve, increasing from about 2.5 at 50 MPa to about 6.6 at 125 MPa. Since a stress-dependent local

exponent is used, the extrapolated A2 reference in the LBR plot is not expected to be strictly linear over the full stress interval. In the LBR plot, the horizontal offset between the extrapolated A2 reference and the A2+B2 alloy corresponds to the LBR-derived B2 precipitate strengthening contribution. At $\dot{\epsilon}_{\min} = 10^{-8} \text{ s}^{-1}$, this contribution is approximately 15 MPa. This value is therefore used as the semi-quantitative estimate of the B2 strengthening contribution based on the extrapolated A2 reference. In contrast, the 45 MPa obtained from Figure 23a is retained only as the direct stress offset in the Norton plot at the selected creep rate.

To verify the reliability of the extrapolated A2 matrix reference, a monolithic A2 alloy was investigated directly at 1030 °C. As shown in Section 4.2.2, this alloy provides a reasonable approximation of the A2 matrix composition in the two-phase A2+B2 microstructure and removes the uncertainties associated with temperature extrapolation. The corresponding $\dot{\epsilon}_{\min}$ values measured for the monolithic A2 alloy at 1030 °C between 75 and 125 MPa are shown in Figure 24a, together with the extrapolated A2 reference and the A2+B2 alloy. At 75 MPa, the monolithic A2 alloy exhibits a $\dot{\epsilon}_{\min}$ comparable to that of the extrapolated A2 reference. At higher stresses, the monolithic A2 alloy shows slightly lower $\dot{\epsilon}_{\min}$ values than the extrapolated A2 reference, indicating somewhat higher creep resistance. This deviation is most likely associated with residual compositional differences between the monolithic A2 alloy and the nominal A2 reference used in the extrapolation. A slightly higher content of refractory elements such as Ta and Mo in the monolithic A2 alloy may have contributed to the improved creep resistance.

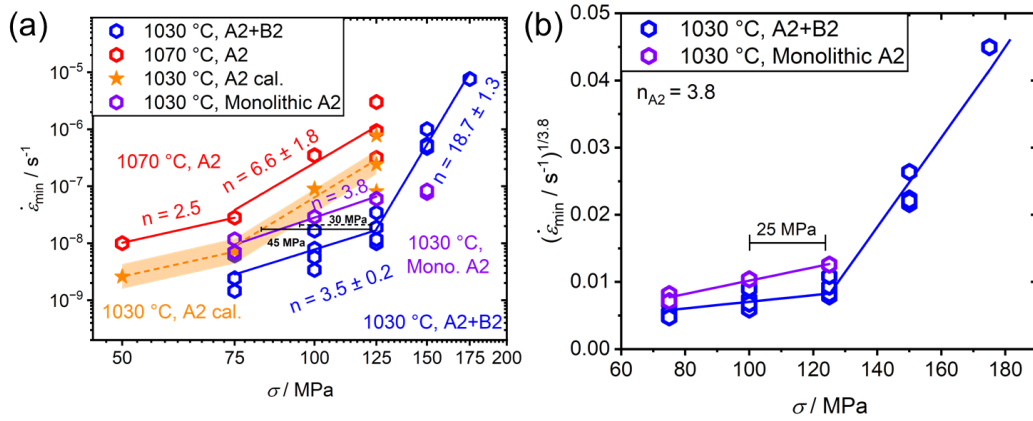


Figure 24. LBR-based estimate of the B2 strengthening contribution using the monolithic A2 alloy. (a) Norton plot of $\dot{\epsilon}_{\min}$ as a function of σ , comparing the A2+B2 alloy, the 1070 °C A2 condition, the extrapolated A2 reference recalculated to 1030 °C, and the monolithic A2 alloy tested directly at 1030 °C. At $\dot{\epsilon}_{\min} = 10^{-8} \text{ s}^{-1}$, the direct stress offset between the monolithic A2 alloy and the A2+B2 alloy is approximately 30 MPa. (b) LBR plot of $\dot{\epsilon}_{\min}^{1/3.8}$ as a function of σ . The horizontal offset between the monolithic A2 alloy and the A2+B2 alloy gives the LBR-derived B2 strengthening contribution, which is approximately 25 MPa at $\dot{\epsilon}_{\min} = 10^{-8} \text{ s}^{-1}$. Adapted from Ref. [98].

Figure 24a shows that the monolithic A2 alloy exhibits a nearly constant stress exponent of $n_{A2, \text{Mono}} = 3.8$ at 1030 °C. Since no clear transition in the creep response is evident with this limited stress range, a single power-law description is considered appropriate for the monolithic A2 alloy. Using the experimentally determined monolithic A2 flow law as the matrix reference, the LBR analysis was repeated. As shown in Figure 24b, the LBR plot of $\dot{\epsilon}_{\min}^{1/3.8}$ as a function of σ reveals a horizontal stress offset between the A2+B2 alloy and the monolithic A2 alloy. At a $\dot{\epsilon}_{\min}$ of 10^{-8} s^{-1} , the LBR-derived B2 strengthening contribution from the monolithic A2 alloy is approximately 25 MPa. This value represents a local estimate at the selected creep rate and should not be interpreted as a constant threshold stress over the entire stress range. In contrast, the 30 MPa obtained from Figure 24a is retained only as the direct stress offset in the Norton plot at the selected creep rate.

The two reference approaches therefore provide B2 strengthening contributions of approximately 15 MPa for the extrapolated A2 reference and approximately 25 MPa for the monolithic A2 alloy. Although the absolute value depends on the chosen matrix baseline, both approaches lead to the same qualitative conclusion, namely that the B2 precipitates provide a measurable but moderate strengthening contribution under the investigated conditions, while the overall creep response remains largely controlled by the A2 matrix. These results

provide a quantitative basis for interpreting the creep behavior of TMT-8Cr-10Al in terms of matrix-controlled deformation with a moderate B2 strengthening contribution.

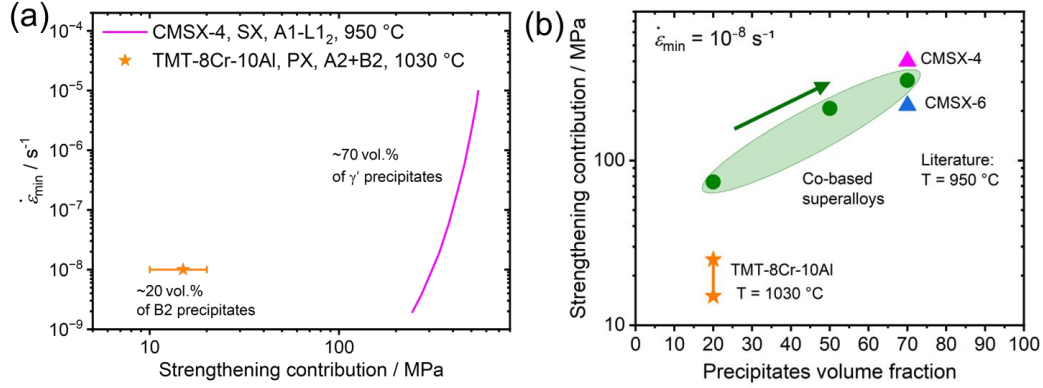


Figure 25. (a) Precipitation-strengthening contribution of TMT-8Cr-10Al at 1030 °C in comparison with CMSX-4 at 950 °C [30]. (b) Precipitation-strengthening contribution as a function of precipitate volume fraction for γ' -strengthened superalloys. Literature data are shown for 950 °C and at a minimum creep rate of 10^{-7} s^{-1} [30]. The result for TMT-8Cr-10Al at 1030 °C is included for comparison. Adapted from Ref. [98].

To further contextualize the magnitude of the precipitation-strengthening contribution of the A2+B2 TMT-8Cr-10Al, the present results are compared with those reported for the γ' -strengthened Ni-based superalloy CMSX-4, as shown in Figure 25a. Although the comparison is limited by the different testing temperatures, 1030 °C for TMT-8Cr-10Al and 950 °C for CMSX-4, the strengthening contribution obtained for the B2 precipitates in TMT-8Cr-10Al is substantially lower.

A major reason for this difference is the significantly lower precipitate volume fraction in TMT-8Cr-10Al, approximately 20 vol.%, compared with about 70 vol.% in CMSX-4. To place this into a broader context, Figure 25b shows the well-established correlation between precipitate volume fraction and strengthening contribution in γ' -strengthened superalloys. As discussed in Sections 2.2.2 to 2.2.5, precipitate strengthening depends strongly on precipitate population, morphology, and stability. Consistent with this framework, γ' -strengthened superalloys show a pronounced increase in strengthening contribution with increasing precipitate volume fraction. Co-based superalloys exhibit a near-linear increase in precipitation strengthening as the γ' volume fraction increases from 20% to 70%. CMSX-4, for example, achieves a strengthening contribution close to 400 MPa at a γ' volume fraction of about 70%, whereas Co-based superalloys with a precipitate fraction near 20% typically show much smaller contributions, less than 100 MPa. This trend is consistent with the comparatively moderate strengthening effect observed in the current TMT-8Cr-10Al.

In addition to precipitate volume fraction, the effectiveness of precipitation strengthening is also influenced by the test temperature relative to the precipitate T_{solv} . In TMT-8Cr-10Al, creep tests were performed at 1030 °C, corresponding to approximately 0.97 T_{solv} . As discussed in Sections 2.1.3 and 2.2.5, such proximity to the solvus temperature reduces precipitate stability and promotes coarsening or partial dissolution during creep, thereby diminishing the strengthening contribution of the precipitates. By contrast, CMSX-4 is typically tested at 950 °C, corresponding to approximately 0.85 T_{solv} , where the γ' phase remains more stable and more effective in sustaining precipitation strengthening.

Despite the relatively modest strengthening contribution of the B2 precipitates, the overall creep strength of TMT-8Cr-10Al remains comparable to that of CMSX-4 at similar minimum creep rates. This observation highlights the inherently high creep resistance of the A2 matrix and indicates considerable potential for further improvement through optimization of the precipitate phase. Increasing the B2 volume fraction and improving its thermal stability at elevated temperatures may lead to a substantially higher strengthening contribution.

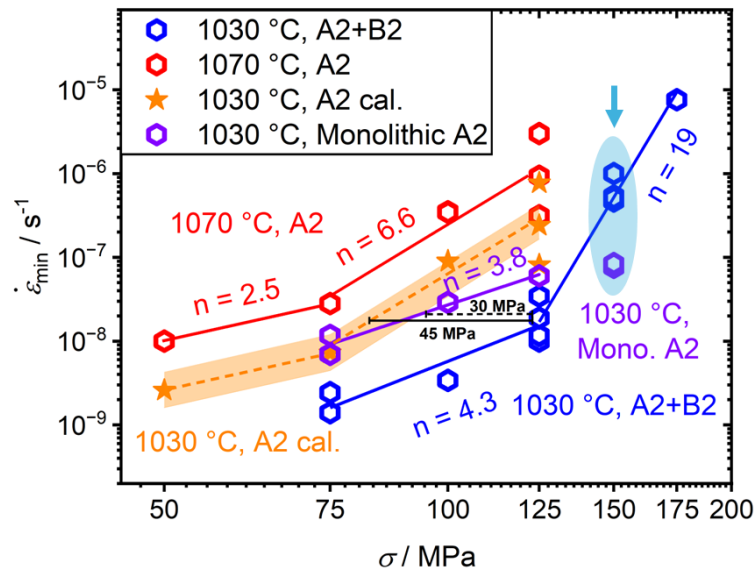


Figure 26. Norton plot with a comparison of minimum creep rates of the extrapolated A2 reference, the monolithic A2 alloy, and the A2+B2 alloy.

At higher applied stresses, specifically at 150 MPa and 1030 °C, the monolithic A2 alloy exhibited a slightly lower $\dot{\epsilon}_{\text{min}}$ than the A2+B2 two-phase alloy, as shown in Figure 26, highlighted by the light-blue shadow area. Considering the uncertainty associated with the matrix reference, this result does not provide clear evidence for an additional B2-related strengthening contribution under this specific condition. The strengthening contribution inferred in the lower-stress range therefore appears to be reduced at 150 MPa. This suggests

that the high-stress creep response is increasingly governed by deformation in the A2 matrix channels rather than by precipitate-controlled resistance.

This comparison must, however, be interpreted with caution. Although the monolithic A2 alloy was designed to approximate the A2 matrix composition determined by APT, it cannot fully reproduce the chemical and microstructural state of the A2 matrix within the two-phase alloy under creep conditions. In particular, the matrix in the A2+B2 alloy is coupled to interfacial equilibrium, elemental partitioning, precipitate coarsening, and possible partial dissolution near T_{solV} , whereas the monolithic A2 alloy represents a single-phase approximation. Small differences in refractory-element content in solid solution may therefore contribute to the slightly improved creep resistance of the monolithic A2 alloy. In addition, coarsening, partial dissolution, or local stress concentrations at coherent A2/B2 interfaces may reduce the effectiveness of the precipitate phase under high-stress conditions.

Beyond these limitations of the matrix reference, the TEM observations in Chapter 5 suggest that the absence of a measurable B2 strengthening contribution at 150 MPa is associated with a change in the dominant deformation response. The specimen crept at 150 MPa is characterized by extended dislocation segments within the A2 channels and frequent changes in glide plane, whereas paired dislocations associated with B2 precipitate cutting are not observed within the examined regions. The high-stress creep response therefore appears to be governed primarily by dislocation activity within the A2 channels rather than by precipitate-controlled resistance.

In summary, the LBR-based analysis provides a useful quantitative separation of matrix and precipitate contributions to creep in the A2+B2 two-phase RHEA system. Under the investigated conditions, the B2 precipitates contribute a moderate strengthening effect in the range of approximately 15–25 MPa at 1030 °C. These results emphasize the importance of precipitate volume fraction, thermal stability, and matrix composition in determining the overall creep performance, and highlight clear opportunities for further optimization of B2-strengthened RHEAs.

5 Dislocation Arrangements after Creep at 1030 °C

This chapter examines the dislocation configurations, dislocation-precipitate interactions, and A2/B2 interface stability of TMT-8Cr-10Al after creep at 1030 °C. While Chapter 4 established the macroscopic creep response, precipitate stability, rafting behavior, and B2 strengthening contribution, the present chapter provides direct TEM evidence from selected creep conditions to support the microstructural interpretation of these results. Particular attention is paid to specimens crept at 100, 125, and 150 MPa, which represent three characteristic stress levels within the investigated creep range.

The TEM observations presented in this chapter have been accepted for publication in Ref. [114]. The TEM specimen preparation and data acquisition presented in Chapter 5 were carried out in collaboration with Dr. Aparajita Pramanik and Prof. Ankur Chauhan (Indian Institute of Science, Bengaluru, India). The creep specimens analyzed here were prepared within the present work. The TEM data were analyzed and interpreted with the help of Dr. Shubhashis Dixit and Dr. R. J. Vikram (Institute for Applied Materials, Karlsruhe Institute of Technology, Germany), and the results are incorporated here to support the microstructural interpretation of the creep behavior at 1030 °C.

5.1 Scope of the TEM Analysis

The TEM analysis addresses two key microstructural aspects relevant to the present study. The first relates to the interaction between dislocations and B2 precipitates after creep at 1030 °C under different applied stresses, while the second addresses the structural stability of the A2/B2 interfaces after long-term high-temperature creep. Accordingly, this chapter does not aim to establish a comprehensive deformation mechanism, but to provide microstructural evidence supporting the interpretation of the stress-dependent creep response developed in Chapter 4.

Based on the creep results presented in Figure 14b, the creep response at 1030 °C changes markedly over the investigated stress range of 75 to 175 MPa, with a clear transition in creep stress sensitivity near 125 MPa. To examine the associated deformation substructures, specimens after creep at 1030 °C under 100, 125, and 150 MPa were selected for detailed TEM characterization. These conditions span the relevant stress range and enable a direct comparison of dislocation configurations and dislocation-precipitate interactions under different applied stresses. Figure 27 shows the corresponding creep rate evolution as a function of creep strain. The specimen tested at 100 MPa was interrupted in the late primary creep regime, where the creep rate was still decreasing and only approaching a $\dot{\epsilon}_{\min}$ value. Therefore, this condition should not be interpreted as having reached a fully developed

steady-state regime. In contrast, the specimens tested at 125 and 150 MPa exhibit an extended region with approximately constant strain rate, allowing $\dot{\epsilon}_{\min}$ to be determined reliably. The three specimens therefore provide representative microstructural states associated with different creep-rate levels at 1030 °C.

For TEM analysis, the investigation was restricted to grains with their $\langle 001 \rangle$ crystallographic direction approximately parallel to the compression axis. As discussed in Sections 2.2.5 and 4.5.2, pronounced N-type rafting of the B2 precipitates develops preferentially in this orientation in the present alloy. Restricting the analysis to such grains therefore provides a consistent basis for comparing dislocation configurations and interface characteristics across the investigated stress levels.

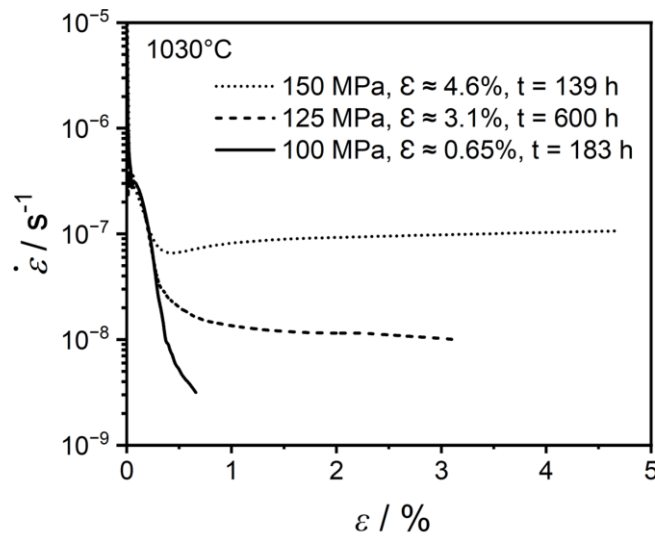


Figure 27. Creep rate evolution of TMT-8Cr-10Al as a function of creep strain for specimens tested at 1030 °C under 100, 125, and 150 MPa. Adapted from Ref. [114].

To further characterize the deformation state, SEM-EBSD analyses were performed on specimens crept at 1030 °C under applied stresses of 100, 125, and 150 MPa. The corresponding results are shown in Figure 28. The kernel average misorientation (KAM) maps (Figures 28a–d) reveal increased local misorientation in all crept samples compared to the homogenized condition, indicating the accumulation of plastic strain during creep. The magnitude and spatial heterogeneity of the local misorientation increase with applied stress and accumulated strain. Higher misorientation values are frequently observed near grain boundaries, suggesting localized strain concentrations in these regions.

The grain boundary character maps (Figures 28e–h), overlaid on image quality (IQ) maps, provide further insight into the deformation substructure. In addition to high-angle grain

boundaries (HAGBs), low-angle grain boundaries (LAGBs) are observed in both the homogenized and crept conditions. In the homogenized condition, only a limited number of LAGBs are present, which is likely associated with the preceding alloy processing and thermal history. After creep, however, the density of LAGBs within grains increases, as highlighted by the black dotted lines. These additional LAGBs are interpreted as deformation-induced subgrain boundaries formed during creep. Point-to-origin misorientation profiles across these deformation-induced LAGBs show misorientation angles of approximately 0.6° – 1.2° . These values are consistent with subgrain structures formed during creep deformation and indicate dynamic recovery processes involving the rearrangement of dislocations into lower-energy configurations.

Overall, the EBSD observations demonstrate a progressive evolution of the deformation substructure with increasing stress and strain. While these observations do not allow a definitive identification of the active deformation mechanism, they provide the microstructural basis for the TEM analysis presented in the following sections.

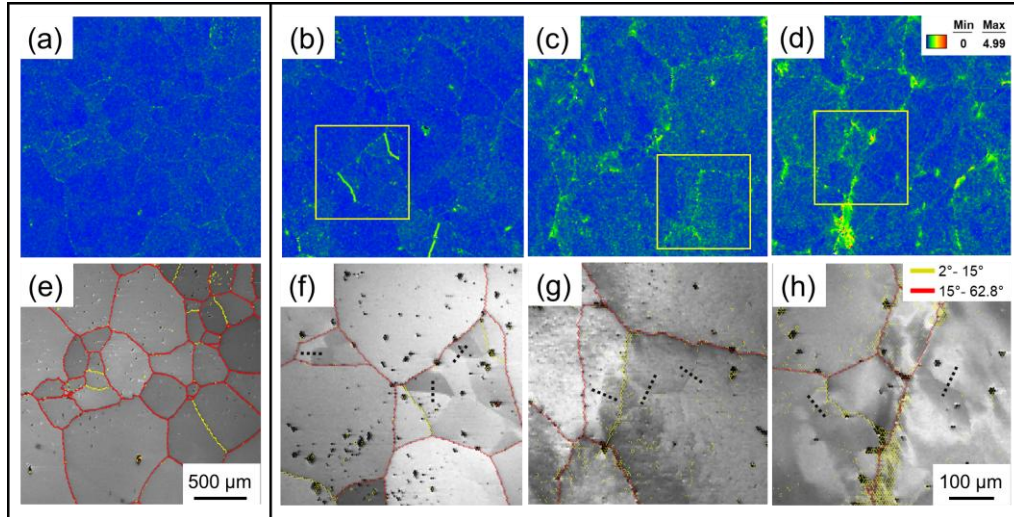


Figure 28. EBSD maps of TMT-8Cr-10Al in the homogenized and furnace-cooled condition and after creep at 1030 °C. (a–d) KAM maps of the homogenized condition (a) and after creep at 100 MPa (b), 125 MPa (c), and 150 MPa (d), showing increased local misorientation after creep. (e–h) Corresponding grain boundary character maps overlaid with image quality (IQ) maps for the same conditions. HAGBs are shown in red and LAGBs in yellow. Yellow boxes in (b–d) indicate regions selected for higher-magnification analysis in (f–h). Black dotted lines highlight the additional deformation-induced LAGBs, interpreted as subgrain boundaries formed during creep. Scale bars are 500 μm in (a, e), and 100 μm in (b–d) and (f–h). Adapted from Ref. [114].

5.2 Dislocation-Precipitate Interactions at 100 MPa

Figure 29a shows the TEM-SADP acquired along the [001] zone axis from the specimen after creep at 1030 °C under 100 MPa. In addition to the fundamental A2/B2 spots, weak B2 superlattice peaks are visible, confirming the persistence of long-range ordering in the precipitates after creep. The corresponding TEM-DF micrograph obtained using a B2 (100) superlattice reflection is shown in Figure 29b, where the B2 precipitates appear in bright contrast against the darker A2 matrix. The rafted B2 precipitates exhibit an elongated morphology along $\langle 100 \rangle$, consistent with the coherent cube-on-cube orientation relationship established in Section 4.4.

A low-magnification, two-beam TEM-BF micrograph is shown in Figure 29c. Only a small number of dislocations are visible, appearing as curved dark lines. The overall dislocation density is low, consistent with the limited creep strain of approximately 0.65%. At higher-magnification, a representative dislocation-precipitate interaction is shown in Figure 29d. The dislocation segments exhibit curved configurations in the vicinity of a B2 precipitate (indicated by the black arrow), suggesting local interaction with the precipitate.

The trace analysis procedure is described in Appendix A1. For the dislocation shown in Figure 29d, the projected trace is geometrically compatible with glide on either $(\bar{1}01)$ with a Burgers vector of $\frac{1}{2}[\bar{1}\bar{1}1]$ or on (101) with a Burgers vector of $\frac{1}{2}[\bar{1}11]$. Because these traces overlap on the (001) projection, the two possibilities cannot be distinguished unambiguously. The projected edge and screw orientations were therefore evaluated for both possible slip-plane configurations, as shown in Appendix A1, Figure A1. In both cases, the dislocation exhibits a mixed edge-screw character, which is compatible with climb-assisted motion [114]. No paired dislocations associated with B2 precipitate cutting are observed at this stress within the examined regions. Instead, dislocations remain isolated and interact locally with individual precipitates, consistent with local climb-assisted bypass rather than precipitate cutting.

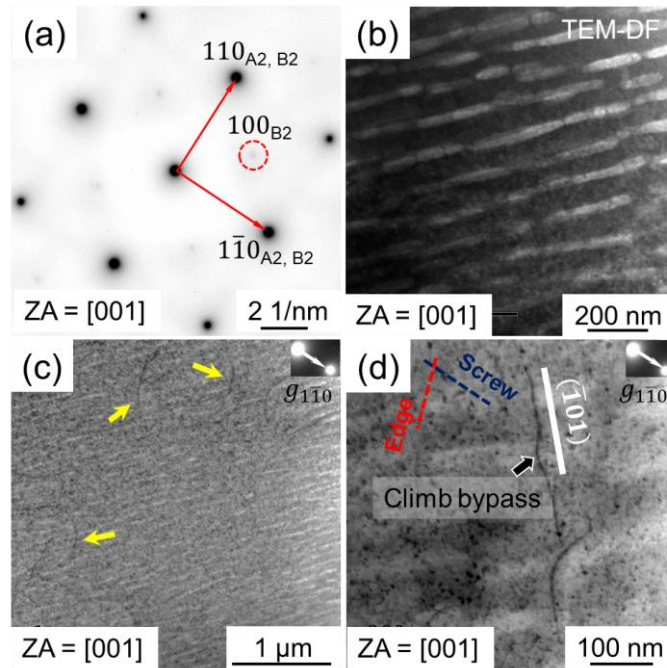


Figure 29. TEM results of the specimen after creep at 1030 °C under 100 MPa. (a) TEM-SADP along the [001] zone axis (ZA) showing fundamental A2/B2 spots and weak B2 superlattice peaks. (b) TEM-DF micrograph obtained using a (100) B2 superlattice peak reveals bright-contrast B2 precipitates. (c) Low-magnification, two-beam TEM-BF micrograph providing an overview of the crept microstructure with few marked curved dislocations. (d) A relatively high-magnification, two-beam TEM-BF micrograph showing a curved dislocation segment (black arrow) interacting with a B2 precipitate, consistent with local climb-assisted bypass. The blue and red dashed lines indicate the projected screw and edge orientations, respectively; the solid white line indicates the trace of the (101) plane. Adapted from Ref. [114].

5.3 Dislocation-Precipitate Interactions at 125 MPa

The TEM-SADP acquired along the [001] zone axis from the specimen after creep at 1030 °C under 125 MPa is shown in Figure 30a. Both the fundamental A2/B2 peaks and the weak B2 (100) superlattice spots are visible. The corresponding TEM-DF micrograph obtained using the B2 (100) superlattice reflection under near two-beam conditions is presented in Figure 30b, where the B2 precipitates appear in bright contrast and exhibit a rafted morphology along the $\langle 100 \rangle$ direction as described in Section 4.5.2.

A low-magnification, two-beam TEM-BF micrograph of this condition is shown in Figure 30c. Compared with the specimen after creep at 100 MPa, a higher density of dislocations is observed. This is consistent with the higher accumulated creep strain and the extended

interval with nearly constant creep rate shown in Figure 27. Representative dislocation-precipitate interactions at higher magnification are shown in Figures 30d–i.

In Figure 30d, dislocations in the vicinity of B2 precipitates exhibit curved segments, as indicated by pink arrows. The same region imaged under the reversed diffraction vector is shown in Figure 30e, using $g = [1\bar{1}0]$ and $-g = [\bar{1}10]$. Under the reversed diffraction condition, one of the curved segments displays a jog-like configuration, indicated by the black arrow. Additional dislocations with cusp-like features are also observed in the examined specimen (in Appendix A1, Figure A2, indicated by yellow arrows). These features resemble jogged screw dislocations reported in the literature [115,116] and are consistent with climb-assisted dislocation motion.

In contrast to the specimen after creep at 100 MPa, paired dislocations are frequently observed at 125 MPa, as shown in Figures 30d–f and Appendix A1, Figure A2. These paired dislocations extend from the A2 matrix into the B2 precipitates and are interpreted as evidence for B2 precipitate cutting by coupled superpartial dislocations. Following the trace-analysis procedure described in Appendix A1, the projected traces of the paired segments are geometrically compatible with $\{110\}$ or $\{112\}$ slip planes, as shown in Figure 30f.

To distinguish paired dislocations gliding on the same slip plane from dipole configurations (i.e., a pair of dislocations moving on parallel slip planes in opposite directions), the same region was imaged under the reversed diffraction condition, as shown in Figures 30d–e. The spacing and contrast of the paired dislocations in the A2 matrix remain essentially unchanged upon g -reversal, indicating that the two segments lie on the same slip plane rather than forming a dipole. By contrast, the paired segments inside the B2 precipitate show contrast reversal, as highlighted by dashed ellipses. This behavior is consistent with coupled superpartial dislocations with identical Burgers vectors gliding on the same slip plane in the ordered B2 phase.

In the paired configurations, one segment exhibits weaker contrast than the other under a given diffraction condition. Under g -reversal, the contrast of this segment changes. This contrast asymmetry is attributed to the local strain fields and elastic interaction between the closely spaced paired segments [117,118]. For better visualization, a color-filtered overlay of the same region is provided in Appendix A1, Figure A3.

In addition, the spacing between the paired dislocations changes across the A2/B2 interface, with a smaller separation inside the B2 precipitate than in the surrounding A2 matrix. This indicates that the two dislocations are more strongly coupled inside the ordered phase. Such stronger coupling is expected because separation of superpartials in B2 creates an APB ribbon; the associated APB energy provides a restoring force that limits the separation

between the paired dislocations. The reduced spacing inside the precipitate therefore supports the interpretation of cutting of ordered precipitates by coupled superpartials.

Besides these paired configurations, long dislocation segments are observed within the A2 channels, as shown in Figure 30g. Their traces are consistent with slip on $\{110\}$ or $\{112\}$ planes, following the same trace analysis procedure described in Appendix A1. Occasional dipole-like configurations are also identified in the examined regions and are confirmed by imaging under opposite diffraction vectors, as shown in Figure 30h and Figure 30i.

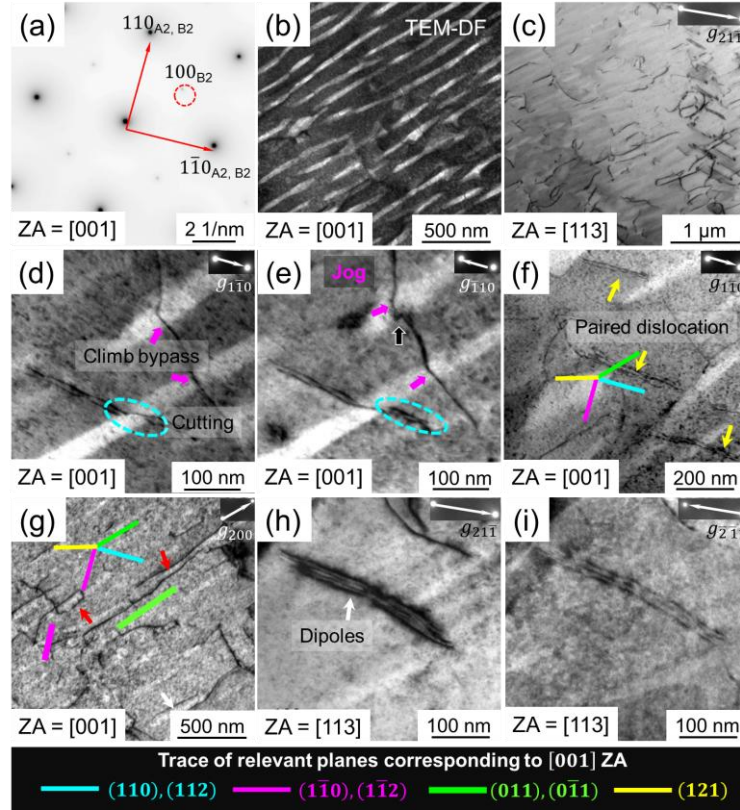


Figure 30. TEM results of the specimen after creep at 1030 °C under 125 MPa. (a) TEM-SADP along the [001] ZA showing fundamental A2/B2 spots and weak B2 superlattice peaks. (b) TEM-DF micrograph obtained using a (100) B2 superlattice peak. (c) Low-magnification, two-beam TEM-BF micrograph taken along the [113] ZA, showing the deformed microstructure. (d) Two-beam TEM-BF micrograph acquired using $g = [110]$, showing dislocation climb (pink arrows) and superpartial-mediated cutting of a B2 precipitate (ellipse). (e) Same area as in (d), imaged under reversed diffraction vector, $-g = [\bar{1}10]$, revealing the jogged climb segment (black arrow) and contrast reversal of superpartials within B2. (f) Paired dislocations (yellow arrow) near precipitates, indicative of earlier or forthcoming cutting events. (g) Long dislocation segments within A2 channels (red arrow), with corresponding trace analysis marked by solid lines. (h) and (i) Two-beam TEM-BF. Adapted from Ref. [114].

Overall, the microstructure at 125 MPa is characterized by a substantially higher dislocation density than at 100 MPa and by the coexistence of several dislocation configurations, including isolated curved segments, jogged features, paired dislocations associated with B2 precipitates, and long dislocation segments within the A2 channels. These observations indicate a more developed dislocation substructure and more frequent dislocation-precipitate interactions than at 100 MPa, involving both climb-assisted bypass and precipitate cutting.

5.4 Dislocation-Precipitate Interactions at 150 MPa

The TEM-SADP acquired along the [001] zone axis from the specimen after creep at 1030 °C under 150 MPa is shown in Figure 31a. In addition to the fundamental A2/B2 peaks, weak B2 superlattice reflections remain visible. The corresponding TEM-DF micrograph obtained using a B2 (100) superlattice reflection is presented in Figure 31b, where the B2 precipitates appear in bright contrast and retain an elongated morphology along $\langle 100 \rangle$.

A low-magnification TEM-BF micrograph of this condition is shown in Figure 31c. Compared with the specimens after creep under 100 and 125 MPa, the microstructure contains longer dislocation segments. Many of these dislocations exhibit abrupt changes in line direction, resulting in step-like configurations, as indicated by yellow arrows in Figure 31c.

A representative example is shown in Figures 31d–e, where the same dislocation configuration is imaged under diffraction conditions close to the [001] and [113] zone axes, respectively. The observed line traces indicate that the different segments of the same dislocation lie on different crystallographic planes. For the dislocation configuration shown in Figures 31d–e, the combination of the [001] and [113] projections indicates that adjacent segments are geometrically compatible with different $\{110\}$ and $\{112\}$ slip planes. It should be noted that the dislocation configurations illustrated in Figure 31f can overlap and appear like those observed in Figure 31d when viewed along the [001] ZA. The Burgers vector was determined as $\frac{1}{2}[\bar{1}11]$ using the visibility criterion, as summarized in Appendix A1, Figure A4. Additional curved or step-like dislocation configurations were observed in other regions of the same specimen, as shown in Appendix A1, Figure A5. These dislocations exhibit changes in contrast when imaged under different diffraction vectors, indicating that the observed line contrast is diffraction-condition dependent. The assignment of specific slip planes is therefore based on the combined trace analysis, Burgers-vector analysis, and projection geometry, rather than on a single projected image.

Additional TEM observations along the [113] zone axis are shown in Figure 31g. Several dislocation segments interact locally with B2 precipitates, as indicated by blue arrows. These local configurations resemble climb-assisted bypass features observed at lower stresses, indicating that climb-assisted interactions may still occur at 150 MPa. However, in contrast to

the 125 MPa condition, no paired dislocations associated with B2 precipitate cutting were identified within the examined regions.

Overall, the dislocation substructure at 150 MPa is characterized by long dislocation segments in the A2 channels, frequent changes in line direction, and isolated dislocation-precipitate interactions, whereas paired dislocation configurations associated with B2 precipitates are not observed in the examined regions. Compared with the microstructure at 125 MPa, the observations at 150 MPa therefore indicate a shift towards more extended dislocation activity in the A2 channels and less frequent paired configurations within the B2 precipitates.

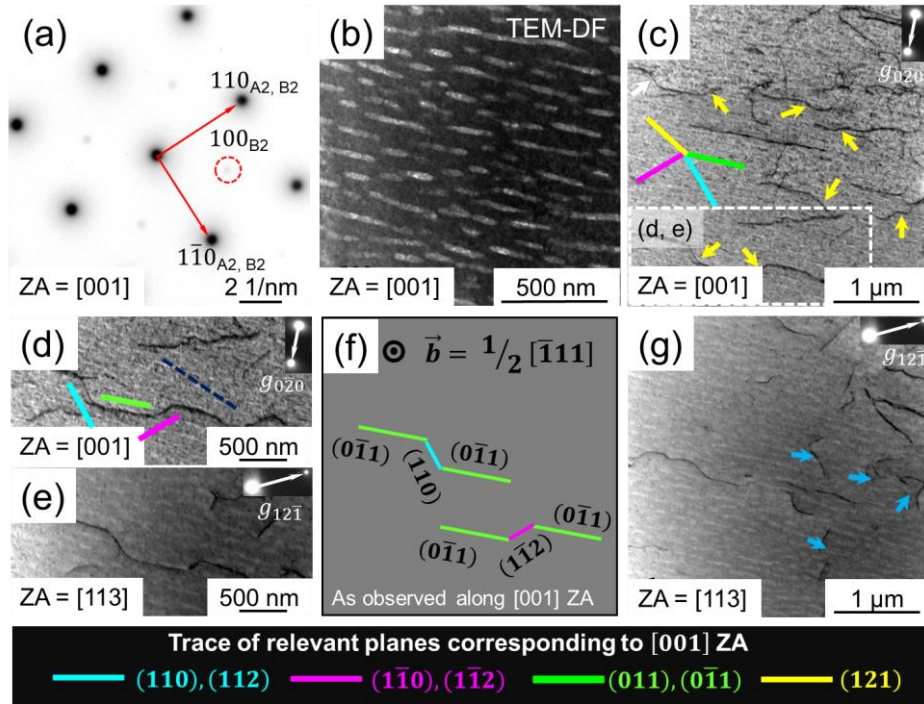


Figure 31. TEM results of the specimen after creep at 1030 °C under 150 MPa. (a) TEM-SADP along a [001] ZA showcasing fundamental A2/B2 spots and weak B2 superlattice peaks. (b) TEM-DF micrograph taken using a [100] B2 superlattice reflection. (c) Low-magnification, two-beam TEM-BF micrograph showing long dislocation segments within A2 channels, many of which exhibit abrupt changes in line direction (yellow arrows). (d, e) Representative dislocation configuration imaged along the [001] and [113] ZA, respectively. The trace analysis in (d), together with the schematic representation in (f), indicates that adjacent dislocation segments are compatible with different {110} and {112} planes for the assigned $1/2\langle 111 \rangle$ -type Burgers vector. (g) TEM-BF micrograph acquired close to the [113] zone axis, showing local dislocation-precipitate interactions, including configurations compatible with climb-assisted bypass. Adapted from Ref. [114].

5.5 Coherency of the A2/B2 Interface after Creep

As discussed in Sections 2.2.4 and 2.2.5, the stability of the matrix-precipitate interface is crucial for both precipitate strengthening and creep-induced morphology evolution. To assess the structural integrity and coherency of the A2/B2 interface after creep deformation, high-resolution STEM-HAADF analyses were performed on the specimen after creep at 1030 °C under 125 MPa.

Atomic-resolution micrographs were acquired along the [001] zone axis at several representative interface locations, as shown in Figure 32a. Figures 32b₁, c₁, and d₁ display the corresponding atomic-resolution images, while Figures 32b₂, c₂, and d₂ present inverse FFT (IFFT) images reconstructed using masked (110) reflections, one example of which is shown in Figure 32b₃. The continuity of the (110) lattice planes across multiple interface regions indicates that the A2 and B2 phases remain crystallographically coherent after creep deformation. Within the examined regions, no evidence of interfacial decohesion, interfacial dislocation networks, or localized structural disorder was noted. The preservation of lattice continuity across the rafted interfaces indicates that the A2/B2 interface retains coherency even after prolonged high-temperature creep exposure. Similar coherency is observed in the specimen after creep at 150 MPa (not shown), indicating that interface stability persists across the investigated stress range [114].

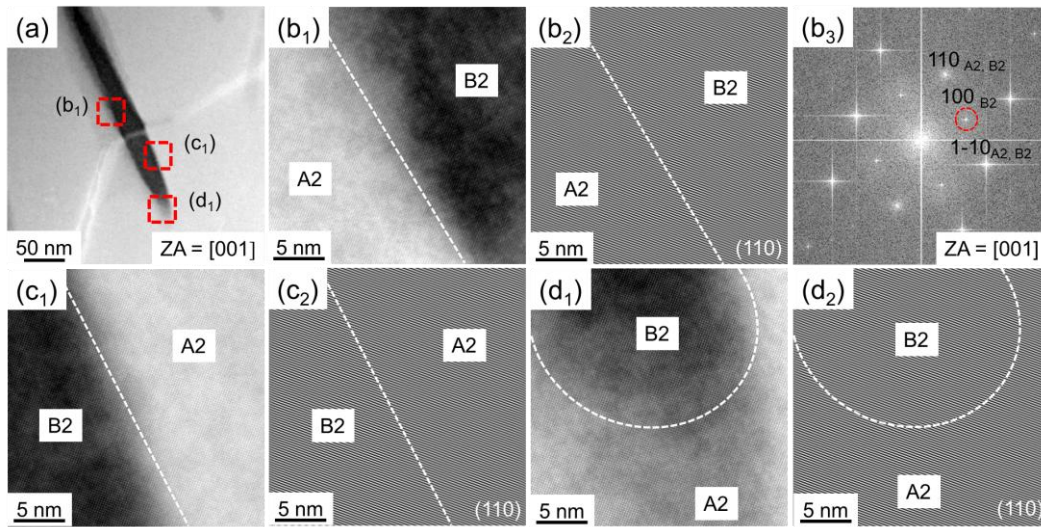


Figure 32. A2/B2 interface in the specimen after creep at 1030 °C under 125 MPa to a strain of 3.1%. (a) Low-magnification STEM-HAADF micrograph showing an elongated, rafted B2 precipitate in the A2 matrix. (b₁, c₁ and d₁) Atomic-resolution STEM-HAADF micrographs of different sections of the precipitate-matrix interface, as indicated in (a). (b₂, c₂ and d₂) Corresponding IFFT images obtained using the (110) spots from the FFT patterns. One representative FFT pattern corresponding to (b₁) is shown in (b₃). Adapted from Ref. [114].

These observations are consistent with previous reports on undeformed specimens annealed for up to 1000 h at 1000 °C [119], and support the conclusion that the A2/B2 interface in TMT-8Cr-10Al remains structurally stable under both thermal and mechanical loading. Accordingly, the stress-dependent differences in creep response observed in this study are more reasonably associated with changes in dislocation activity and dislocation-precipitate interactions than with interfacial degradation or loss of coherency.

5.6 Microstructural Implications for Creep at 1030 °C

The TEM observations presented in this chapter show that the local deformation substructure changes with applied stress, while the rafted A2+B2 microstructure retains a coherent interface at 1030 °C. As established in Sections 2.2.4 and 2.2.5, creep in precipitation-strengthened systems involves the interaction between dislocation motion in the matrix and the resistance imposed by precipitates. The present observations indicate that the creep behavior at 1030 °C is associated with changes in the character of dislocation-precipitate interactions under the investigated conditions.

At low stress (100 MPa), the observed dislocation configurations are most consistent with localized climb-assisted bypass of the B2 precipitates. Within the examined regions, no evidence was found for cutting of the ordered precipitates, which would require coordinated motion of paired dislocations [120]. This interpretation is consistent with experimental observations on dislocation configurations in B2-strengthened ferritic alloys [121–125] and with the theoretical considerations by Arzt et al. [126] and Blum et al. [127], which suggest that climb-assisted configurations can become relevant at comparatively low applied stresses. The observed configurations are also consistent with the relatively low apparent stress exponent, although the underlying deformation processes cannot be uniquely determined from this value alone.

At intermediate stress (125 MPa), the dislocation-precipitate interaction becomes more complex. In addition to configurations consistent with climb-assisted bypass, paired dislocations are observed in association with B2 precipitates. These paired segments are consistent with the cutting of the ordered phase by strongly coupled dislocation pairs and are in agreement with behavior previously reported for room-temperature deformation [119]. In addition, curved and jogged dislocation configurations indicate that climb-assisted motion remains active locally. The deformation substructure at this stress is therefore characterized by the coexistence of climb-assisted bypass and precipitate cutting, rather than by a single dominant interaction mode. Additional paired dislocations near precipitates, which may correspond to earlier or forthcoming cutting events [128], are shown in Figure 30f and in Appendix A1, Figure A2. The occurrence of coupled cutting at 125 MPa is physically plausible under the present creep conditions. This assessment is based on the Hühner-Reppich model

introduced in Section 2.2.4, Eq. (2.12), together with the line-tension approximation in Eq. (2.13). This model relates the critical shear stress for ordered precipitate cutting to the dislocation line tension, precipitate size, volume fraction, and APB energy. The parameters used for the semi-quantitative estimate are summarized in Appendix A2, Table A1. At 1030 °C, corresponding to a homologous temperature of about 0.52 with respect to the T_{solidus} , the shear modulus is expected to remain at roughly 75% of its room temperature value [129], which reduces the dislocation line tension. For an applied normal stress of 125 MPa, the corresponding shear stress is approximately 41 MPa when a Taylor factor of 3.06 is used [119]. Using the parameters listed in Appendix A2, Table A1, a critical shear stress of the same order is obtained when γ_{APB} at 1030 °C is reduced to about 60% of its room-temperature value [81]. Such a reduction in γ_{APB} appears reasonable because APB energy is expected to decrease as long-range order decreases at elevated temperature. For comparison, previous work on B2 Fe₃Al reported a decrease in γ_{APB} of approximately 24% at a lower homologous temperature of about 0.38 [130]. Although this estimate remains semi-quantitative because the relevant high-temperature parameters for the present alloy are not fully available, it supports the interpretation that the paired dislocations observed in Figures 30d–e correspond to coupled cutting of ordered B2 precipitates.

At high stress (150 MPa), the dominant feature is the presence of extended dislocation segments in the A2 channels, combined with frequent changes in line direction. These configurations are consistent with repeated glide on different but crystallographically related slip planes and may reflect cross-slip-like activity of screw or mixed dislocations. This is in line with the behavior expected for BCC and BCC-derived systems, where screw dislocations can change glide plane between related slip systems [53,131–133]. A schematic of the corresponding slip geometry is provided in Appendix A1, Figure A6. In contrast to the 125 MPa condition, no paired dislocations associated with precipitate cutting were identified in the examined regions at 150 MPa. Instead, the deformation substructure is dominated by extended channel dislocations with multiple changes in glide plane, while local dislocation-precipitate interactions remain present.

Across all investigated stress conditions, the B2 precipitates retain the rafted morphology along $\langle 100 \rangle$, as described in Section 4.5. In such a rafted microstructure, the relevant length scale for glide-controlled bypass is the effective channel width between adjacent B2 precipitates rather than a general average particle spacing. The Orowan stress was therefore estimated using the measured channel widths λ according to Eq. (2.14). The microstructural parameters used for this estimate are summarized in Appendix A2, Table A2. Using a temperature-corrected shear modulus of approximately 60 GPa, the calculated Orowan shear stresses lie between approximately 52 and 71 MPa. Converted to normal stress using a Taylor factor of 3.06 [119], these values correspond to about 159–217 MPa, which exceed the applied

creep stresses investigated here, 100–150 MPa. Classical looping is therefore not expected to be a favorable bypass mode under the present conditions.

In this context, repeated changes in glide planes provide a plausible explanation for the channel dislocations observed at 150 MPa. More generally, these TEM observations are consistent with the expectation that thermally activated glide-related processes become increasingly relevant at higher applied stresses [71,134]. However, the localized nature of TEM observations means that the simultaneous operation of additional deformation pathways at larger length scales cannot be excluded.

All observations discussed here correspond to creep at 1030 °C, which is approximately $0.52 T_s$ ($T_s \approx 2250$ °C) and corresponds to $0.97 T_{\text{solv}}$ as determined in Section 4.1. The deformation substructures described above therefore represent behavior under conditions close to the upper stability limit of the precipitates. At lower temperatures, slower diffusion would be expected to reduce climb-assisted bypass, while precipitate cutting could become more active, as is commonly observed in γ' -strengthened Ni-based superalloys [87,92,135]. Cross-slip activity in the A2 channels may remain relevant, but whether dislocations can propagate into adjacent channels would additionally depend on rafting kinetics, interparticle spacing along the rafting direction, and the applied stress.

A full mapping of these mechanism transitions would therefore require a systematic investigation over a broader temperature-stress range. In the present study, however, the accessible range is limited by both available testing times and the brittle response of the alloy. Higher stresses above 175 MPa lead to premature failure, while lower temperatures further restrict the experimentally accessible parameter range.

5.7 Comparison with the Ni-Based Superalloy CMSX-4

The dislocation activity observed in TMT-8Cr-10Al differs from that commonly reported for precipitation-strengthened Ni-based superalloys such as CMSX-4. In γ' -strengthened Ni-based superalloys, creep deformation is often accompanied by the formation of extended interfacial dislocation networks [87,92,136,137]. Under the present creep conditions, no comparable network formation is observed within the examined regions in TMT-8Cr-10Al.

This difference can be rationalized in the context of the microstructural framework established in Chapters 4 and 5. The sign and magnitude of the lattice misfit are expected to influence rafting, coherency stresses, and local interfacial dislocation behavior. As shown in Section 4.4, the lattice misfit between the B2 precipitates and the A2 matrix in TMT-8Cr-10Al is positive, whereas Ni-based superalloys such as CMSX-4 typically exhibit a negative misfit in the range of about -0.1 to -0.3% [138–140]. However, the sign of the lattice misfit alone cannot explain the absence of extended interfacial dislocation networks. Positive lattice misfit has

also been reported in Co-based superalloys, where rafting and interface-related dislocation activity can occur depending on alloy composition, temperature, stress state, and accumulated creep strain [141,142]. Therefore, the different misfit state is considered here as a contributing factor rather than as the primary reason for the different deformation substructure.

The comparatively small accumulated creep strain in the present experiments is likely a more important factor. In Ni-based superalloys, the development of interfacial dislocation networks is closely linked to continued creep deformation, dislocation accumulation at γ/γ' interfaces, and the progressive evolution of the rafted microstructure during prolonged exposure [143,144]. In the present alloy, the accumulated creep strains remain below 5% at 125 and 150 MPa, while at 100 MPa, the specimen was interrupted in the late primary creep regime before a fully developed steady-state regime was reached. This limited strain may be insufficient for the formation of a regular and extended interfacial dislocation network, even though local dislocation-precipitate interactions are clearly observed.

A further difference arises from the crystallography and dislocation mobility of the two matrix phases. CMSX-4 is based on a γ matrix strengthened by ordered γ' precipitates, whereas TMT-8Cr-10Al consists of a BCC A2 matrix strengthened by ordered B2 precipitates. As discussed in Section 2.1.2, plastic deformation in BCC and BCC-derived phases is strongly affected by the mobility of screw or mixed dislocations, their non-planar core structure, and their ability to change glide planes. The TEM observations in this chapter show curved and jogged dislocation configurations, particularly at 125 MPa, as well as extended channel dislocations with frequent changes in line direction at 150 MPa. These features suggest that dislocation motion in the A2 channels is locally constrained and may not favor the same degree of interfacial dislocation accumulation as typically reported in superalloys [116].

Taken together, these observations indicate that the deformation substructure in TMT-8Cr-10Al evolves differently from that in γ' -strengthened Ni-based superalloys, despite the competitive creep resistance of the present alloy. The comparison therefore highlights that similar macroscopic creep performance does not necessarily imply identical underlying dislocation arrangements.

6 Re and Ru Effects on Microstructural Stability of TMT-8Cr-10Al

This chapter investigates the influence of Re and Ru additions on the microstructural stability of TMT-8Cr-10Al, with particular emphasis on precipitate stability at elevated temperatures. As outlined in Sections 2.1.3 and 2.2.5, the stability of A2+B2 microstructures is governed by thermodynamic driving forces, elemental partitioning, interfacial and elastic contributions, and diffusion-controlled microstructural evolution. In this context, modifications in alloy chemistry may significantly affect the precipitate solvus temperature, the ordering temperature, the morphology of the B2 precipitates, and the coarsening behavior.

The following sections first establish the initial phase constitution of the Re- and Ru-modified alloys, followed by the effect of Re and Ru on T_{solv} and T_{ord} . The subsequent sections address microstructural evolution during aging, elemental partitioning, coarsening behavior, and the mechanistic origin of the observed changes in precipitate stability. These results are interpreted using the thermodynamic and microstructural framework introduced in Chapter 2.

6.1 Initial Microstructure and Phase Constitution

The homogenized and furnace-cooled Re- and Ru-modified alloys were first characterized by representative low-magnification SEM-BSE micrographs as shown in Figure 33. All investigated alloys exhibit coarse equiaxed grains with typical sizes of 200 to 500 μm . No secondary phases or large-scale compositional heterogeneities are detected, indicating successful chemical homogenization on the microscale for all investigated compositions. The dark spots visible in the micrographs are pores formed during sample preparation.

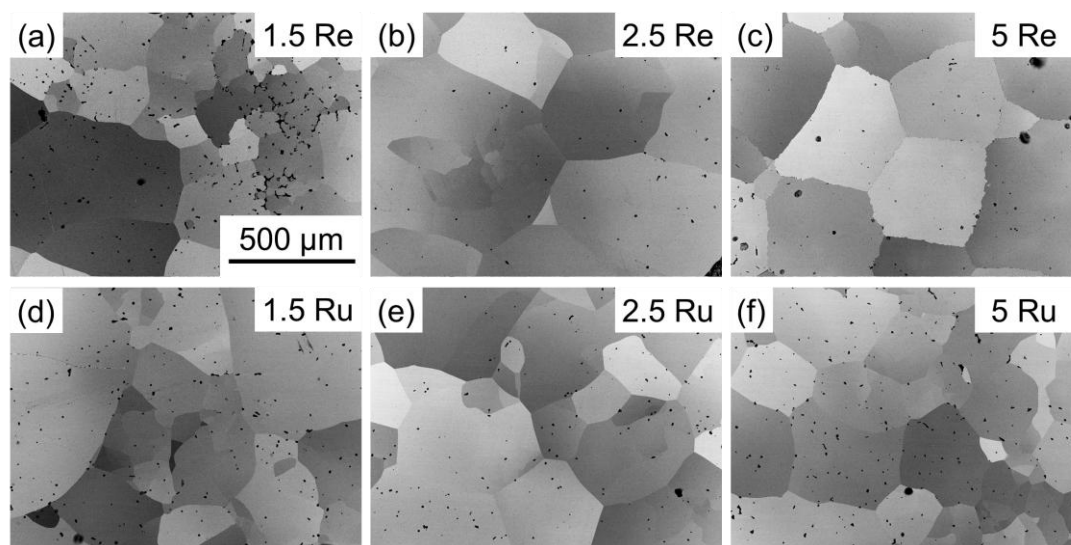


Figure 33. SEM-BSE micrographs showing the grain structure of the Re- and Ru-modified alloys in the homogenized and furnace-cooled condition. (a) 1.5Re, (b) 2.5Re, (c) 5Re, (d) 1.5Ru, (e) 2.5Ru and (f) 5Ru. All alloys exhibit coarse equiaxed grains with sizes on the order of several hundred micrometers. The dark spots visible in the micrographs correspond to pores formed during sample preparation.

High-magnification SEM-BSE micrographs are presented in Figure 34. All Re- and Ru-modified alloys show a two-phase microstructure consisting of a continuous bright matrix phase with dispersed, nanoscale darker precipitates. Based on the contrast established in Section 4.2.1 and consistent with previous reports for TMT-8Cr-10Al [13,25,98], the bright regions correspond to the A2 matrix, while the darker features represent ordered B2 precipitates. This observation confirms that all investigated alloys retain a nanoscale A2+B2 two-phase microstructure in the homogenized and furnace-cooled condition.

The B2 precipitates exhibit a refined, nearly spherical morphology in all investigated compositions. Image analysis according to Section 3.2.9 shows that the average precipitate diameter lies in the range of approximately 10–15 nm, depending on composition, with standard deviations of approximately 1–3 nm. The precipitates are relatively uniformly distributed throughout the A2 matrix for all investigated compositions. No additional phases or morphologically distinct intermetallic compounds were observed within the resolution limits of SEM-BSE micrographs.

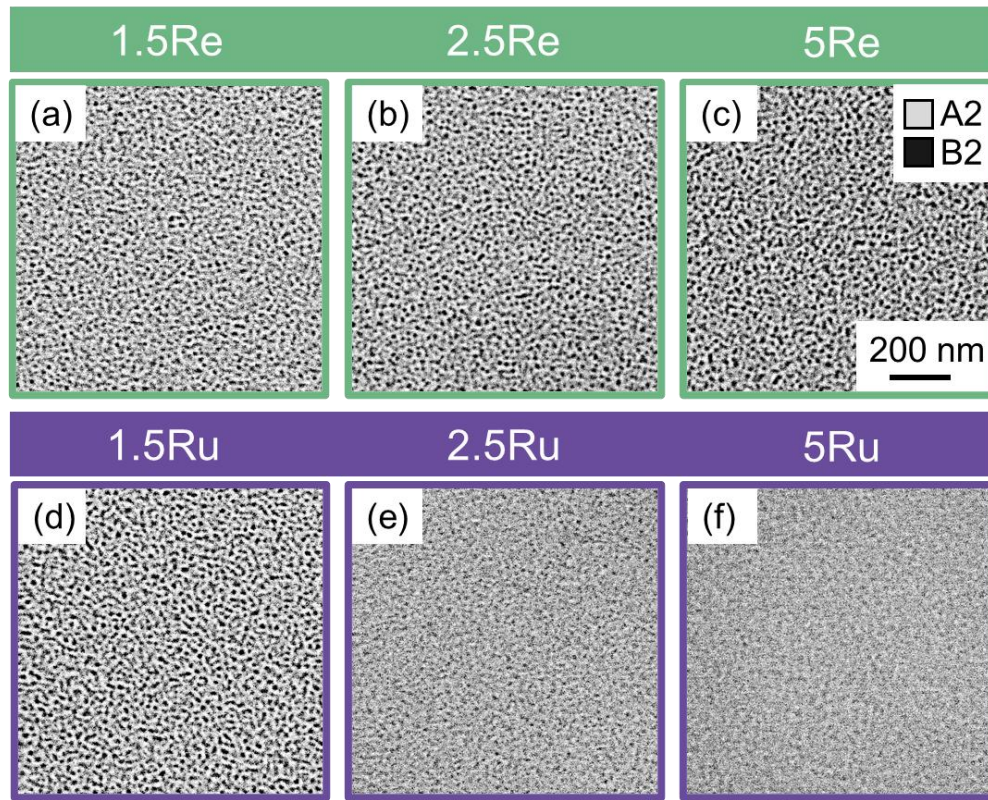


Figure 34. SEM-BSE micrographs of homogenized and furnace-cooled Re- and Ru-modified alloys. (a–c) Re-modified alloys with increasing Re content, and (d–f) Ru-modified alloys with increasing Ru content. All samples display a two-phase microstructure comprising a disordered A2 matrix and ordered B2 precipitates at the nanoscale. Green and purple labels denote the Re- and Ru-modified alloy series, respectively, and the same color coding is used throughout this chapter.

To verify the phase constitution inferred from SEM-BSE observations, XRD analyses were performed on the modified alloys and compared with the base alloy TMT-8Cr-10Al. As shown in Figure 35, the diffraction patterns remain essentially unchanged, and no additional reflections are detected. This indicates that the addition of Re or Ru does not alter the overall phase constitution of the alloy within the resolution limits of the present measurements.

As already discussed for the base alloy in Section 4.2.2, the absence of detectable B2 superlattice peaks in laboratory XRD does not exclude the presence of ordered B2 precipitates, but is consistent with the low B2 volume fraction and the intrinsically weak structure factor of the corresponding superlattice peaks [44]. In combination with the SEM-BSE observations, the XRD results support the conclusion that all investigated Re- and Ru-modified alloys retain the A2+B2 phase constitution in the homogenized and furnace-cooled state.

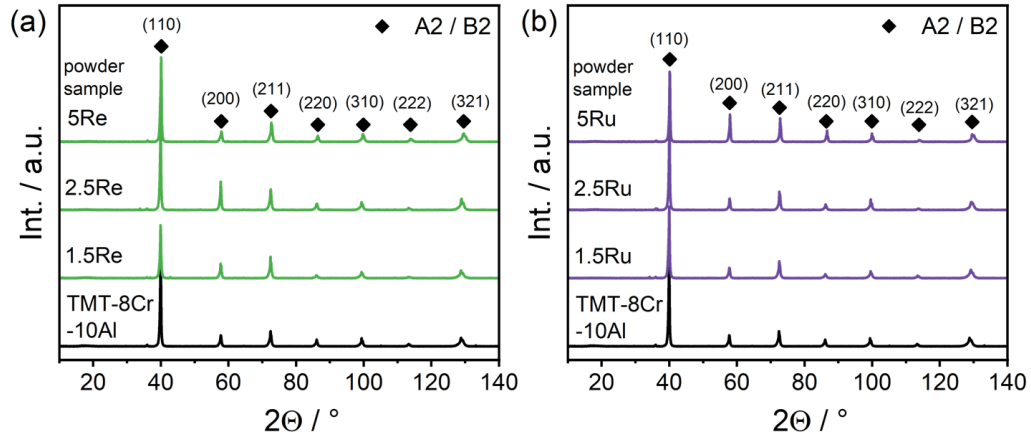


Figure 35. XRD patterns of powder samples of the base TMT-8Cr-10Al alloy and the Re- and Ru-modified alloys in the homogenized and furnace-cooled condition. (a) Re-modified alloys and (b) Ru-modified alloys. All peaks correspond to a BCC lattice and are denoted as A2/B2 reflections, since the fundamental reflections of the disordered A2 matrix and the ordered B2 phase overlap.

6.2 Effect of Re and Ru on Solvus and Ordering Temperatures

The thermal stability of the B2 phase in the Re- and Ru-modified alloys is characterized by T_{solv} and T_{ord} . As discussed in Section 2.1.3, these two temperatures reflect the thermodynamic stability of the A2+B2 two-phase microstructure. The T_{ord} values of the homogenized and furnace-cooled modified alloys were determined by DSC at three different heating rates of 10, 20, and 40 K/min. Representative DSC results, plotted as the temperature derivative of the enthalpy, dH/dT , are provided in Appendix A3, Figure A7. For all investigated compositions, the dH/dT curves show a single, well-defined peak within the examined temperature range, and no additional thermal features are detected for any alloy or heating rate.

In the equilibrium case, the order-disorder transition of the B2 phase would not be expected to show a pronounced dependence on heating rate. In the present multicomponent alloys, however, the experimentally observed DSC peak temperature shows a small shift with heating rate, as shown in Appendix A3, Figure A7. This apparent rate dependence is attributed to the fact that the loss of B2 order during heating is coupled with the concurrent compositional redistribution between the matrix and the secondary phase. Accordingly, for each composition, T_{ord} was determined by extrapolating the peak temperatures obtained at different heating rates to a nominal heating rate of 0 K/min, approximating quasi-equilibrium conditions.

The same evaluation procedure was applied to all alloys, as detailed in Appendix A3, Figure A8. The associated uncertainty is approximately ± 5 K. For comparison, the T_{ord} of the base alloy TMT-8Cr-10Al, $(1050 \pm 5)^\circ\text{C}$, is also included in Figure 36. The T_{solv} was determined independently by systematic microstructure analysis after isothermal experiments at selected temperatures, following the same evaluation procedure as described for the base alloy TMT-8Cr-10Al in Section 4.1. Representative micrographs illustrating this evaluation criterion are provided in Appendix A3, Figure A9. For reference, the T_{solv} of the base alloy, $(1065 \pm 5)^\circ\text{C}$, as determined in Section 4.1 is also included in Figure 36.

Figure 36 summarizes the experimentally determined T_{ord} and T_{solv} values as a function of Re and Ru content, respectively. For both alloy series, T_{ord} and T_{solv} increase systematically with increasing alloying element content. For the Re-modified alloys, T_{ord} increases from $(1050 \pm 5)^\circ\text{C}$ for the base alloy to approximately $(1109 \pm 5)^\circ\text{C}$ at 5 at.% Re, corresponding to an increase of approximately 60 K. Over the same composition range, T_{solv} rises from (1065 ± 5) to $(1135 \pm 5)^\circ\text{C}$, corresponding to an increase of approximately 70 K. For the Ru-modified alloys, T_{ord} increases from $(1050 \pm 5)^\circ\text{C}$ to approximately $(1102 \pm 5)^\circ\text{C}$ at 5 at.% Ru, corresponding to an increase of about 50 K, while T_{solv} increases from $(1065 \pm 5)^\circ\text{C}$ to approximately $(1115 \pm 5)^\circ\text{C}$, corresponding to an increase of approximately 50 K. A direct comparison between the two alloying elements shows that the increases in both T_{ord} and T_{solv} are more pronounced for Re than for Ru at comparable concentrations.

In all investigated compositions, T_{solv} remains consistently higher than the corresponding T_{ord} . This separation indicates that the loss of long-range order precedes the complete dissolution of the B2 phase upon heating. Thus, a temperature interval exists in which chemical phase separation may persist while the secondary phase is disordered. The increase in both T_{solv} and T_{ord} indicates enhanced thermal stability of the A2+B2 microstructure, with Re having the stronger effect. The origin of this stabilization is discussed further in the following sections in relation to thermodynamic and kinetic effects.

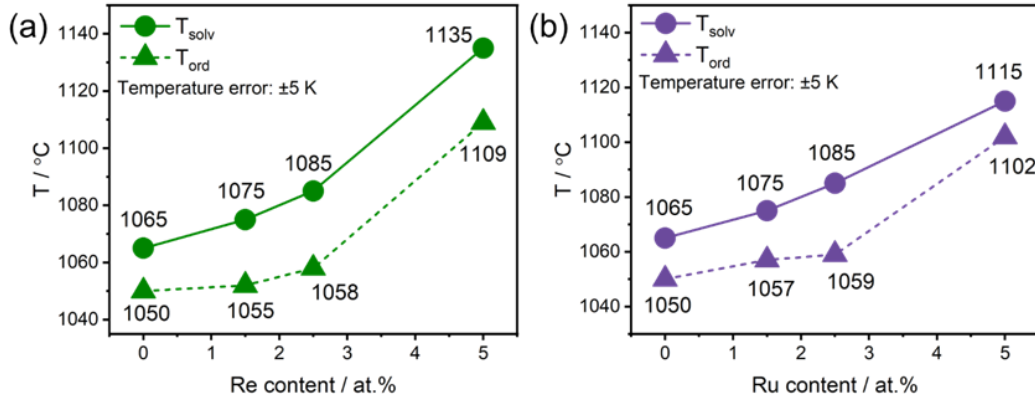


Figure 36. Dependence of the ordering temperature, T_{ord} , and the solvus temperature, T_{solv} , on alloying element content for (a) Re-modified and (b) Ru-modified alloys. The T_{ord} values were determined from DSC measurements by extrapolating the peak temperatures obtained at different heating rates to a nominal heating rate of 0 K/min. The T_{solv} values were determined independently by systematic isothermal annealing experiments followed by water quenching. Error bars indicate an estimated uncertainty of ± 5 K. Lines are guides to the eye.

To visualize the experimentally observed change in phase stability induced by Re addition, a semi-quantitative phase diagram section was constructed for the base alloy TMT-8Cr-10Al and the 5Re alloy, as shown in Figure 37. The 5Re alloy was selected because it exhibits the largest increase in both T_{solv} and T_{ord} , and therefore provides the clearest representation of the shift in phase stability. The diagram combines phase-boundary information obtained from quenching experiments and DSC measurements with tie-line compositions determined by APT, which are presented in Section 6.4.

It should be emphasized that this diagram does not represent a fully equilibrated thermodynamic phase diagram, but rather a semi-quantitative representation of the phase relations within the investigated composition range. Its purpose is to illustrate the experimentally observed shift of the miscibility gap and ordering regime caused by Re addition, rather than to provide a complete thermodynamic assessment.

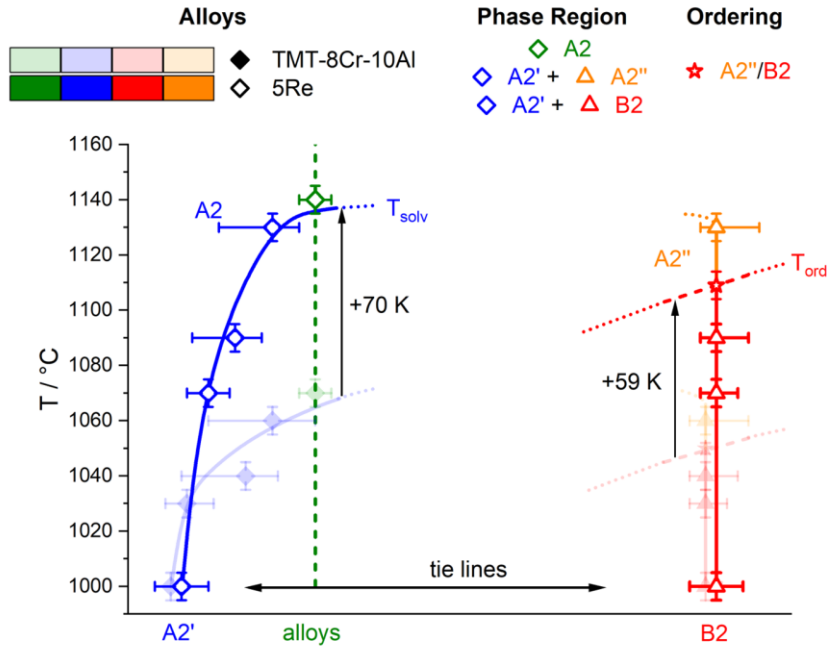


Figure 37. Schematic section along the approximate tie line illustrating the miscibility gap and ordering reaction. The T_{solv} is defined as the temperature above which the secondary phase completely dissolves $A2' + A2'' \rightarrow A2$, while the T_{ord} is defined as the temperature at which long-range B2 order within the secondary phase disappears, corresponding to the $B2 \rightarrow A2''$ order-disorder transition. The data for the base alloy were obtained from Refs. [35,145]. Temperature uncertainties are conservatively estimated; the compositions are obtained at 1000 °C and rounded to 1 at.%. Schematic compositions at temperatures higher than 1000 °C are estimated based on the phase fractions obtained after quenching.

6.3 Microstructure Evolution during Aging at 1000 °C

To assess the influence of Re and Ru additions on the thermal stability of the A2+B2 microstructure, both modified alloy series were subjected to systematic isothermal heat treatments at 1000 °C for durations of 10, 100, and 1000 h. This temperature was selected to maintain consistency with previous investigations on TMT-8Cr-10Al [25,27], enabling direct comparison of the temporal evolution of B2 precipitates. Representative SEM-BSE micrographs of the Re- and Ru-modified alloys after isothermal heat treatment are shown in Figure 38 and Figure 39, respectively.

In the Re-modified alloys, Figure 38, the B2 precipitates remain fine and uniformly dispersed after 10 h at 1000 °C, with no pronounced deviation from the initial homogenized and furnace-cooled condition. After 100 h, a clear increase in precipitate size is observed, while the overall morphology and spatial distribution remain largely unchanged. After 1000 h, pronounced

composition-dependent differences in precipitate morphology develop. In the 1.5Re alloy (Figure 38g), the precipitates evolve into elongated features with a clear directional alignment, resulting in a markedly anisotropic dispersion with only a small fraction of nearly equiaxed particles remaining. With increasing Re content, the degree of anisotropy decreases. In the 2.5Re alloy (Figure 38h), the precipitates are shorter and a larger fraction of nearly equiaxed precipitates is observed. In the 5Re alloy (Figure 38i), the microstructure is dominated by nearly equiaxed precipitates, while elongated features are largely suppressed.

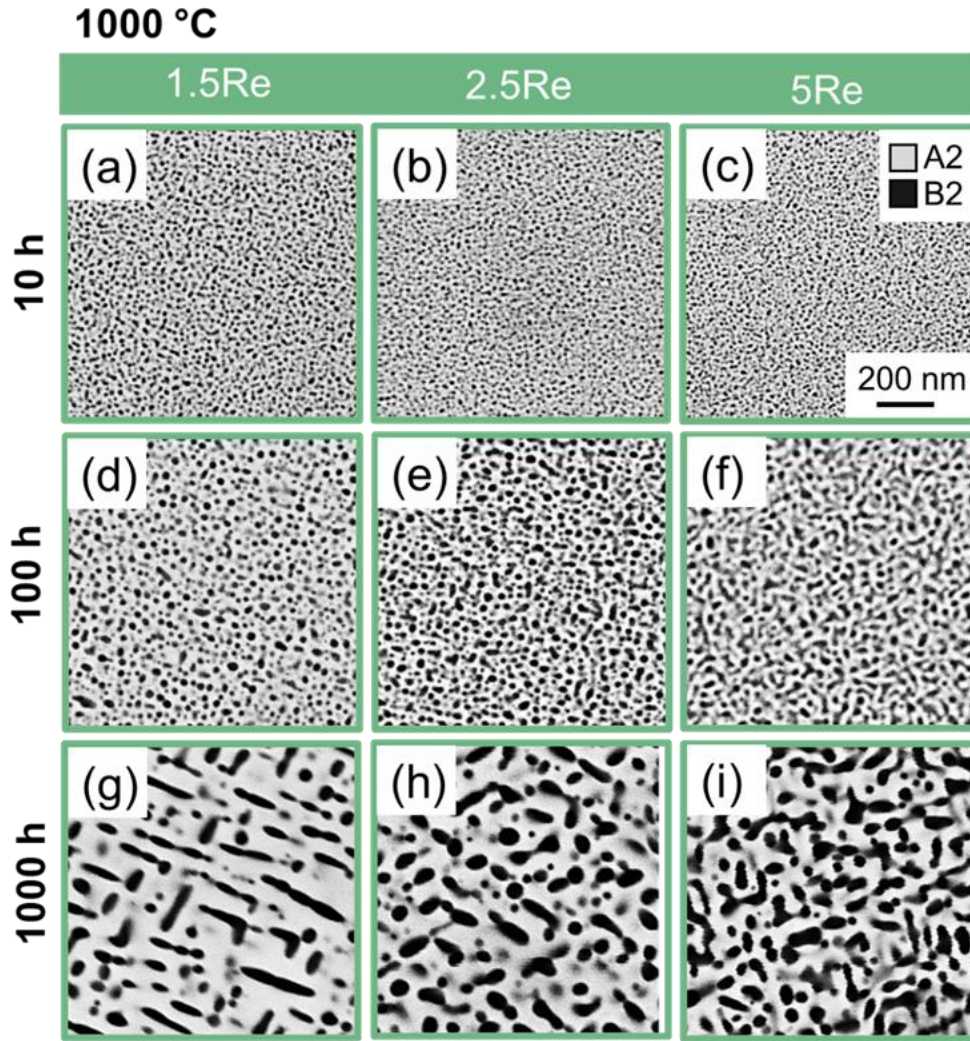


Figure 38. SEM-BSE micrographs showing the microstructural evolution of the Re-modified alloys after isothermal annealing at 1000 °C for (a–c) 10 h, (d–f) 100 h, and (g–i) 1000 h.

In contrast, the Ru-modified series exhibits a distinctly different morphological evolution during isothermal heat treatment at 1000 °C, as shown in Figure 39. After 10 h, the

microstructure still consists of fine, nearly equiaxed B2 precipitates, closely resembling the initial homogenized and furnace-cooled condition. After 100 h, directional growth becomes apparent, and elongated B2 precipitates begin to form. After 1000 h, all Ru-modified alloys develop a characteristic grid-like microstructure composed of elongated, plate-like B2 precipitates aligned along two approximately orthogonal crystallographic directions. This morphology indicates a pronounced directional coarsening behavior. With increasing Ru content, the width of the plate-like precipitates systematically decreases, whereas their length remains comparatively less affected. As a result, the precipitate network becomes progressively finer and more densely distributed with increasing Ru addition. This behavior suggests that Ru influences the coarsening process and morphological stability of the B2 phase in a manner distinct from Re.

In addition to the interconnected B2 network, isolated bright A2 regions are occasionally observed after 1000 h, as indicated by red arrows in Figures 39g–i. These regions are spatially associated with local impingement of adjacent B2 precipitates. As confirmed by subsequent TEM characterization, the microstructure remains composed only of the A2 matrix and B2 precipitates. The isolated bright regions are therefore interpreted as residual A2 matrix segments resulting from precipitate impingement rather than as an additional phase.

At the qualitative level, the aging experiments show that both Re and Ru modify the long-term precipitate evolution relative to the base alloy, but in different ways. As outlined in Section 2.2.5, coarsening behavior is controlled not only by diffusion kinetics but also by interfacial and elastic effects. In this context, the Re additions progressively suppress anisotropic precipitate elongation, whereas the Ru additions promote the formation of an extended plate-like network. These differences will be examined quantitatively in the following sections.

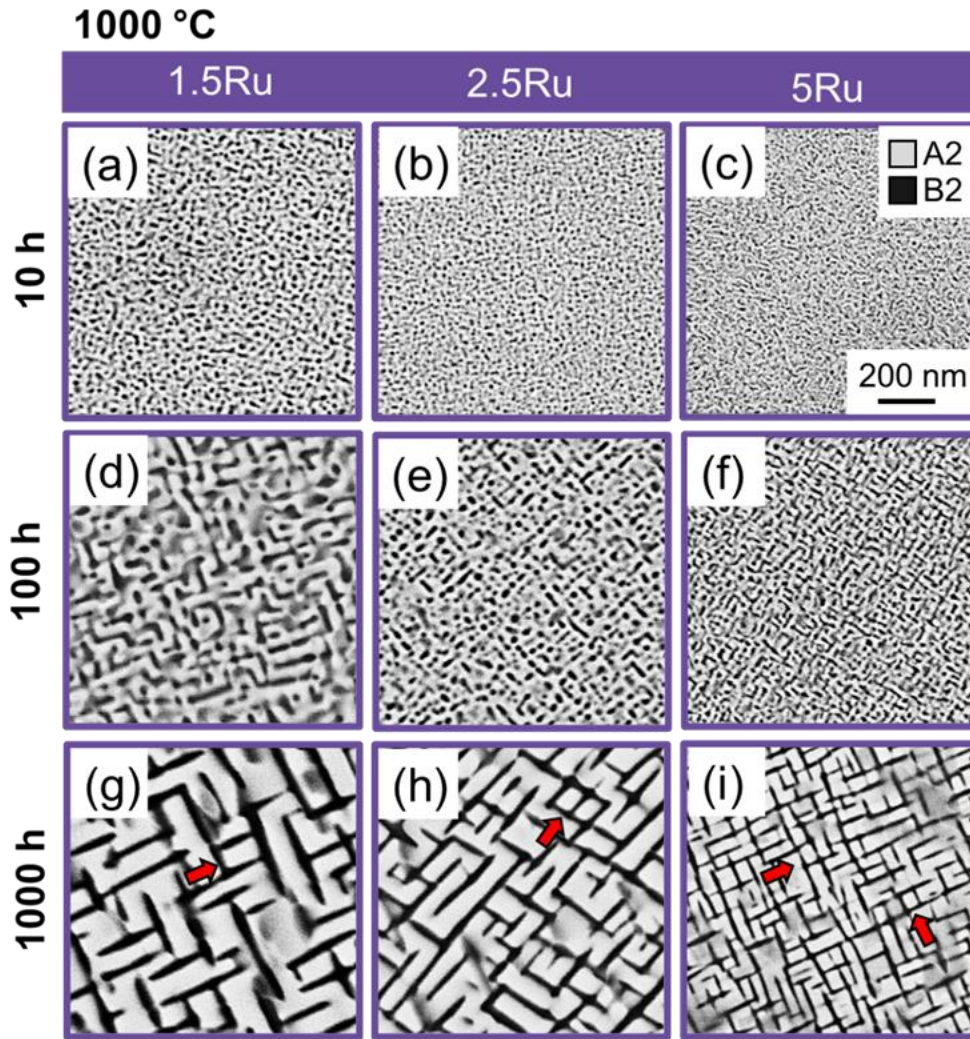


Figure 39. SEM-BSE micrographs showing the microstructural evolution of the Ru-modified alloys after isothermal annealing at 1000 °C for (a–c) 10 h, (d–f) 100 h, and (g–i) 1000 h. The red arrows indicate isolated A2 regions that are spatially separated from the interconnected B2 network.

To assess the phase constitution after long-term isothermal exposure, TEM analyses were performed on the 5Re- and 5Ru-modified alloys aged at 1000 °C for 1000 h. Representative TEM results acquired from regions containing both matrix and precipitates are shown in Figure 40.

The TEM-BF images of both alloys (Figure 40a and Figure 40d), obtained with the selected-area aperture inserted, reveal pronounced diffraction contrast arising from the coexistence of matrix and precipitate regions. The corresponding TEM-SAED patterns acquired along the

$\langle 001 \rangle$ zone axis are presented in Figure 40b and Figure 40e. In both 5Re- and 5Ru-modified alloys, the diffraction patterns exhibit the fundamental spots of the A2 phase together with the weaker superlattice spots characteristic of the ordered B2 phase. No additional diffraction peaks associated with other ordered intermetallic phases are observed. TEM-DF images formed using the highlighted $\{100\}$ superlattice spot in the corresponding TEM-SAED patterns are shown in Figure 40c and Figure 40f. In both cases, bright contrast is observed only from the ordered regions, whereas the surrounding matrix remains dark, indicating that the highlighted features correspond to B2-ordered precipitates embedded in the A2 matrix. These observations confirm that, even after prolonged aging, the microstructure remains a two-phase A2+B2 system without evidence of additional phase formation. Furthermore, the isolated A2 regions observed in the Ru-modified alloys after 1000 h are therefore interpreted as residual matrix segments resulting from precipitate impingement, rather than indicating the occurrence of additional phase transformations.

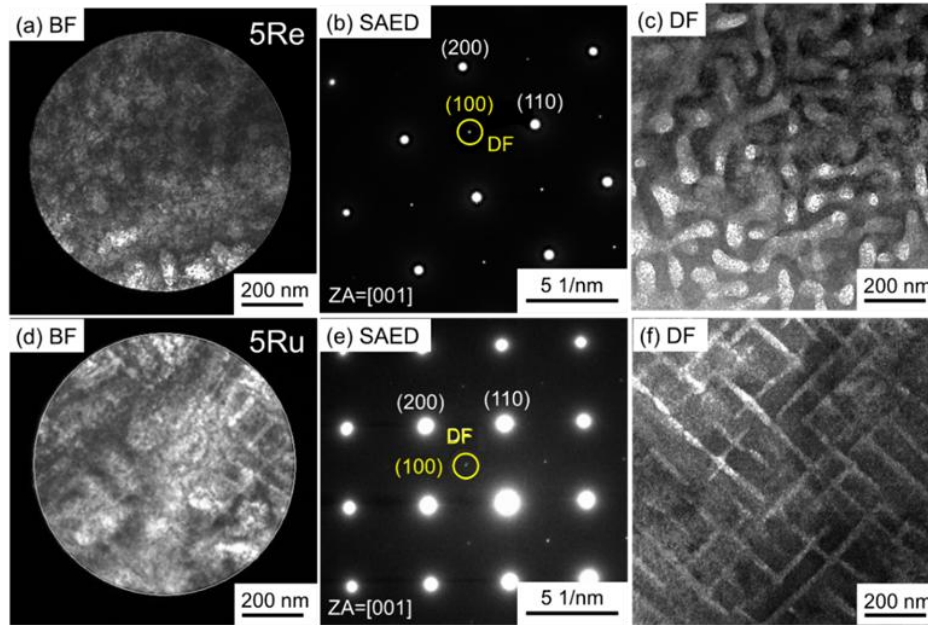


Figure 40. TEM characterization of the 5Re- and 5Ru-modified alloys after isothermal heat treatment at 1000 °C for 1000 h. (a, d) TEM-BF images acquired with the selected-area aperture inserted, indicating the regions used for diffraction analysis. (b, e) TEM-SAED patterns obtained along the $\langle 001 \rangle$ zone axis. The bright fundamental A2 spots, such as $\{110\}$ and $\{200\}$, are labeled in white. The weak $\{100\}$ superlattice spot associated with B2 ordering is highlighted by a yellow circle and selected for TEM-DF imaging. (c, f) TEM-DF images formed using the $\{100\}$ superlattice spot, revealing the spatial distribution of B2-type ordered domains in both modified alloys.

To further characterize the 3D morphology of the B2 precipitates after long-term isothermal exposure, FIB milling was employed to prepare three orthogonal cross sections in the 5Re- and 5Ru-modified alloys aged at 1000 °C for 1000 h. SEM-BSE micrographs acquired from the top, lateral, and front surfaces (Figure 41a and Figure 41c) provide complementary two-dimensional projections of the precipitate morphology along three perpendicular directions.

By combining these three orthogonal views, quasi-3D representations of the precipitate morphology were generated (Figure 41b and Figure 41d), enabling direct visualization of precipitate shape and spatial connectivity that cannot be unambiguously inferred from individual 2D SEM-BSE micrographs. The resulting morphologies for both alloys are fully consistent with the corresponding SEM-BSE observations in Figure 38 and Figure 39.

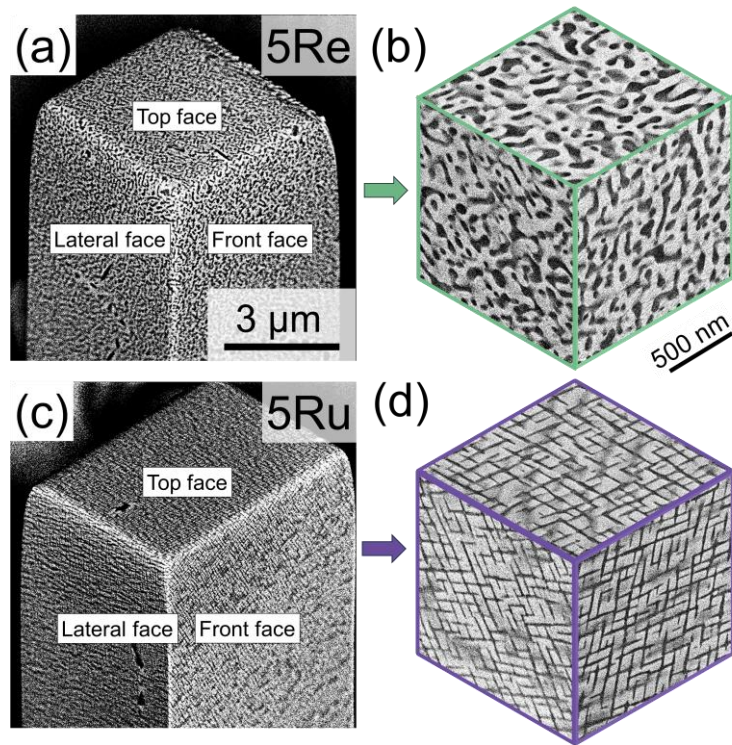


Figure 41. (a, c) FIB-prepared cuboids of 5Re and 5Ru alloys aged at 1000 °C for 1000 h, showing three orthogonal surfaces (top, lateral, front) prepared for multi-directional SEM-BSE imaging. (b, d) Quasi-3D representation of the precipitate morphologies in 5Re and 5Ru alloys generated from SEM-BSE images acquired on the corresponding three surfaces.

Quantitative image analysis was performed on SEM-BSE micrographs (Figure 38 and Figure 39) to characterize the evolution of the B2 precipitates during isothermal aging. The B2 precipitate size, area fraction, circularity, and number density were determined using ImageJ, and the results are summarized in Table 4.

For the Re-modified alloys, the precipitates remain sufficiently well separated over the investigated aging durations to allow their geometric dimensions to be measured directly. The major and minor axis, a_p and b_p , were determined, and the equivalent particle radius, r_{eq} , was calculated according to Eq. (2.18). As introduced in Section 2.2.5, the equivalent particle radius r_{eq} was used as a representative size parameter for non-spherical precipitates. In contrast, in the Ru-modified alloys, the precipitates increasingly overlap and form interconnected networks during prolonged aging, making it difficult to determine their full dimensions reliably. In this case, the minor axis length b_p was therefore used as the most robust characteristic size parameter, because it reflects precipitate thickening and can be consistently determined for both isolated and partially interconnected morphologies.

For the Re-modified alloys, the average precipitate size increases with aging time, indicating substantial coarsening. The evolution is described using the mean minor-axis length $\bar{b}_p$. In the 1.5Re alloy, $\bar{b}_p$ increases from (12 ± 4) nm after 10 h to (30 ± 10) nm after 1000 h. Similar behavior is observed in the 2.5Re alloy, while in the 5Re alloy the precipitates remain slightly finer after long-term aging, suggesting a reduced coarsening rate at higher Re contents. A similar time-dependent increase in precipitate size is observed in the Ru-modified alloys. However, the precipitates remain significantly finer at higher Ru content. In the 5Ru alloy, $\bar{b}_p$ increases from (8 ± 4) nm at 10 h to only (18 ± 6) nm after 1000 h, indicating a pronounced reduction in coarsening compared with both the lower-Ru-content alloys and the Re-modified series.

The number density of B2 precipitates decreases significantly with increasing aging time for all investigated compositions, consistent with coarsening processes. In contrast, the B2 area fraction remains nearly constant at approximately 25% for all alloys and aging conditions, indicating that the overall phase fraction is largely preserved during aging. The procedure used to determine the B2 area fraction from SEM-BSE micrographs is described in Appendix A3, Figure A10 and Figure A11. The circularity decreases with increasing aging time, particularly in the Ru-modified alloys. For example, in the 5Ru alloy the circularity decreases from 0.93 ± 0.12 after 10 h to 0.49 ± 0.31 after 1000 h, reflecting a strong deviation from equiaxed morphologies and the development of anisotropic precipitate structures.

Overall, this quantitative image analysis shows that aging at 1000 °C leads to coupled changes in precipitate size, morphology, and number density, while the B2 phase fraction remains essentially unchanged within the investigated aging range. These results provide the basis for the coarsening analysis presented in the following section.

Table 4. Quantitative characterization of B2 precipitates in Re- and Ru-modified alloys after isothermal aging at 1000 °C for different durations. The table summarizes the mean major axis ($\bar{a}_p$), mean minor axis ($\bar{b}_p$), mean equivalent particle radius ($\bar{r}_{eq}$), B2 area fraction, circularity, and number density of precipitates obtained from SEM-BSE image analysis. For the Ru-modified alloys, $\bar{a}_p$ and $\bar{r}_{eq}$ are not reported because partial interconnection of the plate-like precipitates prevents reproducible assignment of the full precipitate length to individual particles.

Alloy	Time / h	Mean major axis, $\bar{a}_p$ / nm	Mean minor axis, $\bar{b}_p$ / nm	Mean equivalent radius, $\bar{r}_{eq}$ / nm	Area% B2	Circularity	Number density N_A / $10^2 \mu m^{-2}$
1.5Re	10	15 ± 6	12 ± 4	13 ± 5	25 ± 3	0.92 ± 0.11	770 ± 45
	100	31 ± 11	19 ± 7	19 ± 8	26 ± 4	0.88 ± 0.12	419 ± 30
	1000	71 ± 20	30 ± 10	39 ± 13	25 ± 3	0.67 ± 0.20	92 ± 5
2.5Re	10	15 ± 7	10 ± 4	11 ± 4	25 ± 4	0.96 ± 0.07	917 ± 47
	100	30 ± 16	19 ± 6	19 ± 6	25 ± 3	0.76 ± 0.17	235 ± 14
	1000	59 ± 28	30 ± 9	36 ± 15	26 ± 4	0.71 ± 0.18	92 ± 14
5Re	10	15 ± 7	10 ± 4	10 ± 4	25 ± 4	0.96 ± 0.08	999 ± 66
	100	27 ± 14	17 ± 8	20 ± 7	25 ± 4	0.80 ± 0.17	331 ± 15
	1000	45 ± 22	28 ± 11	33 ± 13	25 ± 4	0.77 ± 0.17	134 ± 2
1.5Ru	10	-	12 ± 5	-	25 ± 4	0.88 ± 0.13	562 ± 42
	100	-	19 ± 9	-	25 ± 3	0.70 ± 0.24	176 ± 4
	1000	-	28 ± 13	-	25 ± 4	0.60 ± 0.34	79 ± 12
2.5Ru	10	-	10 ± 4	-	26 ± 4	0.93 ± 0.10	909 ± 95
	100	-	17 ± 6	-	25 ± 4	0.81 ± 0.16	374 ± 20
	1000	-	26 ± 10	-	25 ± 3	0.50 ± 0.34	95 ± 12
5Ru	10	-	8 ± 4	-	25 ± 4	0.93 ± 0.12	920 ± 119
	100	-	12 ± 5	-	26 ± 3	0.81 ± 0.18	633 ± 29
	1000	-	18 ± 6	-	25 ± 4	0.49 ± 0.31	196 ± 10

6.4 Elemental Partitioning between A2 Matrix and B2 Precipitates

APT analyses were performed to quantify the elemental partitioning behavior between the A2 matrix and B2 precipitates. The 2.5Re and 2.5Ru alloys after isothermal heat treatment at 1000 °C for 100 h were selected as representative compositions for detailed characterization, as this condition provides sufficiently coarsened precipitates with well-defined A2/B2 interfaces and enables reliable compositional measurements. In addition, previous studies on the base TMT-8Cr-10Al have shown that near-equilibrium partitioning is approached under comparable aging conditions [25,27], supporting the representativeness of the 100 h condition for elemental partitioning analysis.

Representative APT reconstructions for the 2.5Re and 2.5Ru alloys are shown in Figure 42a and Figure 42d, respectively. The B2 precipitates were identified using 35 at.% Ti isoconcentration surfaces, clearly delineating the B2 precipitates from the surrounding A2 matrix. One-dimensional concentration profiles were extracted from cylindrical volumes normal to the A2/B2 interfaces to quantify the compositional variations across the two phases. Representative concentration profiles are shown in Figure 42b and Figure 42c for the 2.5Re alloy and in Figure 42e and Figure 42f for the 2.5Ru alloy.

The quantified compositions of the A2 matrix and B2 precipitates for all investigated alloys are summarized in Table 5, together with the corresponding phase compositions of the base alloy aged under the same conditions for comparison. Consistent with the framework introduced in Section 2.1.2, a clear partitioning between the A2 matrix and B2 precipitates is observed in all alloys. Al and Ti are enriched in the B2 precipitates, whereas Ta and Mo preferentially partition to the A2 matrix. Cr shows nearly identical concentrations in both phases and therefore exhibits negligible partitioning. These trends are consistent with previous observations for the base alloy [13,25,98].

Despite these common partitioning trends, distinct differences are observed between Re- and Ru-modified alloys. To quantify the specific partitioning behavior of Re and Ru, the partition coefficient k_i was calculated as:

$$k_i = \frac{C_i^{A2}}{C_i^{B2}} \quad (6.1)$$

where C_i^{A2} and C_i^{B2} denote the atomic fraction of element i in the A2 matrix and B2 precipitates, respectively. Values of k_i significantly larger than 1 indicate preferential partitioning of element i to the A2 matrix, whereas k_i values significantly smaller than 1 indicate enrichment in the B2 precipitates. Values of k_i close to 1 indicate weak or negligible partitioning within the experimental uncertainty. In the Re-modified alloys, Re is preferentially enriched in the A2 matrix ($k_{Re} > 1$). The Re concentration profiles across the

A2/B2 interface are smooth and continuous, with no evidence of interfacial segregation. In contrast, Ru exhibits weak partitioning, with similar concentrations in both phases ($k_{\text{Ru}} \approx 1$). In addition, the concentration difference parameter ΔC_i , which reflects the compositional contrast between the two phases and contributes to the chemical driving force for coarsening, was calculated as:

$$\Delta C_i = \frac{(C_i^{\text{B2}} - C_i^{\text{A2}})^2}{C_i^{\text{A2}}} \quad (6.2)$$

The resulting ΔC_i values for the base alloy and the Re- and Ru-modified alloys are summarized in Table 5.

Overall, the APT results show that the additions of Re and Ru modify the partitioning characteristics in different ways while preserving the general partitioning behavior of the base alloy. In particular, the strong partitioning of Re to the A2 matrix, in contrast to the comparatively weak partitioning of Ru, is expected to influence both thermodynamic stability and diffusion-controlled microstructural evolution. These effects are discussed further in the following sections.

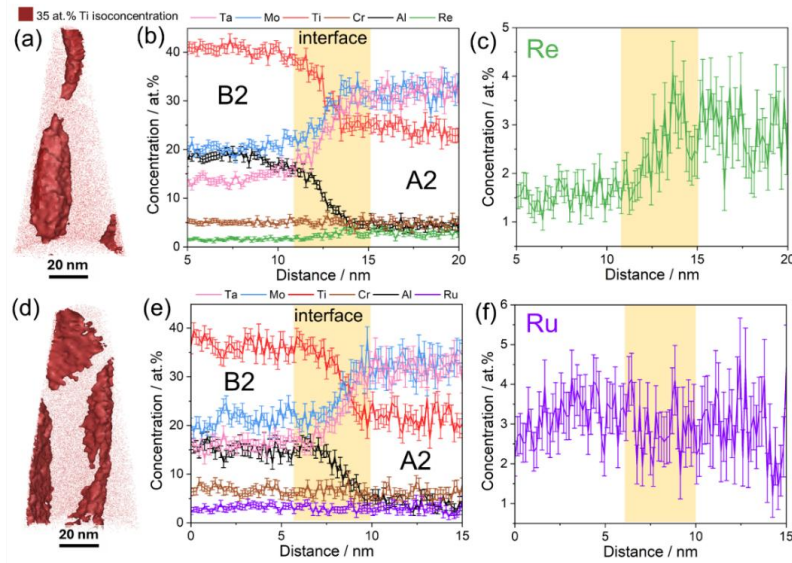


Figure 42. APT results of the (a–c) 2.5Re and (d–f) 2.5Ru alloys aged at 1000 °C for 100 h. (a, d) 3D reconstructions showing Ti isoconcentration surfaces at 35 at.% delineating the B2 phase. (b, c) and (e, f) 1D cylindrical concentration profiles across representative A2/B2 interfaces for the 2.5Re and 2.5Ru alloys, respectively, illustrating elemental partitioning between the matrix and precipitates.

Table 5. Average compositions of the A2 matrix and B2 precipitates measured by atom probe tomography after aging at 1000 °C for 100 h. The partitioning coefficients k_i and composition differences ΔC_i between the two phases are also listed. Data for the base alloy are taken from Ref. [27].

1000 °C/100 h	Phase	Compositions (at.%)						
		Ta	Mo	Ti	Cr	Al	Re	Ru
2.5Re	A2	31.7 ± 1.6	31.8 ± 1.6	24.4 ± 1.4	4.9 ± 0.7	4.3 ± 0.7	2.9 ± 0.6	-
	B2	17.6 ± 0.7	20.0 ± 0.8	41.0 ± 1.0	5.0 ± 0.4	18.8 ± 0.8	1.5 ± 0.2	-
	k_i	1.8	1.6	0.6	1.0	0.2	1.9	
	ΔC_i	7.8	4.4	11.4	0	48.7	0.7	-
5Re	A2	32.4 ± 2.9	29.8 ± 2.8	22.2 ± 2.6	5.7 ± 1.4	3.7 ± 1.2	6.1 ± 1.5	-
	B2	19.5 ± 2.1	21.2 ± 2.2	36.8 ± 2.5	6.6 ± 1.3	11.8 ± 1.7	4.1 ± 1.0	-
	k_i	1.7	1.4	0.6	0.9	0.3	1.5	
	ΔC_i	5.2	2.5	9.6	0	17.6	0.7	-
2.5Ru	A2	31.8 ± 2.3	32.4 ± 2.3	21.7 ± 2.0	5.9 ± 1.2	4.2 ± 1.0	-	2.9 ± 0.8
	B2	16.5 ± 1.3	21.5 ± 1.4	36.1 ± 1.6	6.5 ± 0.8	14.9 ± 1.2	-	3.4 ± 0.6
	k_i	1.9	1.5	0.6	0.9	0.3		0.9
	ΔC_i	7.4	3.7	9.6	0	27.4	-	0.1
5Ru	A2	32.7 ± 2.5	33.0 ± 2.5	20.6 ± 2.2	6.1 ± 1.3	3.5 ± 1.0	-	3.8 ± 1.0
	B2	18.3 ± 1.7	22.6 ± 1.9	34.8 ± 2.1	6.1 ± 1.1	12.3 ± 1.5	-	5.6 ± 1.0
	k_i	1.8	1.5	0.6	1.0	0.3	-	0.7

	ΔC_i	6.3	3.3	9.8	0	22	-	0.9
Base alloy	A2	30.4 ± 0.3	31.5 ± 0.2	25.0 ± 0	7.0 ± 0.1	5.7 ± 0	-	-
	B2	14.8 ± 0.2	21.7 ± 0.3	38.4 ± 0	6.6 ± 0.3	18.2 ± 0.2	-	-
	k_i	2.1	1.5	0.7	1.1	0.3	-	-
	ΔC_i	8	3	7	0	27	-	-

6.5 Coarsening Behavior of B2 Precipitates

The coarsening kinetics of the B2 precipitates during isothermal aging at 1000 °C were evaluated using the precipitate size statistics summarized in Table 4. As described in Section 6.3, precipitate size was quantified using several characteristic length parameters, including $\bar{a}_p$, $\bar{b}_p$ and $\bar{r}_{eq}$. Since the precipitates develop markedly different morphologies in the investigated alloys, the choice of characteristic length scale affects both the absolute value and the physical interpretation of the extracted coarsening-rate constant. The mean minor axis $\bar{b}_p$ was therefore used as the primary parameter for cross-alloy comparison, because it can be determined most robustly for both isolated precipitates and partially interconnected plate-like morphologies.

For the Re-modified alloys, where the precipitates remain sufficiently separated, the mean major particle dimension, $\bar{a}_p$, and the mean equivalent radius, $\bar{r}_{eq}$, were additionally evaluated to assess anisotropic growth. In contrast, for the Ru-modified alloys, an $\bar{a}_p$ -based coarsening rate was not extracted because prolonged aging leads to partial interconnection and overlap of adjacent plate-like precipitates, so that the full precipitate length can no longer be assigned to individual particles in a reproducible manner. Therefore, the Ru-modified alloys are evaluated using the minor-axis-based characteristic length, $\bar{b}_p$, which reflects precipitate thickening rather than full anisotropic growth.

For all alloys investigated, the increase in precipitate size with aging time is accompanied by a pronounced decrease in precipitate number density, while the B2 area fraction remains approximately constant. Following the generalized coarsening relation introduced in Section 2.2.5, Eq. (2.16), the bulk-diffusion-controlled case with $m = 3$ was used for the present analysis [85]. The cube of the characteristic length was fitted as a function of aging time t , and the slope was taken as an apparent coarsening-rate constant κ . The resulting fits are shown in Figure 43. In addition to the Re- and Ru-modified alloys, the base alloy and the previously reported Zr-modified alloy in the same alloy system are included here as reference

materials for comparison [27]. In the published study, precipitate size was evaluated using an area-equivalent circular radius since the precipitates are more isolated and exhibit a more regular morphology. For consistency, the κ values of the base alloy and the Zr-modified alloy were re-evaluated here using the minor-axis definition of precipitate size adopted in the present work.

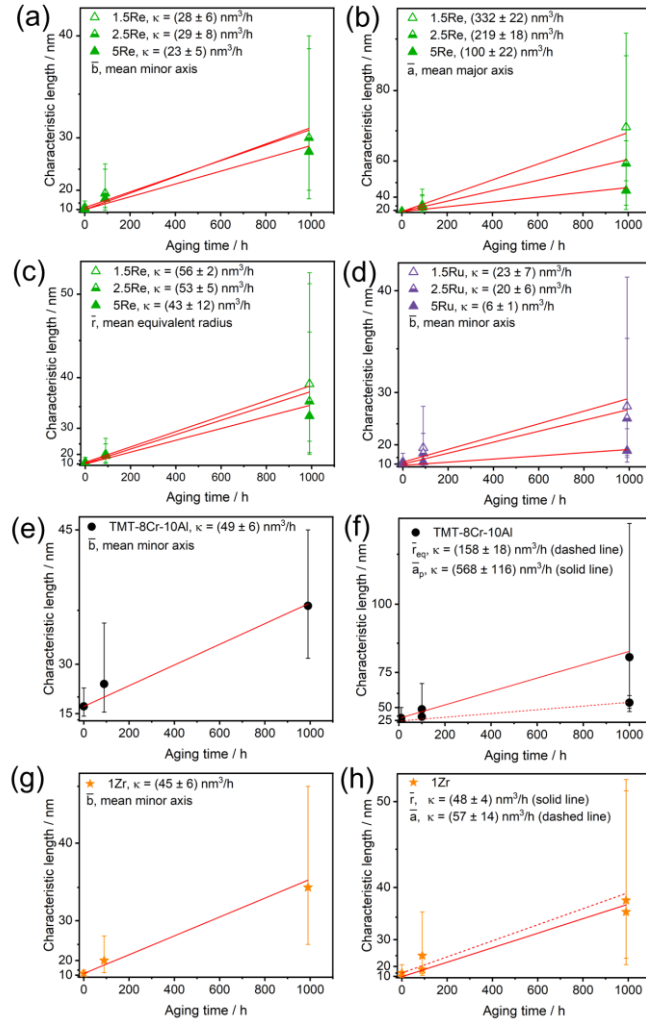


Figure 43. Evolution of characteristic length parameters during isothermal aging at 1000 °C for TMT-8Cr-10Al base alloy and Re-, Ru-, and Zr-modified alloys. The mean minor axis $\bar{b}_p$, mean major axis $\bar{a}_p$, and mean equivalent radius $\bar{r}_{eq}$ are used for (a–c) Re-modified alloys, (d) Ru-modified alloys, (e, f) TMT-8Cr-10Al, and (g, h) 1Zr alloy. The y-axes are cube-scaled but labelled with the corresponding characteristic length in nm. Linear fits were applied to $\bar{b}_p^3$, $\bar{a}_p^3$ and $\bar{r}_{eq}^3$ as a function of aging time to determine the coarsening-rate constant κ , as indicated in each panel. For the Ru-modified alloys, only the $\bar{b}_p$ -based coarsening rate is shown because partial interconnection of the plate-like precipitates prevents reproducible measurement of the full particle length.

The resulting κ values are summarized in Figure 44a. The main panel shows the κ value determined from $\bar{b}_p$, which enables a direct comparison across all investigated compositions. For completeness, the inset additionally shows the κ obtained from the $\bar{a}_p$, $\bar{b}_p$, and $\bar{r}_{eq}$.

Although the absolute values depend on the chosen size parameter, the Re-modified alloys show the same overall trend for all three measures. Based on the $\bar{b}_p$ -based evaluation, the base alloy exhibits the highest κ of approximately $49 \text{ nm}^3 \text{ h}^{-1}$, while the Zr-modified alloy shows a slightly lower value of about $45 \text{ nm}^3 \text{ h}^{-1}$. In contrast, substantially lower κ values are obtained for the Re- and Ru-modified alloys. For the Re-modified alloys, κ decreases to approximately $23\text{--}29 \text{ nm}^3 \text{ h}^{-1}$, whereas the Ru-modified alloys exhibit the lowest $\bar{b}_p$ -based κ values, with approximately $6\text{--}24 \text{ nm}^3 \text{ h}^{-1}$.

To account for differences in thermodynamic stability among the investigated alloys, the κ values were further analyzed as a function of the normalized temperature T/T_{solv} , as shown in Figure 44b. In this representation, the κ values of the present RHEA alloys exhibit a systematic dependence on the normalized temperature. The T_{solv} of the Zr-modified alloy, $(1065 \pm 5)^\circ\text{C}$, was determined in the present study using the same experimental procedure as for the other alloys, although it was not reported previously.

To place the coarsening kinetics of the present A2+B2 RHEA system in context with established precipitation-strengthened high-temperature alloys, κ values were compared with literature data reported for Ni-based and Cr-based superalloys [27,146]. Figure 44c shows the κ values as a function of T/T_{solv} , while Figure 44d presents the same comparison using the normalized temperature T/T_{solidus} . In both representations, the RHEA alloys exhibit substantially lower κ values than those reported for conventional Ni- and Cr-based superalloys.

The T_{solidus} values of the RHEA systems used for normalization were estimated using CALPHAD calculations performed with the Pandat software package (CompuTherm LLC). For the base, Ru- and Zr-modified alloys, the calculations were carried out using the RHEA thermodynamic database, whereas the Re-modified alloys were evaluated using the Nb-based database due to current database limitations. The calculated T_{solidus} for the Re-modified alloys should be regarded as approximate reference values used only for normalization. It should be noted that additions of refractory elements such as Re and Ru are often associated with an increased T_{solidus} in conventional superalloys [78]. The absence of a clear increase in the present CALPHAD estimations may reflect both the complex multicomponent interactions in the present alloy system and the known limitations of current thermodynamic databases when extrapolated to refractory multicomponent alloys [14,23].

Overall, the analysis shows that Re and especially Ru additions substantially reduce the $\bar{b}_p$ -based coarsening rate of the B2 precipitates relative to the base alloy. In this respect, the modified alloys exhibit a markedly improved microstructural stability at 1000°C . The origin of this stabilization is discussed further in the following sections in relation to compositional partitioning, thermodynamic phase stability, and precipitate morphology.

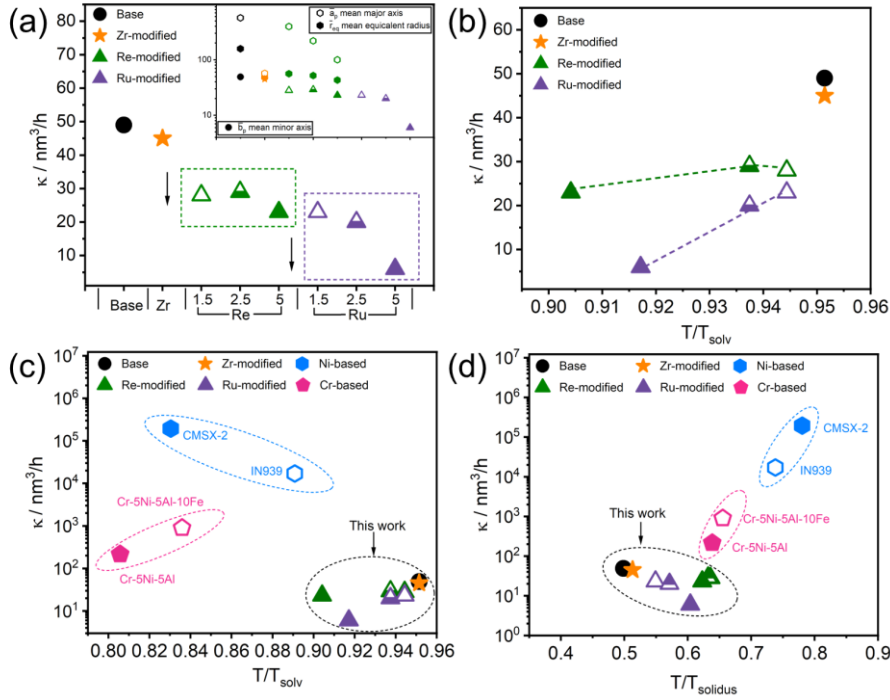


Figure 44. Coarsening kinetics of B2 precipitates in the investigated A2+B2 RHEAs. (a) Coarsening rate constant κ for the base alloy and Zr-, Re-, and Ru-modified alloys, obtained from the linear fits of $\bar{b}_p^3$ as a function of aging time. The inset compares κ values derived from different characteristic lengths ($\bar{a}_p$, $\bar{b}_p$ and $\bar{r}_{eq}$). (b) Dependence of the $\bar{b}_p$ -based κ on the normalized temperature T/T_{solv} for the investigated RHEA system. (c–d) Comparison of κ for the present RHEA alloys with literature data for Ni-based and Cr-based superalloys as a function of T/T_{solv} and $T/T_{solidus}$, respectively. Dashed lines are guides to the eye.

6.6 Effects of Alloying Elements on Partitioning and Phase Stability

The elemental partitioning behavior between the A2 matrix and B2 precipitates, established in Section 6.4, provides key insight into the chemical factors governing phase stability in the present RHEA system. As outlined in Section 2.1.2, phase stability in A2+B2 alloys is closely related to the distribution of alloying elements between the disordered A2 matrix and the ordered B2 precipitates. However, the present results show that the thermodynamic stabilization of B2 precipitates cannot be interpreted solely in terms of direct solute enrichment of the ordered phase.

A comparison of Re- and Ru-modified alloys reveals distinctly different partitioning behaviors. As shown in Section 6.4, Re partitions preferentially to the A2 matrix, whereas Ru exhibits only weak partitioning between the two phases. A similar tendency has been reported in Ni-

based superalloys, where Re strongly partitions to the γ matrix, while Ru shows nearly equal partitioning between the γ matrix and γ' precipitates [147,148]. In many ordered intermetallic systems, alloying elements often exhibit a pronounced site preference within the ordered phases, as demonstrated for transition-metal additions in B2-NiAl [149,150]. However, the present APT results indicate that Ru does not show a strong preference for either the A2 matrix or the B2 precipitates in the investigated RHEA system.

To examine whether the addition of Re or Ru alters the intrinsic chemistry of the ordered phase, the elemental ratios within the B2 precipitates were evaluated using the APT compositions listed in Table 5. In the base alloy, the ratio of Ti to (Al+Mo) is approximately 0.96, which is close to the expected 1:1 ratio for a Ti(Al,Mo)-type B2 chemical framework [27]. After the addition of Re or Ru, the Ti/(Al+Mo) ratio remains close to unity for all investigated compositions, ranging from approximately 0.99 to 1.08. These results indicate that the fundamental Ti(Al,Mo)-type B2 chemical balance of the ordered phase is largely maintained.

Despite their clearly different partitioning behavior, both Re and Ru additions lead to a systematic increase in T_{solv} and T_{ord} , as shown in Section 6.2, indicating an extended thermodynamic stability range of the precipitates in the modified alloys. In all investigated compositions, T_{ord} remains lower than T_{solv} , indicating that the ordered B2 precipitates are expected to lose long-range order during heating before eventually dissolving into the A2 matrix. The temperature interval between the service temperature and T_{solv} thus provides a useful measure of the thermodynamic stability margin of the precipitates during long-term exposure. A key result of the present study is that thermodynamic stabilization is not directly correlated with the partitioning direction of the alloying element. Although Re and Ru exhibit markedly different partitioning behavior, both additions lead to comparable increases in T_{solv} and T_{ord} . This decoupling indicates that, in the present multicomponent alloy system, the thermodynamic stabilization of B2 precipitates cannot be interpreted solely from whether an alloying element enriches the ordered phase.

A particularly instructive comparison is provided by the previously reported Zr-modified alloy in the same base alloy system [27]. In that case, Zr partitions strongly to the B2 and leads to the formation of a Ti(Al,Mo,Zr)-type B2 phase through preferential incorporation into one of the two sublattices. Based on the reported APT data [27], the atomic ratio Ti/(Al+Mo+Zr) is approximately 0.95, confirming that the composition remains close to the stoichiometry of an ordered B2 phase. However, despite this pronounced enrichment of the ordered phase, both T_{solv} and T_{ord} remain essentially unchanged relative to the base alloy [27]. The T_{solv} of the Zr-modified alloy was additionally verified in the present study by supplementary heat-treatment experiments, with representative micrographs provided in Appendix A3, Figure A9. This comparison further demonstrates that direct enrichment of the ordered phase is neither

a necessary nor a sufficient condition for increasing the thermodynamic stability range of precipitates in the present alloy system.

6.7 Precipitate Morphology and Elastic Interactions

A pronounced difference in precipitate morphology is observed between the Re- and Ru-modified alloys after long-term aging at 1000 °C. As shown in Section 6.3, the base alloy exhibits elongated B2 precipitates after 1000 h exposure. With increasing Re additions, the precipitates become progressively more equiaxed, with an increased fraction of spherical precipitates. In contrast, the Ru-modified alloys develop plate-like precipitates that form partially interconnected structures during prolonged aging.

As discussed in Section 2.2.5, precipitate morphology reflects the balance between interfacial energy and elastic strain energy contributions. The relative importance of these contributions is often described through the Thornton-Thompson parameter:

$$L = \frac{\delta^2 l C_{44}}{\gamma} \quad (6.3)$$

where l represents the precipitate size, C_{44} the elastic stiffness coefficient associated with shear deformation in cubic crystals, and γ the interfacial energy [151]. In the present work, a precise estimation of L is not possible because several required parameters are not available. The square of the lattice misfit (δ^2) is therefore used as a qualitative indicator of the relative magnitude of elastic strain energy. Higher δ^2 values therefore indicate a stronger elastic contribution to the overall energy balance.

For the base alloy, the elongated precipitates observed after aging are consistent with the positive lattice misfit of approximately $(+0.6 \pm 0.4) \%$ determined in Section 4.4 and with significant elastic interactions between the A2 matrix and B2 precipitates at 1000 °C [98]. To assess the effect of Re and Ru additions on the lattice misfit, approximate values were estimated using the experimentally measured APT phase compositions (Table 5) combined with elemental BCC lattice constants from the thermodynamic database presented in Ref. [34]. Although these values should be regarded as approximate, they indicate that the magnitude of lattice misfit remains significant in all alloys investigated, with calculated values ranging from 0.13 % to 0.25 % (see Appendix A3, Table A4), and they capture the systematic variation in misfit magnitude among the investigated alloys.

With increasing Re addition, the fraction of elongated precipitates decreases and a progressively larger population of spherical precipitates is observed. Re has a metallic radius of approximately 137 pm. Based on the APT-measured phase compositions shown in Table 5, simple phase-average atomic radius estimates yield average atomic radii of approximately 142.6 pm for the A2 phase and 143.1 pm for the B2 phase in the base alloy, and 142.4 pm for

the A2 phase and 143.0 pm for the B2 phase in the 5Re alloy. Although these estimates are approximate and do not account for temperature- or environment-dependent effects, they indicate that the observed reduction in lattice misfit cannot be rationalized by atomic-size effects alone. Instead, the change in lattice misfit is more likely associated with the redistribution of elements between the A2 and B2 phases after Re addition. In particular, the strong partitioning of Re to the A2 matrix alters the overall chemical composition of the matrix phase, which in turn significantly modifies its lattice parameter. A reduced lattice misfit weakens the elastic contribution and shifts the balance towards interfacial-energy-dominated morphologies, favoring the formation of more spherical precipitates. This interpretation is consistent with the estimated reduction in lattice misfit magnitude summarized in Appendix A3, Table A4. Additional support is provided by the interrupted creep experiment on the 5Re alloy at 1080 °C and 125 MPa, in which the microstructure after approximately 3 % strain and 115 h is dominated by spherical precipitates (in Appendix A3, Figure A14).

In contrast, the Ru-modified alloys exhibit plate-like precipitates despite the relatively weak partitioning of Ru between the two phases. The estimated misfit values for these alloys remain relatively large, corresponding to δ^2 values that exceed those of the Re-modified alloys (Appendix A3, Table A4). Since the elastic strain energy associated with coherent precipitates scales with δ^2 , these higher δ^2 values indicate a stronger elastic contribution in the Ru-modified alloys. Under such conditions, elastic strain fields associated with neighboring precipitates can interact and promote precipitate growth along preferred, crystallographic directions [78,152]. This favors the development of plate-like precipitates extending along two approximately orthogonal directions and results in the characteristic grid-like morphology observed after long-term aging.

This interpretation should be considered together with the coarsening analysis presented in Section 6.5. Because the coarsening-rate constant was derived from the minor precipitate axis only, the low κ values obtained for the Ru-modified alloys mainly reflect limited thickening and do not contradict the pronounced directional growth observed here. Thus, the low $\bar{b}_p$ -based κ values do not contradict the pronounced formation of plate-like precipitates; rather, they indicate that the plate-like network grows anisotropically while thickening remains limited. The mechanistic implications of this point are discussed further in Section 6.8.

A similar perspective is obtained for the previously reported Zr-modified alloy [27], in which Zr partitions strongly to the B2 phase while the precipitates remain predominantly spherical. In that case, the estimated lattice misfit in this alloy is relatively small compared to the Re- and Ru-modified alloys, which reduces the elastic contribution to the total energy and favors equiaxed precipitate shapes. These observations highlight the strong sensitivity of coherent precipitate morphologies to elastic strain energy in multiphase refractory alloys [152].

6.8 Coarsening Mechanisms of B2 Precipitates

The κ values derived from the mean minor axis $\bar{b}_p$ of the precipitates show that Re and Ru additions reduce precipitate thickening relative to the base alloy. For comparison, the previously reported Zr-modified alloy in the same base alloy system [27] is included as an additional reference case. These results show that reduced coarsening in this alloy family may result from different thermodynamic, kinetic, or morphology-related contributions depending on the alloying additions.

As introduced in Section 2.2.5, the coarsening rate constant of a multicomponent precipitation-strengthened system is governed by both thermodynamic and kinetic contributions [84,153,154]. According to Eq. (2.17), coarsening is affected by interfacial energy, precipitate volume fraction, matrix diffusivity of the rate-controlling species, and the chemical partitioning between matrix and precipitate. Among these parameters, the volume fraction of the B2 precipitates remains similar across all investigated alloys (approximately 25–30%), indicating that variations in precipitate volume fraction are unlikely to account for the pronounced differences in coarsening rate.

In complex multicomponent alloys, coarsening is generally not controlled by the elements exhibiting the largest partitioning but rather by the slowest diffusing species that participate in the coupled redistribution required for precipitate growth and dissolution [155,156]. The changes in the concentration difference ΔC_i for Ta and Mo between the base and modified alloys are relatively small, suggesting that their contributions to the coarsening rate constant remain comparable. Consequently, the observed reduction in κ value is more likely associated with changes in either the effective matrix diffusivity of the rate-controlling species or the thermodynamic contribution from the A2/B2 interface. For the previously reported Zr-modified alloy [27], the lower κ value was attributed mainly to a reduced interfacial thermodynamic contribution associated with the lower lattice misfit between A2 and B2 phases. In this case, the smaller lattice misfit reduces the elastic contribution to the total interfacial term and thereby decreases the thermodynamic driving force for coarsening [27,151].

In the Re-modified alloys, the lower κ values are more plausibly attributed to a combination of kinetic and thermodynamic effects. As established in Section 6.4, Re partitions strongly to the A2 matrix and therefore directly modifies the phase through which long-range solute transport must occur during coarsening. In Ni-based superalloys, the diffusivity of Re in the γ matrix is typically about one order of magnitude lower than that of other refractory solutes such as W and Ta at comparable elevated temperatures [78,157–159]. Although corresponding diffusion data are not available for the present alloy system, the strong partitioning of Re to the A2 matrix is therefore expected to reduce the effective atomic

mobility and may act as a kinetic bottleneck for the solute redistribution required for precipitate growth. Second, the estimated lattice misfit values summarized in Appendix A3, Table A4 indicate a decreasing trend in the magnitude of misfit for Re-modified alloys. A smaller misfit decreases the elastic contribution to the total interfacial energy and thereby reduces the thermodynamic driving force for coarsening. The reduced κ values in the Re-modified alloys are therefore most reasonably interpreted as the result of combined diffusional and misfit-related effects.

The Ru-modified alloys exhibit a different microstructural evolution. In contrast to Re, Ru exhibits only weak partitioning between the A2 and B2 phases, as shown in Section 6.4, and is therefore expected to have a less pronounced influence on the effective matrix diffusivity. The low $\bar{b}_p$ -based κ values in the Ru-modified alloys are instead most likely related to the anisotropic precipitate morphology evolution discussed in Section 6.7, where plate-like feature growth and partial interconnection indicate a dominant role of elastic interactions. Under such conditions, coarsening can no longer be described as isotropic growth of isolated precipitates. Rather, extension along the long axis may continue, while thickening along the minor axis remains limited. This is reflected by the slow increase in $\bar{b}_p$, for example from (8 ± 4) nm after 10 h to only (18 ± 6) nm after 1000 h in the 5Ru alloy.

This behavior is consistent with the expected response of coherent precipitate systems with substantial lattice misfit, in which elastic interactions between neighboring precipitates may lead to deviations from classical LSW behavior. Model studies have shown that elastic strain fields can promote directional coarsening and precipitate alignment along elastically preferred crystallographic orientations [160], while additional deviations may arise from precipitate interactions at finite volume fractions and from diffusion across coherent interfaces [161,162]. In the Ru-modified alloys, these effects are likely to contribute to the pronounced anisotropy of precipitate growth. Once plate-like precipitates and partially interconnected networks are established, overlapping elastic strain fields and geometric constraints can further hinder independent thickening and coalescence of neighboring precipitates. The particularly slow increase of the minor-axis dimension therefore indicates that elastic interactions play an important role in the coarsening behavior of these alloys.

Overall, the present results indicate that the reduced κ values originate from different dominant factors depending on the alloying addition. In the Zr-modified alloy, the reduced coarsening is mainly associated with a decreased interfacial thermodynamic contribution resulting from lower lattice misfit. In the Re-modified alloys, both reduced matrix diffusivity and lower lattice misfit are likely to contribute. In the Ru-modified alloys, the low $\bar{b}_p$ -based coarsening behavior primarily reflects anisotropic precipitate growth controlled by elastic interactions, where thickening remains limited while directional growth along the plate length

continues. These findings demonstrate that alloying additions influence coarsening through distinct thermodynamic, kinetic, and elastic-interaction effects in A2+B2 microstructures.

6.9 Comparison to Ni- and Cr-Based Superalloys

The coarsening behavior of the present A2+B2 RHEA system is compared with that reported for precipitation-strengthened Ni- and Cr-based superalloys. As shown in Figures 44c–d, the κ values of the present modified alloys are considerably lower than those reported for typical superalloys when plotted as a function of homologous temperature, either T/T_{solv} or T/T_{solidus} . This comparison indicates that the present A2+B2 RHEAs exhibit comparatively high microstructural stability under the investigated aging conditions.

In conventional $\gamma+\gamma'$ Ni-based superalloys, precipitate coarsening is generally interpreted within a well-established framework based on compositional partitioning between the γ matrix and the ordered γ' phase [84,161,163]. The γ' precipitates adopt the $L1_2$ -ordered $\text{Ni}_3(\text{Al,Ti,Ta})$ -type structure, where elements such as Al, Ti, and Ta preferentially partition to γ' , whereas elements such as Cr, Mo, and W are enriched in the γ matrix [164–166]. During precipitate growth and dissolution, these compositional differences give rise to diffusion fluxes between matrix and precipitates, and κ is often controlled by the slowest-diffusing solute species in the γ matrix [84,161,163]. By contrast, the present A2+B2 alloy system exhibits pronounced partitioning of several refractory alloying elements between the A2 matrix and B2 precipitates, as shown in Section 6.4. Precipitate growth in this system therefore requires the coupled redistribution of several alloying elements across the coherent A2/B2 interface. Compared with $\gamma+\gamma'$ superalloys, such multicomponent diffusion processes are expected to reduce the effective mass transport controlling precipitate growth and thereby contribute to the relatively low coarsening rates observed in the present alloys.

An additional distinction arises in the Ru-modified alloys, where the B2 precipitates develop plate-like features that become partially interconnected during prolonged aging. As discussed in Sections 6.7 and 6.8, this morphology promotes anisotropic growth and suppresses thickening along the minor axis. Under such conditions, the precipitate coarsening is influenced not only by classical capillarity-driven coarsening but also by elastic interactions between neighboring precipitates. Because the precipitate spacing is very small, diffusion fields surrounding individual precipitates can overlap, leading to soft impingement where the concentration gradients driving diffusion are progressively reduced [62,167]. The partial interconnection of plate-like precipitates further introduces geometric constraints comparable to hard impingement, which suppress thickening of the precipitates and thereby reduce the coarsening rate [168,169].

The comparison with Ni- and Cr-based superalloys therefore indicates the high coarsening resistance of the present A2+B2 refractory alloy system arises from the combined effects of

multicomponent partitioning, diffusion-controlled redistribution of refractory elements, and morphology-dependent restrictions on precipitate thickening. In this sense, precipitate coarsening in the present refractory A2+B2 alloys follows a mechanistic framework that differs from that established for conventional $\gamma+\gamma'$ Ni-based superalloys.

7 Conclusions

This work systematically investigates the creep behavior of the polycrystalline 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al (at.%) as one of the first RHEAs with a confirmed A2 matrix strengthened by B2 precipitates. In this context, the alloy serves as a refractory analogue to precipitation-strengthened Ni-based superalloys and provides a model system for establishing microstructure-property relationships in A2+B2 RHEAs. The work aims to identify the factors governing high-temperature creep resistance and microstructural stability and to use this understanding as a basis for alloy optimization. Based on the main results, the following conclusions can be drawn:

(1) Exceptional creep resistance of polycrystalline A2+B2 RHEAs

The polycrystalline A2+B2 refractory high-entropy alloy TMT-8Cr-10Al exhibits excellent creep resistance at 1000–1030 °C. Its minimum creep rates are three to four orders of magnitude lower than those of single-phase A2 or B2 RHEAs and are comparable to those of the single-crystalline Ni-based superalloy CMSX-4 within the stress range of 75–125 MPa. This performance is achieved despite less favorable testing conditions, namely the polycrystalline microstructure and temperatures close to the precipitate T_{solv} .

The creep deformation of TMT-8Cr-10Al is primarily controlled by the A2 matrix, while B2 precipitates provide a measurable but moderate strengthening contribution to the overall creep resistance. Although the precipitate strengthening contribution is limited in magnitude, it plays an essential role in modifying the creep response and stabilizing the microstructure under deformation. High creep resistance in A2+B2 RHEAs therefore arises from the combined effect of a strong matrix and an effective, though not dominant, precipitation-strengthening effect.

(2) Creep-induced rafting in A2+B2 RHEAs

Directional coarsening of B2 precipitates (N-type rafting) was observed for the first time in specifically oriented grains of a polycrystalline A2+B2 RHEA under high-temperature creep conditions. The observed rafting behavior is consistent with the positive lattice misfit and compressive loading conditions. These results demonstrate that misfit-driven elastic interactions govern the directional morphology evolution of the precipitates during creep.

(3) Three-dimensional representation of rafted B2 precipitates

The quasi-three-dimensional representation of the rafted B2 precipitates indicates that the precipitates form laterally extended plate-like structures while the A2 matrix remains interconnected through channel regions rather than being effectively blocked by the

precipitates. This geometry implies that precipitation strengthening is governed not only by precipitate presence, but also by the spatial connectivity of the matrix channels that control dislocation transport during creep.

(4) Dislocation-precipitate interactions after creep at 1030 °C

TEM observations at 1030 °C reveal different local deformation substructures among the selected creep conditions. At the low stress of 100 MPa, the observations are consistent with localized climb-assisted bypass of B2 precipitates. At intermediate stress of 125 MPa, dislocation configurations consistent with both climb-assisted bypass and precipitate cutting are observed. At the higher stress of 150 MPa, extended dislocation segments in the A2 channels with repeated changes in line direction become increasingly prominent. These results indicate that creep deformation in A2+B2 RHEAs cannot be described by a single interaction mode, but instead involves a combination of dislocation-precipitate interaction processes whose relative importance changes with creep conditions.

(5) High interfacial stability under creep conditions

High-resolution microscopy confirms that the A2/B2 interfaces remain fully coherent after creep across the investigated stress range. No evidence of interfacial decohesion or loss of coherency was found in the examined regions. The stress-dependent creep behavior is therefore associated primarily with changes in dislocation activity and dislocation-precipitate interactions rather than with interfacial degradation.

(6) Enhancement of microstructural stability by Re and Ru additions

Dilute additions of Re and Ru both significantly enhance the microstructural stability of precipitates in A2+B2 TMT-8Cr-10Al-based alloys. Both elements increase the ordering temperature and the solvus temperature and substantially reduce the coarsening rate during long-term thermal exposure at 1000 °C. However, the resulting precipitate morphologies differ markedly: Re additions promote a transition toward more equiaxed precipitates consistent with reduced lattice misfit, whereas Ru additions lead to plate-like precipitates aligned along perpendicular directions, indicating strong elastic interactions.

(7) Distinct alloying factors controlling precipitate stability

Re and Ru stabilize the A2+B2 microstructure through different dominant factors. Re partitions strongly to the A2 matrix and is therefore expected to reduce effective matrix diffusivity, while also lowering the lattice misfit and thus the thermodynamic driving force for coarsening. Ru, by contrast, partitions only weakly, but promotes morphology changes associated with stronger elastic interactions and anisotropic precipitate growth. These results demonstrate that precipitate stabilization in A2+B2 RHEAs can be achieved through different alloying concepts involving diffusion kinetics, lattice misfit, and elastic interactions.

(8) Decoupling of partitioning behavior and thermodynamic stabilization

The present results show that thermodynamic stabilization of the B2 precipitates cannot be inferred simply from whether an alloying element enriches the ordered phase. Re partitions strongly to the matrix, Ru partitions only weakly, and Zr partitions strongly to the B2 phase, yet these alloying additions do not affect T_{solv} , T_{ord} , and coarsening behavior in the same way. This demonstrates that the relationship between alloy chemistry, partitioning, and precipitate stability in multicomponent A2+B2 alloys is more complex than in conventional binary or ternary model systems.

(9) Distinct coarsening mechanisms in modified alloys

The reduced coarsening rates derived from the precipitate minor axis in the modified alloys arise from different dominant factors. In the Zr-modified alloy, the reduced coarsening is mainly associated with a lower lattice misfit and a reduced elastic contribution to the interfacial driving force. In the Re-modified alloys, both reduced matrix diffusivity and lower lattice misfit are likely to contribute. In the Ru-modified alloys, the low apparent coarsening rates mainly reflect anisotropic precipitate growth and morphology-dependent restrictions on thickening. These findings show that precipitate coarsening in A2+B2 RHEAs is controlled by a combination of thermodynamic, kinetic, and elastic-interaction effects.

In summary, this thesis demonstrates that precipitation-strengthened A2+B2 RHEAs constitute a promising platform for next-generation high-temperature structural materials. By establishing quantitative links between solvus stability, precipitate morphology, creep deformation behavior, and alloy chemistry, this work not only advances the fundamental understanding of A2+B2 RHEAs but also defines concrete design principles for overcoming current limitations and further enhancing their high-temperature performance. At the same time, several open aspects remain that are critical for the further development of this alloy class. In particular, the quantitative role of lattice misfit in controlling rafting kinetics and its evolution with temperature and composition remains to be clarified, as it directly governs the stress-driven microstructural evolution during creep. Furthermore, although Re and Ru additions were shown to improve precipitate stability and modify coarsening behavior, their direct impact on creep behavior has not yet been investigated and should be addressed in future work. In addition, the oxidation behavior at elevated temperatures has not yet been addressed, although it is expected to play a decisive role for the applicability of these alloys under service conditions. Addressing these aspects will be essential for further advancing the fundamental understanding and guiding the development of A2+B2 RHEAs toward improved high-temperature performance.

8 References

- [1] J.H. Perepezko, The hotter the engine, the better, *Science* 326 (2009) 1068–1069. <https://doi.org/10.1126/science.1179327>.
- [2] T. M. Pollock, S. Tin, Nickel-based superalloys for advanced turbine engines: chemistry, microstructure and properties, *Journal of Propulsion and Power* 22 (2006) 361–374. <https://doi.org/10.2514/1.18239>.
- [3] F. Schleifer, M. Holzinger, Y.-Y. Lin, U. Glatzel, M. Fleck, Phase-field modeling of γ/γ'' microstructure formation in Ni-based superalloys with high γ'' volume fraction, *Intermetallics* 120 (2020) 106745. <https://doi.org/10.1016/j.intermet.2020.106745>.
- [4] R.A. Ricks, A.J. Porter, R.C. Ecoh, The growth of γ' precipitates in nickel-base superalloys, *Acta Metallurgica* 31 (1983) 43–53. [https://doi.org/10.1016/0001-6160\(83\)90062-7](https://doi.org/10.1016/0001-6160(83)90062-7).
- [5] P.M. Sarosi, R. Srinivasan, G.F. Eggeler, M.V. Nathal, M.J. Mills, Observations of a $\langle 010 \rangle$ dislocations during the high-temperature creep of Ni-based superalloy single crystals deformed along the $[001]$ orientation, *Acta Materialia* 55 (2007) 2509–2518. <https://doi.org/10.1016/j.actamat.2006.11.045>.
- [6] X. Wu, S.K. Makineni, C.H. Liebscher, G. Dehm, J. Rezaei Mianroodi, P. Shanthraj, B. Svendsen, D. Bürger, G. Eggeler, D. Raabe, B. Gault, Unveiling the Re effect in Ni-based single crystal superalloys, *Nature Communications* 11 (2020) 389. <https://doi.org/10.1038/s41467-019-14062-9>.
- [7] J.-P. Couzinié, O.N. Senkov, D.B. Miracle, G. Dirras, Comprehensive data compilation on the mechanical properties of refractory high-entropy alloys, *Data in Brief* 21 (2018) 1622–1641. <https://doi.org/10.1016/j.dib.2018.10.071>.
- [8] O.N. Senkov, D.B. Miracle, K.J. Chaput, J.-P. Couzinie, Development and exploration of refractory high entropy alloys—A review, *Journal of Materials Research* 33 (2018) 3092–3128. <https://doi.org/10.1557/jmr.2018.153>.
- [9] J.-W. Yeh, S.-K. Chen, S.-J. Lin, J.-Y. Gan, T.-S. Chin, T.-T. Shun, C.-H. Tsau, S.-Y. Chang, Nanostructured high-entropy alloys with multiple principal elements: novel alloy design concepts and outcomes, *Advanced Engineering Materials* 6 (2004) 299–303. <https://doi.org/10.1002/adem.200300567>.
- [10] S. Naka, T. Khan, Designing novel multiconstituent intermetallies: contribution of modern alloy theory in developing engineered materials, *Journal of Phase Equilibria* 18 (1997) 635–649. <https://doi.org/10.1007/bf02665823>.
- [11] O.N. Senkov, G.B. Wilks, J.M. Scott, D.B. Miracle, Mechanical properties of Nb₂₅Mo₂₅Ta₂₅W₂₅ and V₂₀Nb₂₀Mo₂₀Ta₂₀W₂₀ refractory high entropy alloys, *Intermetallics* 19 (2011) 698–706. <https://doi.org/10.1016/j.intermet.2011.01.004>.
- [12] V. Soni, B. Gwalani, T. Alam, S. Dasari, Y. Zheng, O.N. Senkov, D. Miracle, R. Banerjee, Phase inversion in a two-phase, BCC+B₂, refractory high entropy alloy, *Acta Materialia* 185 (2020) 89–97. <https://doi.org/10.1016/j.actamat.2019.12.004>.

- [13] S. Laube, S. Schellert, A. Srinivasan Tirunilai, D. Schliephake, B. Gorr, H.-J. Christ, A. Kauffmann, M. Heilmaier, Microstructure tailoring of Al-containing compositionally complex alloys by controlling the sequence of precipitation and ordering, *Acta Materialia* 218 (2021) 117217. <https://doi.org/10.1016/j.actamat.2021.117217>.
- [14] D.B. Miracle, O.N. Senkov, A critical review of high entropy alloys and related concepts, *Acta Materialia* 122 (2017) 448–511. <https://doi.org/10.1016/j.actamat.2016.08.081>.
- [15] C.-J. Liu, C. Gadelmeier, S.-L. Lu, J.-W. Yeh, H.-W. Yen, S. Gorsse, U. Glatzel, A.-C. Yeh, Tensile creep behavior of HfNbTaTiZr refractory high entropy alloy at elevated temperatures, *Acta Materialia* 237 (2022) 118188. <https://doi.org/10.1016/j.actamat.2022.118188>.
- [16] C. Gadelmeier, Y. Yang, U. Glatzel, E.P. George, Creep strength of refractory high-entropy alloy TiZrHfNbTa and comparison with Ni-base superalloy CMSX-4, *Cell Reports Physical Science* 3 (2022) 100991. <https://doi.org/10.1016/j.xcrp.2022.100991>.
- [17] H.W. Yao, J.W. Qiao, J.A. Hawk, H.F. Zhou, M.W. Chen, M.C. Gao, Mechanical properties of refractory high-entropy alloys: experiments and modeling, *Journal of Alloys and Compounds* 696 (2017) 1139–1150. <https://doi.org/10.1016/j.jallcom.2016.11.188>.
- [18] V. Soni, O.N. Senkov, B. Gwalani, D.B. Miracle, R. Banerjee, Microstructural design for improving ductility of an initially brittle refractory high entropy alloy, *Scientific Reports* 8 (2018) 8816. <https://doi.org/10.1038/s41598-018-27144-3>.
- [19] J. Qi, X. Fan, D.I. Hoyos, M. Widom, P.K. Liaw, J. Poon, Integrated design of aluminum-enriched high-entropy refractory B2 alloys with synergy of high strength and ductility, *Science Advances* 10 (2024) eadq0083. <https://doi.org/10.1126/sciadv.adq0083>.
- [20] H. Chen, A. Kauffmann, S. Laube, I.-C. Choi, R. Schwaiger, Y. Huang, K. Lichtenberg, F. Müller, B. Gorr, H.-J. Christ, M. Heilmaier, Contribution of lattice distortion to solid solution strengthening in a series of refractory high entropy alloys, *Metallurgical and Materials Transactions A* 49 (2018) 772–781. <https://doi.org/10.1007/s11661-017-4386-1>.
- [21] O.N. Senkov, D. Isheim, D.N. Seidman, A.L. Pilchak, Development of a refractory high entropy superalloy, *Entropy* 18 (2016) 102. <https://doi.org/10.3390/e18030102>.
- [22] D.B. Miracle, M.-H. Tsai, O.N. Senkov, V. Soni, R. Banerjee, Refractory high entropy superalloys (RSAs), *Scripta Materialia* 187 (2020) 445–452. <https://doi.org/10.1016/j.scriptamat.2020.06.048>.
- [23] Y. Zhang, T.T. Zuo, Z. Tang, M.C. Gao, K.A. Dahmen, P.K. Liaw, Z.P. Lu, Microstructures and properties of high-entropy alloys, *Progress in Materials Science* 61 (2014) 1–93. <https://doi.org/10.1016/j.pmatsci.2013.10.001>.
- [24] O.N. Senkov, B. Crossman, S.I. Rao, J.P. Couzinie, D.B. Miracle, T.M. Butler, R. Banerjee, M. Mills, Mechanical properties of an Al₁₀Nb₂₀Ta₁₅Ti₃₀V₅Zr₂₀ A2/B2 refractory superalloy and its constituent phases, *Acta Materialia* 254 (2023) 119017. <https://doi.org/10.1016/j.actamat.2023.119017>.
- [25] S. Laube, A. Kauffmann, S. Schellert, S. Seils, A.S. Tirunilai, C. Greiner, Y.M. Eggeler, B. Gorr, H.-J. Christ, M. Heilmaier, Formation and thermal stability of two-phase

- microstructures in Al-containing refractory compositionally complex alloys, *Science and Technology of Advanced Materials* 23 (2022) 692–706. <https://doi.org/10.1080/14686996.2022.2132118>.
- [26] D. Bürger, A.B. Parsa, M. Ramsperger, C. Körner, G. Eggeler, Creep properties of single crystal Ni-base superalloys (SX): a comparison between conventionally cast and additive manufactured CMSX-4 materials, *Materials Science and Engineering A* 762 (2019) 138098. <https://doi.org/10.1016/j.msea.2019.138098>.
- [27] R.J. Vikram, S. Sen, L. Yang, M.K. Eusterholz, A. Radi, D. Schliephake, J.-P. Couzinié, A. Kauffmann, M. Heilmaier, Ultra-slow coarsening in precipitation-strengthened refractory high-entropy alloys, *Scripta Materialia* 271 (2026) 117026. <https://doi.org/10.1016/j.scriptamat.2025.117026>.
- [28] A.-C. Fan, Y.-S. Chen, C.-C. Chi, D.B. Miracle, J. Shiue, P.-C. Kuo, C.-H. Hsu, K.-C. Yang, M.-Y. Lu, M.-H. Tsai, Stability of the B2 phase in Al-Nb-Ta-Ti-Zr refractory high-entropy superalloys: resolving identification conflicts and offering practical solutions, *Journal of Alloys and Compounds* 1021 (2025) 179591. <https://doi.org/10.1016/j.jallcom.2025.179591>.
- [29] M. Feuerbacher, Dislocations and deformation microstructure in a B2-ordered Al₂₈Co₂₀Cr₁₁Fe₁₅Ni₂₆ high-entropy alloy, *Scientific Reports* 6 (2016) 29700. <https://doi.org/10.1038/srep29700>.
- [30] A. Bezold, N. Volz, F. Xue, C.H. Zenk, S. Neumeier, M. Göken, On the precipitation-strengthening contribution of the Ta-containing Co₃(Al,W)-phase to the creep properties of γ/γ' Cobalt-base superalloys, *Metallurgical and Materials Transactions A* 51 (2020) 1567–1574. <https://doi.org/10.1007/s11661-020-05626-2>.
- [31] R. Lagneborg, B. Bergman, The stress/creep rate behaviour of precipitation-hardened alloys, *Metal Science* 10 (1976) 20–28. <https://doi.org/10.1179/030634576790431462>.
- [32] B. Reppich, M. Heilmaier, K. Liebig, G. Schumann, K.-D. Stein, T. Woller, Microstructural modelling of the creep behaviour of particle-strengthened superalloys, *Steel Research* 61 (1990) 251–257. <https://doi.org/10.1002/srin.199000343>.
- [33] J. Wang, A. Barooni, M. Ghazisaeidi, Stability of the B2 phase among refractory metals, *Acta Materialia* 279 (2024) 120323. <https://doi.org/10.1016/j.actamat.2024.120323>.
- [34] S.A. Kube, C. Frey, C. McMullin, B. Neuman, K.M. Mullin, T.M. Pollock, Navigating the BCC-B2 refractory alloy space: stability and thermal processing with Ru-B2 precipitates, *Acta Materialia* 265 (2024) 119628. <https://doi.org/10.1016/j.actamat.2023.119628>.
- [35] C. Frey, B. Neuman, K. Mullin, A. Botros, J. Lamb, C.S. Holgate, S.A. Kube, T.M. Pollock, On the stability of coherent HfRu- and ZrRu-B2 precipitates in Nb-based alloys, *Materials & Design* 247 (2024) 113385. <https://doi.org/10.1016/j.matdes.2024.113385>.
- [36] B.J. Crossman, J. Wang, L. Perrière, S.A. Chen, J.-P. Couzinié, M. Ghazisaeidi, M.J. Mills, Multi-modal characterization of the B2 phase in the Ta-Re binary system, *Acta Materialia* 293 (2025) 121097. <https://doi.org/10.1016/j.actamat.2025.121097>.

- [37] B. Cantor, I.T.H. Chang, P. Knight, A.J.B. Vincent, Microstructural development in equiatomic multicomponent alloys, *Materials Science and Engineering: A* 375 (2004) 213–218. <https://doi.org/10.1016/j.msea.2003.10.257>.
- [38] W. Steurer, Single-phase high-entropy alloys – a critical update, *Materials Characterization* 162 (2020) 110179. <https://doi.org/10.1016/j.matchar.2020.110179>.
- [39] C.K.H. Borg, C. Frey, J. Moh, T.M. Pollock, S. Gorsse, D.B. Miracle, O.N. Senkov, B. Meredig, J.E. Saal, Expanded dataset of mechanical properties and observed phases of multi-principal element alloys, *Scientific Data* 7 (2020) 430. <https://doi.org/10.1038/s41597-020-00768-9>.
- [40] B. Gorr, S. Schellert, F. Müller, H.-J. Christ, A. Kauffmann, M. Heilmaier, Current status of research on the oxidation behavior of refractory high entropy alloys, *Advanced Engineering Materials* 23 (2021) 2001047. <https://doi.org/10.1002/adem.202001047>.
- [41] B. Gorr, F. Müller, M. Azim, H.-J. Christ, T. Müller, H. Chen, A. Kauffmann, M. Heilmaier, High-temperature oxidation behavior of refractory high-entropy alloys: effect of alloy composition, *Oxidation of Metals* 88 (2017) 339–349. <https://doi.org/10.1007/s11085-016-9696-y>.
- [42] B. Gorr, M. Azim, H.-J. Christ, T. Müller, D. Schliephake, M. Heilmaier, Phase equilibria, microstructure, and high temperature oxidation resistance of novel refractory high-entropy alloys, *Journal of Alloys and Compounds* 624 (2015) 270–278. <https://doi.org/10.1016/j.jallcom.2014.11.012>.
- [43] O.N. Senkov, S.V. Senkova, C. Woodward, Effect of aluminum on the microstructure and properties of two refractory high-entropy alloys, *Acta Materialia* 68 (2014) 214–228. <https://doi.org/10.1016/j.actamat.2014.01.029>.
- [44] H. Chen, A. Kauffmann, S. Seils, T. Boll, C.H. Liebscher, I. Harding, K.S. Kumar, D.V. Szabó, S. Schlabach, S. Kauffmann-Weiss, F. Müller, B. Gorr, H.-J. Christ, M. Heilmaier, Crystallographic ordering in a series of Al-containing refractory high entropy alloys Ta–Nb–Mo–Cr–Ti–Al, *Acta Materialia* 176 (2019) 123–133. <https://doi.org/10.1016/j.actamat.2019.07.001>.
- [45] Q. Gao, S. Guo, Z. Hu, Q. Guan, J. Guan, S. Guo, H. Liu, G. Liu, X. Liu, Q. Zhang, P. Lyu, Al content regulation of microstructure, wear resistance, and corrosion behavior in Mo_{0.5}NbTiZrAl_x high-entropy alloys, *Journal of Alloys and Compounds* 1017 (2025) 179037. <https://doi.org/10.1016/j.jallcom.2025.179037>.
- [46] S. Dasari, V. Soni, A. Sharma, O.N. Senkov, D.B. Miracle, H.L. Fraser, Y. Wang, R. Banerjee, Concomitant clustering and ordering leading to B2 + BCC microstructures in refractory high entropy alloys, *Transactions of the Indian Institute of Metals* 75 (2022) 907–916. <https://doi.org/10.1007/s12666-021-02518-w>.
- [47] J.K. Jensen, B.A. Welk, R.E.A. Williams, J.M. Sosa, D.E. Huber, O.N. Senkov, G.B. Viswanathan, H.L. Fraser, Characterization of the microstructure of the compositionally complex alloy Al₁Mo_{0.5}Nb₁Ta_{0.5}Ti₁Zr₁, *Scripta Materialia* 121 (2016) 1–4. <https://doi.org/10.1016/j.scriptamat.2016.04.017>.

- [48] O.N. Senkov, C. Woodward, D.B. Miracle, Microstructure and properties of aluminum-containing refractory high-entropy alloys, *JOM* 66 (2014) 2030–2042. <https://doi.org/10.1007/s11837-014-1066-0>.
- [49] T.E. Whitfield, E.J. Pickering, L.R. Owen, C.N. Jones, H.J. Stone, N.G. Jones, The effect of Al on the formation and stability of a BCC – B2 microstructure in a refractory metal high entropy superalloy system, *Materialia* 13 (2020) 100858. <https://doi.org/10.1016/j.mtla.2020.100858>.
- [50] S. Laube, G. Winkens, A. Kauffmann, J. Li, C. Kirchlechner, M. Heilmaier, Strength of disordered and ordered Al-containing refractory high-entropy alloys, *Advanced Engineering Materials* 26 (2024) 2301797. <https://doi.org/10.1002/adem.202301797>.
- [51] Z.D. Han, H.W. Luan, X. Liu, N. Chen, X.Y. Li, Y. Shao, K.F. Yao, Microstructures and mechanical properties of $\text{Ti}_{1-x}\text{NbMoTaW}$ refractory high-entropy alloys, *Materials Science and Engineering: A* 712 (2018) 380–385. <https://doi.org/10.1016/j.msea.2017.12.004>.
- [52] E.P. George, D. Raabe, R.O. Ritchie, High-entropy alloys, *Nature Reviews Materials* 4 (2019) 515–534. <https://doi.org/10.1038/s41578-019-0121-4>.
- [53] J.W. Christian, Some surprising features of the plastic deformation of body-centered cubic metals and alloys, *Metallurgical Transactions A* 14 (1983) 1237–1256. <https://doi.org/10.1007/BF02664806>.
- [54] D. Caillard, J.L. Martin, Thermally activated mechanisms in crystal plasticity, *MRS Bulletin* 30 (2005) 318–320. <https://doi.org/10.1557/mrs2005.94>.
- [55] R. Nakamura, K. Takasawa, Y. Yamazaki, Y. Iijima, Single-phase interdiffusion in the B2 type intermetallic compounds NiAl, CoAl and FeAl, *Intermetallics* 10 (2002) 195–204. [https://doi.org/10.1016/S0966-9795\(01\)00125-X](https://doi.org/10.1016/S0966-9795(01)00125-X).
- [56] R.D. Noebe, R.R. Bowman, M.V. Nathal, Physical and mechanical properties of the B2 compound NiAl, *International Materials Reviews* 38 (1993) 193–232. <https://doi.org/10.1179/imr.1993.38.4.193>.
- [57] N.S. Stoloff, R.G. Davies, The mechanical properties of ordered alloys, *Progress in Materials Science* 13 (1968) 1–84. [https://doi.org/10.1016/0079-6425\(68\)90018-2](https://doi.org/10.1016/0079-6425(68)90018-2).
- [58] I. Baker, A review of the mechanical properties of B2 compounds, *Materials Science and Engineering: A* 192–193 (1995) 1–13. [https://doi.org/10.1016/0921-5093\(94\)03200-9](https://doi.org/10.1016/0921-5093(94)03200-9).
- [59] M. Yamaguchi, H. Inui, K. Ito, High-temperature structural intermetallics, *Acta Materialia* 48 (2000) 307–322. [https://doi.org/10.1016/S1359-6454\(99\)00301-8](https://doi.org/10.1016/S1359-6454(99)00301-8).
- [60] R. Darolia, NiAl alloys for high-temperature structural applications, *JOM* 43 (1991) 44–49. <https://doi.org/10.1007/BF03220163>.
- [61] C.-G. Oertel, R. Schaarschuch, G.H. Cao, H.N. Tian, J. Freudenberger, H.-G. Brokmeier, W. Skrotzki, Effect of martensitic phase transformation on the ductility of polycrystalline YCu, *Scripta Materialia* 65 (2011) 779–782. <https://doi.org/10.1016/j.scriptamat.2011.07.030>.

- [62] A.J. Ardell, Precipitation hardening, *Metallurgical Transactions A* 16 (1985) 2131–2165. <https://doi.org/10.1007/BF02670416>.
- [63] N.S. Stoloff, V.K. Sikka, *Physical metallurgy and processing of Intermetallic Compounds*, Springer, Boston, MA, 2012.
- [64] X.S. Liu, P.F. Yu, R. Li, A.X. Li, S.B. Yu, M.H. Jiang, J.S. Zhang, C.N. Che, D. Huang, G. Li, Investigating the strengthening and deformation behavior of B2-strengthened high entropy alloys with high Co and Al contents, *Materials Science and Engineering: A* 887 (2023) 145756. <https://doi.org/10.1016/j.msea.2023.145756>.
- [65] S. Banerjee, P. Mukhopadhyay, *Phase transformations: examples from titanium and zirconium alloys*, Elsevier, Oxford, 2010.
- [66] J. Weertman, Steady-state creep through dislocation climb, *Journal of Applied Physics* 28 (1957) 362–364. <https://doi.org/10.1063/1.1722747>.
- [67] R. Raj, M.F. Ashby, On grain boundary sliding and diffusional creep, *Metallurgical Transactions* 2 (1971) 1113–1127. <https://doi.org/10.1007/BF02664244>.
- [68] J.G. Harper, L.A. Shepard, J.E. Dorn, Creep of aluminum under extremely small stresses, *Acta Metallurgica* 6 (1958) 509–518. [https://doi.org/10.1016/0001-6160\(58\)90114-7](https://doi.org/10.1016/0001-6160(58)90114-7).
- [69] S. Gavras, S. Zhu, M.A. Easton, M.A. Gibson, H. Dieringa, Compressive creep behavior of high-pressure die-cast aluminum-containing magnesium alloys developed for elevated temperature applications, *Frontiers in Materials* 6 (2019) 262. <https://doi.org/10.3389/fmats.2019.00262>.
- [70] B.F. Dyson, Creep and fracture of metals: mechanisms and mechanics, *Revue de Physique Appliquée* 23 (1988) 605–613. <https://doi.org/10.1051/rphysap:01988002304060500>.
- [71] M.E. Kassner, *Fundamentals of creep in metals and alloys*, Butterworth-Heinemann, Oxford, 2015.
- [72] M. Heilmaier, B. Reppich, On the microstructural origin of primary creep in nickel-base superalloys, *Materials Science and Engineering: A* 234–236 (1997) 501–504. [https://doi.org/10.1016/S0921-5093\(97\)00258-X](https://doi.org/10.1016/S0921-5093(97)00258-X).
- [73] H. Mughrabi, Revisiting “steady-state” monotonic and cyclic deformation: emphasizing the quasi-stationary state of deformation, *Metallurgical and Materials Transactions A* 51 (2020) 1441–1456. <https://doi.org/10.1007/s11661-019-05618-x>.
- [74] A.K. Mukherjee, J.E. Bird, J.E. Dorn, Experimental correlations for high-temperature creep, *Transactions of the Metallurgical Society of AIME* 242 (1969) 1559–1566.
- [75] H. Nakajima, K. Nonaka, W. Sprengel, M. Koiwa, Self-diffusion and interdiffusion in intermetallic compounds, *Materials Science and Engineering: A* 239–240 (1997) 819–827. [https://doi.org/10.1016/S0921-5093\(97\)00672-2](https://doi.org/10.1016/S0921-5093(97)00672-2).
- [76] G. Neumann, C. Tuijn, *Self-diffusion and impurity diffusion in pure metals: handbook of experimental data*, Elsevier, Oxford, 2011.

- [77] W. Blum, B. Reppich, On the stress dependence of the stationary deformation rate, *Acta Metallurgica* 17 (1969) 959–966. [https://doi.org/10.1016/0001-6160\(69\)90040-6](https://doi.org/10.1016/0001-6160(69)90040-6).
- [78] R.C. Reed, *The superalloys: fundamentals and applications*, Cambridge University Press, Cambridge, 2008.
- [79] N. Volz, F. Xue, A. Bezold, C.H. Zenk, S.G. Fries, J. Schreuer, S. Neumeier, M. Göken, Design of a Co–Al–W–Ta alloy series with varying γ' volume fraction and their thermophysical properties, *Metallurgical and Materials Transactions A* 52 (2021) 3931–3944. <https://doi.org/10.1007/s11661-021-06353-y>.
- [80] E. Nembach, G. Neite, Precipitation hardening of superalloys by ordered γ' -particles, *Progress in Materials Science* 29 (1985) 177–319. [https://doi.org/10.1016/0079-6425\(85\)90001-5](https://doi.org/10.1016/0079-6425(85)90001-5).
- [81] B. Reppich, Particle strengthening, *Materials Science and Technology* 6 (1993) 311–357.
- [82] E. Orowan, Internal stresses in metals and alloys, in: *Symposium on Internal Stresses in Metals and Alloys*, Institute of Metals, London, 1948: pp. 451–453.
- [83] D.J. Bacon, U.F. Kocks, R.O. Scattergood, The effect of dislocation self-interaction on the orowan stress, *Philosophical Magazine* 28 (1973) 1241–1263. <https://doi.org/10.1080/14786437308227997>.
- [84] I.M. Lifshitz, V.V. Slyozov, The kinetics of precipitation from supersaturated solid solutions, *Journal of Physics and Chemistry of Solids* 19 (1961) 35–50. [https://doi.org/10.1016/0022-3697\(61\)90054-3](https://doi.org/10.1016/0022-3697(61)90054-3).
- [85] B. Niethammer, Derivation of the LSW-theory for ostwald ripening by homogenization methods, *Archive for Rational Mechanics and Analysis* 147 (1999) 119–178. <https://doi.org/10.1007/s002050050147>.
- [86] K. Kim, P.W. Voorhees, Ostwald ripening of spheroidal particles in multicomponent alloys, *Acta Materialia* 152 (2018) 327–337. <https://doi.org/10.1016/j.actamat.2018.04.041>.
- [87] L. Agudo Jácome, P. Nörtershäuser, C. Somsen, A. Dlouhý, G. Eggeler, On the nature of γ' phase cutting and its effect on high temperature and low stress creep anisotropy of Ni-base single crystal superalloys, *Acta Materialia* 69 (2014) 246–264. <https://doi.org/10.1016/j.actamat.2014.01.021>.
- [88] A. Altincekic, E. Balikci, Precipitate rafting in a polycrystalline superalloy during compression creep, *Metallurgical and Materials Transactions A* 45 (2014) 5923–5936. <https://doi.org/10.1007/s11661-014-2558-9>.
- [89] D. Barba, A. Egan, S. Utada, Y. Gong, Y.T. Tang, V. Mazanova, M.J. Mills, R.C. Reed, Deformation mechanisms rationalisation to design for creep resistance in polycrystalline Ni-based superalloys, *Metallurgical and Materials Transactions A* 54 (2023) 1886–1901. <https://doi.org/10.1007/s11661-022-06922-9>.

- [90] Y.M. Eggeler, M.S. Titus, A. Suzuki, T.M. Pollock, Creep deformation-induced antiphase boundaries in L12-containing single-crystal cobalt-base superalloys, *Acta Materialia* 77 (2014) 352–359. <https://doi.org/10.1016/j.actamat.2014.04.037>.
- [91] A.I. Epishin, B. Fedelich, B. Viguier, S. Schrieffer, I.L. Svetlov, N.V. Petrushin, R. Saillard, A. Proietti, D. Poquillon, A. Chyrkin, Creep of single-crystals of nickel-base γ -alloy at temperatures between 1150 °C and 1288 °C, *Materials Science and Engineering: A* 825 (2021) 141880. <https://doi.org/10.1016/j.msea.2021.141880>.
- [92] S.M. Hafez Haghighat, G. Eggeler, D. Raabe, Effect of climb on dislocation mechanisms and creep rates in γ' -strengthened Ni base superalloy single crystals: a discrete dislocation dynamics study, *Acta Materialia* 61 (2013) 3709–3723. <https://doi.org/10.1016/j.actamat.2013.03.003>.
- [93] F. Xue, H.J. Zhou, Q.Y. Shi, X.H. Chen, H. Chang, M.L. Wang, Q. Feng, Creep behavior in a γ' strengthened Co–Al–W–Ta–Ti single-crystal alloy at 1000 °C, *Scripta Materialia* 97 (2015) 37–40. <https://doi.org/10.1016/j.scriptamat.2014.10.015>.
- [94] A. Bauer, S. Neumeier, F. Pyczak, R.F. Singer, M. Göken, Creep properties of different γ' -strengthened Co-base superalloys, *Materials Science and Engineering A* 550 (2012) 333–341. <https://doi.org/10.1016/j.msea.2012.04.083>.
- [95] A. Bauer, S. Neumeier, F. Pyczak, M. Göken, Creep strength and microstructure of polycrystalline γ' -strengthened cobalt-base superalloys, *Superalloys* 12 (2012) 695–703. https://doi.org/10.7449/2012/Superalloys_2012_695_703.
- [96] M. Kamaraj, Rafting in single crystal nickel-base superalloys — An overview, *Sadhana* 28 (2003) 115–128. <https://doi.org/10.1007/BF02717129>.
- [97] T. Saito, H. Harada, T. Yokokawa, M. Osawa, K. Kawagishi, S. Suzuki, Raft structure of nickel base single-crystal superalloys, *Materials Transactions* 65 (2024) 1443–1457. <https://doi.org/10.2320/matertrans.MT-M2024081>.
- [98] L. Yang, S. Sen, D. Schliephake, R.J. Vikram, S. Laube, A. Pramanik, A. Chauhan, S. Neumeier, M. Heilmaier, A. Kauffmann, Creep behavior of a precipitation-strengthened A2-B2 refractory high entropy alloy, *Acta Materialia* 288 (2025) 120827. <https://doi.org/10.1016/j.actamat.2025.120827>.
- [99] W.H. Bragg, W.L. Bragg, The reflection of X-rays by crystals, *Proceedings of the Royal Society A* 88 (1913) 428–438. <https://doi.org/10.1098/rspa.1913.0040>.
- [100] J.B. Nelson, D.P. Riley, An experimental investigation of extrapolation methods in the derivation of accurate unit-cell dimensions of crystals, *Proceedings of the Physical Society* 57 (1945) 160. <https://doi.org/10.1088/0959-5309/57/3/302>.
- [101] C.A. Schneider, W.S. Rasband, K.W. Eliceiri, NIH Image to ImageJ: 25 years of image analysis, *Nature Methods* 9 (2012) 671–675. <https://doi.org/10.1038/nmeth.2089>.
- [102] R.W. Balluffi, L.L. Seigle, Growth of voids in metals during diffusion and creep, *Acta Metallurgica* 5 (1957) 449–454. [https://doi.org/10.1016/0001-6160\(57\)90063-9](https://doi.org/10.1016/0001-6160(57)90063-9).
- [103] S. Han, S. Laube, Z. Chen, A. Kauffmann, M. Heilmaier, H. Inui, Operative slip system and dislocation behavior in a brittle refractory high-entropy alloy with the B2 ordered

- structure, *Journal of Alloys and Compounds* 1020 (2025) 179361. <https://doi.org/10.1016/j.jallcom.2025.179361>.
- [104] G.B. Viswanathan, Z.T. Kloenne, J.-P. Couzinié, B.A. Welk, S.J. Kuhr, H.L. Fraser, Deformation mechanisms and their role in the lack of ductility in the refractory-based high entropy alloy AlMo0.5NbTa0.5TiZr, *Acta Materialia* 269 (2024) 119824. <https://doi.org/10.1016/j.actamat.2024.119824>.
- [105] J.-P. Couzinié, M. Heczko, V. Mazánová, O.N. Senkov, M. Ghazisaeidi, R. Banerjee, M.J. Mills, High-temperature deformation mechanisms in a BCC+B2 refractory complex concentrated alloy, *Acta Materialia* 233 (2022) 117995. <https://doi.org/10.1016/j.actamat.2022.117995>.
- [106] H. Chen, A. Kauffmann, M. Heilmaier, Unpublished results, (2019).
- [107] D.K. Matlock, W.D. Nix, The effect of sample size on the steady state creep characteristics of Ni-6 pct W, *Metallurgical Transactions* 5 (1974) 1401–1412. <https://doi.org/10.1007/BF02646626>.
- [108] M. Heilmaier, M. Krüger, H. Saage, J. Rösler, D. Mukherji, U. Glatzel, R. Völkl, R. Hüttner, G. Eggeler, Ch. Somsen, T. Depka, H.-J. Christ, B. Gorr, S. Burk, Metallic materials for structural applications beyond nickel-based superalloys, *JOM* 61 (2009) 61–67. <https://doi.org/10.1007/s11837-009-0106-7>.
- [109] A. Epishin, B. Fedelich, G. Nolze, S. Schrieffer, T. Feldmann, M.F. Ijaz, B. Viguier, D. Poquillon, Y. Le Bouar, A. Ruffini, A. Finel, Creep of single crystals of nickel-based superalloys at ultra-high homologous temperature, *Metallurgical and Materials Transactions A* 49 (2018) 3973–3987. <https://doi.org/10.1007/s11661-018-4729-6>.
- [110] A. Epishin, Creep of the single-crystal nickel-base superalloy CMSX-4 at a super-solvus temperature, in: *Proceedings of the International Conference on Creep and Fracture of Engineering Materials and Structures*, Saint Petersburg, Russia, 2017.
- [111] W. Xia, X. Zhao, L. Yue, Z. Zhang, Microstructural evolution and creep mechanisms in Ni-based single crystal superalloys: a review, *Journal of Alloys and Compounds* 819 (2020) 152954. <https://doi.org/10.1016/j.jallcom.2019.152954>.
- [112] H. Mughrabi, The importance of sign and magnitude of γ/γ' lattice misfit in superalloys—with special reference to the new γ' -hardened cobalt-base superalloys, *Acta Materialia* 81 (2014) 21–29. <https://doi.org/10.1016/j.actamat.2014.08.005>.
- [113] T. Murakumo, T. Kobayashi, Y. Koizumi, H. Harada, Creep behaviour of Ni-base single-crystal superalloys with various γ' volume fraction, *Acta Materialia* 52 (2004) 3737–3744. <https://doi.org/10.1016/j.actamat.2004.04.028>.
- [114] L. Yang, A. Pramanik, S. Sen, S. Dixit, M. Muench, V. R.J., D. Schliephake, Y.M. Eggeler, A. Chauhan, M. Heilmaier, A. Kauffmann, Creep-induced microstructural evolution in an A2-B2 superalloy, *Advanced Engineering Materials* (2026). <https://doi.org/10.1002/adem.202503180>.

- [115] S. Karthikeyan, G.B. Viswanathan, M.J. Mills, Evaluation of the jogged-screw model of creep in equiaxed γ -TiAl: identification of the key substructural parameters, *Acta Materialia* 52 (2004) 2577–2589. <https://doi.org/10.1016/j.actamat.2004.02.006>.
- [116] G.B. Viswanathan, R.W. Hayes, M.J. Mills, A study based on jogged-screw dislocations for high temperature creep in Ti alloys, *Materials Science and Engineering: A* 319–321 (2001) 706–710. [https://doi.org/10.1016/S0921-5093\(01\)01048-6](https://doi.org/10.1016/S0921-5093(01)01048-6).
- [117] M. Wilkens, E. Hornbogen, Kontrasterscheinungen an versetzungspaaren in nickel-aluminium-legierungen mit kohärenter ausscheidung von geordneten bereichen, *physica status solidi (b)* 4 (1964) 557–568. <https://doi.org/10.1002/pssb.19640040309>.
- [118] S. Zhang, D.E. Mikkola, W.W. Milligan, APB vs. SISF dissociation of superdislocations in cubic L12 Al₃Ti alloys, *Scripta Metallurgica et Materialia* 27 (1992) 1073–1077. [https://doi.org/10.1016/0956-716X\(92\)90476-U](https://doi.org/10.1016/0956-716X(92)90476-U).
- [119] A. Pramanik, C. Konkati, S. Laube, L. Yang, S. Sen, A. Kauffmann, M. Heilmaier, A. Chauhan, Exploring room-temperature deformation mechanisms of a B2-strengthened refractory compositionally complex alloy, *Materials Science and Engineering: A* 931 (2025) 148180. <https://doi.org/10.1016/j.msea.2025.148180>.
- [120] A. Argon, *Strengthening mechanisms in crystal plasticity*, Oxford University Press, Oxford, 2007.
- [121] S.M. Zhu, S.C. Tjong, J.K.L. Lai, Creep behavior of a β' (NiAl) precipitation strengthened ferritic Fe–Cr–Ni–Al alloy, *Acta Materialia* 46 (1998) 2969–2976. [https://doi.org/10.1016/S1359-6454\(98\)00022-6](https://doi.org/10.1016/S1359-6454(98)00022-6).
- [122] N.Q. Vo, C.H. Liebscher, M.J.S. Rawlings, M. Asta, D.C. Dunand, Creep properties and microstructure of a precipitation-strengthened ferritic Fe–Al–Ni–Cr alloy, *Acta Materialia* 71 (2014) 89–99. <https://doi.org/10.1016/j.actamat.2014.02.020>.
- [123] Y. Zhao, Q. Fang, Y. Liu, P. Wen, Y. Liu, Creep behavior as dislocation climb over NiAl nanoprecipitates in ferritic alloy: the effects of interface stresses and temperature, *International Journal of Plasticity* 69 (2015) 89–101. <https://doi.org/10.1016/j.ijplas.2015.02.006>.
- [124] M.J.S. Rawlings, C.H. Liebscher, M. Asta, D.C. Dunand, Effect of titanium additions upon microstructure and properties of precipitation-strengthened Fe–Ni–Al–Cr ferritic alloys, *Acta Materialia* 128 (2017) 103–112. <https://doi.org/10.1016/j.actamat.2017.02.028>.
- [125] S.-I. Baik, S.-Y. Wang, P.K. Liaw, D.C. Dunand, Increasing the creep resistance of Fe–Ni–Al–Cr superalloys via Ti additions by optimizing the B2/L21 ratio in composite nanoprecipitates, *Acta Materialia* 157 (2018) 142–154. <https://doi.org/10.1016/j.actamat.2018.07.025>.
- [126] E. Arzt, M.F. Ashby, Threshold stresses in materials containing dispersed particles, *Scripta Metallurgica* 16 (1982) 1285–1290.
- [127] W. Blum, B. Reppich, Creep of particle-strengthened alloys, in: *Creep Behaviour of Crystalline Solids*, Pineridge Press, Swansea, 1985: pp. 83–135.

- [128] T.M. Pollock, R.D. Field, Chapter 63 dislocations and high-temperature plastic deformation of superalloy single crystals, in: *Dislocations in Solids*, Elsevier, 2002: pp. 547–618. [https://doi.org/10.1016/S1572-4859\(02\)80014-6](https://doi.org/10.1016/S1572-4859(02)80014-6).
- [129] W. Köster, Die temperaturabhängigkeit des elastizitätsmoduls reiner metalle, *International Journal of Materials Research* 39 (1948) 1–9. <https://doi.org/10.1515/ijmr-1948-390101>.
- [130] M. Friák, M. Černý, M. Šob, Temperature-dependent impact of antiphase boundaries on properties of Fe₃Al, *Intermetallics* 151 (2022) 107746. <https://doi.org/10.1016/j.intermet.2022.107746>.
- [131] S. Takeuchi, E. Furubayashi, T. Taoka, Orientation dependence of yield stress in 4.4% silicon iron single crystals, *Acta Metallurgica* 15 (1967) 1179–1191. [https://doi.org/10.1016/0001-6160\(67\)90392-6](https://doi.org/10.1016/0001-6160(67)90392-6).
- [132] R.J. Arsenault, A. Lawley, Work hardening and dislocation structure in Ta and Ta-base alloys, *Philosophical Magazine A* 15 (1967) 549–565. <https://doi.org/10.1080/14786436708220902>.
- [133] F. Maresca, W.A. Curtin, Theory of screw dislocation strengthening in random BCC alloys from dilute to “high-entropy” alloys, *Acta Materialia* 182 (2020) 144–162. <https://doi.org/10.1016/j.actamat.2019.10.007>.
- [134] S.V. Raj, I. Seideniskovitz, A.D. Freed, Modeling the role of dislocation substructure during class M and exponential creep, In: Krausz AS, Krausz K (Eds) *Unified Constitutive Laws of Plastic Deformation*. Academic Press, San Diego 96 (1995) 343–439.
- [135] Q. Yue, L. Liu, W. Yang, C. He, D. Sun, T. Huang, J. Zhang, H. Fu, Stress dependence of the creep behaviors and mechanisms of a third-generation Ni-based single crystal superalloy, *Journal of Materials Science & Technology* 35 (2019) 752–763. <https://doi.org/10.1016/j.jmst.2018.11.015>.
- [136] P. Wollgramm, H. Buck, K. Neuking, A.B. Parsa, S. Schuwalow, J. Rogal, R. Drautz, G. Eggeler, On the role of Re in the stress and temperature dependence of creep of Ni-base single crystal superalloys, *Materials Science and Engineering: A* 628 (2015) 382–395. <https://doi.org/10.1016/j.msea.2015.01.010>.
- [137] X. Wu, P. Wollgramm, C. Somsen, A. Dlouhy, A. Kostka, G. Eggeler, Double minimum creep of single crystal Ni-base superalloys, *Acta Materialia* 112 (2016) 242–260. <https://doi.org/10.1016/j.actamat.2016.04.012>.
- [138] R. Völkl, U. Glatzel, M. Feller-Kniepmeier, Measurement of the lattice misfit in the single crystal nickel based superalloys CMSX-4, SRR99 and SC16 by convergent beam electron diffraction, *Acta Materialia* 46 (1998) 4395–4404. [https://doi.org/10.1016/S1359-6454\(98\)00085-8](https://doi.org/10.1016/S1359-6454(98)00085-8).
- [139] U. Brückner, A. Epishin, T. Link, K. Dressel, The influence of the dendritic structure on the γ/γ' -lattice misfit in the single-crystal nickel-base superalloy CMSX-4, *Materials Science and Engineering: A* 247 (1998) 23–31. [https://doi.org/10.1016/S0921-5093\(97\)00856-3](https://doi.org/10.1016/S0921-5093(97)00856-3).

- [140] J. Coakley, D. Ma, M. Frost, D. Dye, D.N. Seidman, D.C. Dunand, H.J. Stone, Lattice strain evolution and load partitioning during creep of a Ni-based superalloy single crystal with rafted γ' microstructure, *Acta Materialia* 135 (2017) 77–87. <https://doi.org/10.1016/j.actamat.2017.06.021>.
- [141] F. Pyczak, A. Bauer, M. Göken, S. Neumeier, U. Lorenz, M. Oehring, N. Schell, A. Schreyer, A. Stark, F. Symanzik, Plastic deformation mechanisms in a crept L12 hardened Co-base superalloy, *Materials Science and Engineering: A* 571 (2013) 13–18. <https://doi.org/10.1016/j.msea.2013.02.007>.
- [142] Y. Li, F. Pyczak, J. Paul, M. Oehring, U. Lorenz, Z. Yao, Microstructure evolution in L12 hardened Co-base superalloys during creep, *Journal of Materials Research* 32 (2017) 4522–4530. <https://doi.org/10.1557/jmr.2017.362>.
- [143] T.M. Pollock, A.S. Argon, Directional coarsening in nickel-base single crystals with high volume fractions of coherent precipitates, *Acta Metallurgica et Materialia* 42 (1994) 1859–1874. [https://doi.org/10.1016/0956-7151\(94\)90011-6](https://doi.org/10.1016/0956-7151(94)90011-6).
- [144] N. Matan, D.C. Cox, C.M.F. Rae, R.C. Reed, On the kinetics of rafting in CMSX-4 superalloy single crystals, *Acta Materialia* 47 (1999) 2031–2045. [https://doi.org/10.1016/S1359-6454\(99\)00093-2](https://doi.org/10.1016/S1359-6454(99)00093-2).
- [145] C. Frey, B. Neuman, A. Botros, S.A. Kube, T.M. Pollock, Refractory multi-principal element alloys with solution and aged HfRu-B2 precipitates, *Scripta Materialia* 255 (2025) 116411. <https://doi.org/10.1016/j.scriptamat.2024.116411>.
- [146] K. Ma, T. Blackburn, J.P. Magnussen, M. Kerbstadt, P.A. Ferreira, T. Pinomaa, C. Hofer, D.G. Hopkinson, S.J. Day, P.A.J. Bagot, M.P. Moody, M.C. Galetz, A.J. Knowles, Chromium-based bcc-superalloys strengthened by iron supplements, *Acta Materialia* 257 (2023) 119183. <https://doi.org/10.1016/j.actamat.2023.119183>.
- [147] L.J. Carroll, Q. Feng, J.F. Mansfield, T.M. Pollock, Elemental partitioning in Ru-containing nickel-base single crystal superalloys, *Materials Science and Engineering: A* 457 (2007) 292–299. <https://doi.org/10.1016/j.msea.2006.12.036>.
- [148] X. Yao, Q. Ding, X. Wei, J. Wang, Z. Zhang, H. Bei, The effects of key elements Re and Ru on the phase morphologies and microstructure in Ni-based single crystal superalloys, *Journal of Alloys and Compounds* 926 (2022) 166835. <https://doi.org/10.1016/j.jallcom.2022.166835>.
- [149] I.M. Anderson, A.J. Duncan, J. Bentley, Site-distributions of Fe alloying additions to B2-ordered NiAl₁, *Intermetallics* 7 (1999) 1017–1024. [https://doi.org/10.1016/S0966-9795\(99\)00013-8](https://doi.org/10.1016/S0966-9795(99)00013-8).
- [150] C. Jiang, Site preference of transition-metal elements in B2 NiAl: a comprehensive study, *Acta Materialia* 55 (2007) 4799–4806. <https://doi.org/10.1016/j.actamat.2007.04.049>.
- [151] I. Steinbach, Phase-field models in materials science, *Modelling and Simulation in Materials Science and Engineering* 17 (2009) 073001. <https://doi.org/10.1088/0965-0393/17/7/073001>.

- [152] A.G. Khachaturyan, *Theory of Structural Transformations in Solids*, Courier Corporation, Mineola, NY, 2013.
- [153] T. Philippe, P.W. Voorhees, Ostwald ripening in multicomponent alloys, *Acta Materialia* 61 (2013) 4237–4244. <https://doi.org/10.1016/j.actamat.2013.03.049>.
- [154] T. Rieger, J.-M. Joubert, R. Poulain, X. Sauvage, E. Paccou, L. Perrière, I. Guillot, G. Dirras, G. Laplanche, M. Laurent-Brocq, J.-P. Couzinié, Influence of chemical composition on coarsening kinetics of coherent L12 precipitates in FCC complex concentrated alloys, *Journal of Alloys and Compounds* 967 (2023) 171711. <https://doi.org/10.1016/j.jallcom.2023.171711>.
- [155] M. Bonvalet Rolland, A. Borgenstam, Modeling precipitation kinetics in multicomponent alloys during deformation, *Frontiers in Materials* 9 (2022). <https://doi.org/10.3389/fmats.2022.958472>.
- [156] J.Y.C. Fang, W.H. Liu, T. Yang, Y. Wu, Z.B. Jiao, Multicomponent precipitation and strengthening in intermetallic-strengthened alloys, *Frontiers in Materials* 9 (2022). <https://doi.org/10.3389/fmats.2022.931098>.
- [157] A. Volek, F. Pyczak, R.F. Singer, H. Mughrabi, Partitioning of Re between γ and γ' phase in nickel-base superalloys, *Scripta Materialia* 52 (2005) 141–145. <https://doi.org/10.1016/j.scriptamat.2004.09.013>.
- [158] D. Shu, S. Tian, N. Tian, L. Liu, S. Liang, B. Zhang, Influence of Re/Ru on concentration distribution in the γ/γ' phases of nickel-based single crystal superalloys, *Materials & Design* 132 (2017) 198–207. <https://doi.org/10.1016/j.matdes.2017.06.069>.
- [159] K.E. Yoon, R.D. Noebe, D.N. Seidman, Effects of rhenium addition on the temporal evolution of the nanostructure and chemistry of a model Ni–Cr–Al superalloy. I: Experimental observations, *Acta Materialia* 55 (2007) 1145–1157. <https://doi.org/10.1016/j.actamat.2006.08.027>.
- [160] C.H. Su, P.W. Voorhees, The dynamics of precipitate evolution in elastically stressed solids—I. Inverse coarsening, *Acta Materialia* 44 (1996) 1987–1999. [https://doi.org/10.1016/1359-6454\(95\)00284-7](https://doi.org/10.1016/1359-6454(95)00284-7).
- [161] A.J. Ardell, The effect of volume fraction on particle coarsening: theoretical considerations, *Acta Metallurgica* 20 (1972) 61–71. [https://doi.org/10.1016/0001-6160\(72\)90114-9](https://doi.org/10.1016/0001-6160(72)90114-9).
- [162] A.J. Ardell, V. Ozolins, Trans-interface diffusion-controlled coarsening, *Nature Materials* 4 (2005) 309–316. <https://doi.org/10.1038/nmat1340>.
- [163] C. Wagner, Theory of precipitate change by redissolution, *Zeitschrift Für Elektrochemie* 65 (1961) 581–591.
- [164] J. Zhang, W. Lu, F. Lu, Q. Na, P. Yan, H. Su, G. Zhang, Elemental distribution and site occupancy in a Co–Ni–Al–Ti–Ta–W superalloy, *Materials Characterization* 215 (2024) 114199. <https://doi.org/10.1016/j.matchar.2024.114199>.

- [165] U. Hemmersmeier, M. Feller-Kniepmeier, Element distribution in the macro- and microstructure of nickel base superalloy CMSX-4, *Materials Science and Engineering: A* 248 (1998) 87–97. [https://doi.org/10.1016/S0921-5093\(98\)00516-4](https://doi.org/10.1016/S0921-5093(98)00516-4).
- [166] A.K. Jena, M.C. Chaturvedi, The role of alloying elements in the design of nickel-base superalloys, *Journal of Materials Science* 19 (1984) 3121–3139. <https://doi.org/10.1007/BF00549796>.
- [167] P.W. Voorhees, The theory of ostwald ripening, *Journal of Statistical Physics* 38 (1985) 231–252. <https://doi.org/10.1007/BF01017860>.
- [168] R. Shi, D.P. McAllister, N. Zhou, A.J. Detor, R. DiDomizio, M.J. Mills, Y. Wang, Growth behavior of γ'/γ'' coprecipitates in Ni-Base superalloys, *Acta Materialia* 164 (2019) 220–236. <https://doi.org/10.1016/j.actamat.2018.10.028>.
- [169] Y.S. Yoo, Morphological instability of spherical γ' precipitates in a nickel base superalloy, *Scripta Materialia* 53 (2005) 81–85. <https://doi.org/10.1016/j.scriptamat.2005.03.022>.

9 List of Publications

- (1) Liu Yang, Sandipan Sen, Daniel Schliephake, Stephan Laube, Aparajita Pramanik, Ankur Chauhan, Steffen Neumeier, Martin Heilmaier, Alexander Kauffmann. Creep behavior of a precipitation-strengthened A2-B2 refractory high-entropy alloy. *Acta Materialia*, 288 (2025) 120827. <https://doi.org/10.1016/j.actamat.2025.120827>.
- (2) Raja Jothi Vikram, Sandipan Sen, Liu Yang, Michael K. Eusterholz, Amin Radi, Daniel Schliephake, Jean-Philippe Couzinié, Alexander Kauffmann, Martin Heilmaier. Ultra-slow coarsening in precipitation-strengthened refractory high-entropy alloys. *Scripta Materialia*, 271 (2026) 117026. <https://doi.org/10.1016/j.scriptamat.2025.117026>.
- (3) Liu Yang, Aparajita Pramanik, Sandipan Sen, Shubhashis Dixit, Marcel Muench, R. J. Vikram, Daniel Schliephake, Yolita M. Eggeler, Ankur Chauhan, Martin Heilmaier, Alexander Kauffmann. Creep-induced microstructural evolution in an A2-B2 superalloy, *Advanced Engineering Materials*, in press.

10 List of Figures

Figure 1. Comparison of two precipitation-strengthened microstructures. (a) SEM-BSE micrograph of the homogenized 27.3Ta-27.3Mo-27.3Ti-8Cr-10Al, showing a disordered A2 matrix with ordered B2 precipitates. (b) TEM micrograph of the homogenized $\gamma+\gamma'$ CMSX-4 Ni-based superalloy, showing a disordered γ matrix with ordered γ' precipitates [26].

Figure 2. Schematic illustration of the crystal structures of (a) the disordered A2 phase and (b) the ordered B2 phase. In the A2 structure, all constituent atoms occupy the equivalent 2a Wyckoff positions of the BCC lattice. In the B2 structure, chemical ordering produces two inequivalent primitive cubic sublattices: the 1a sublattice at (0,0,0) and the 1b sublattice at $(1/2, 1/2, 1/2)$. The 1b sublattice is displaced by $1/2[111]$ relative to the 1a sublattice, as indicated by the arrow in (b).

Figure 3. Schematic representation of chemical decomposition within a miscibility gap, corresponding to the decomposition $A2 \rightarrow A2' + A2''$. The upper panel shows a simplified temperature-composition diagram with a critical temperature T_c . The lower panel shows the corresponding Gibbs free energy curves at different temperatures. The development of a double-well shape below T_c provides the thermodynamic driving force for phase separation.

Figure 4. Schematic illustration of phase separation pathways in a supersaturated A2 solid solution within a miscibility gap. (a) Phase diagrams showing binodal (solid line) and spinodal (dashed line) boundaries. Decomposition inside the spinodal region (Case I) occurs by spinodal decomposition from an unstable A2 solid solution, whereas decomposition between the binodal and spinodal boundaries (Case II) proceeds by classical nucleation and growth from a metastable A2 solid solution. (b) Corresponding microstructural evolution. Spinodal decomposition (Case I) leads to an interconnected $A2' + A2''$ structure with diffuse interfaces, while nucleation and growth (Case II) results in discrete $A2''$ precipitates in an $A2'$ matrix with sharp interfaces.

Figure 5. Schematic representation of two possible ordering pathways in nucleation-controlled A2+B2 systems. Case I: chemical phase separation first produces two disordered regions, $A2 \rightarrow A2' + A2''$, followed by ordering of the precipitate-forming region, $A2'' \rightarrow B2$, resulting in an $A2'+B2$ microstructure. Case II: compositional decomposition and B2 ordering occur concurrently, leading directly to $A2 \rightarrow A2' + B2$.

Figure 6. Schematic representation of creep deformation at elevated homologous temperatures. (a) Conventional creep strain-time curve showing primary, secondary, and tertiary creep regimes. (b) Corresponding strain-rate evolution plotted as $\log \dot{\epsilon}$ versus ϵ . A steady-state regime is characterized by an extended strain-rate plateau, whereas the $\dot{\epsilon}_{\min}$ may

also occur as a local minimum between primary deceleration and tertiary acceleration without an extended steady state.

Figure 7. Schematic log-log representation of $\dot{\epsilon}_{\min}$ versus σ illustrating the σ_{eff} concept. The threshold stress σ_p produces a horizontal stress shift between the matrix and the precipitation-strengthened alloy. The slope of the matrix curve defines n_m , while the slope obtained for the strengthened alloy using the applied stress corresponds to the apparent stress exponent n_{app} .

Figure 8. Schematic comparison of the LB and LBR methods for determining the threshold stress σ_p . (a) LB method assuming constant n_m and σ_p , yielding a linear representation in transformed coordinates. (b) LBR method using the matrix creep response as a physical reference, where σ_p is obtained as the horizontal stress difference at equal creep rates.

Figure 9. Schematic illustration of stress-assisted rafting for coherent precipitates with positive lattice misfit, $\delta > 0$, under compressive loading. (a) Symmetric misfit-induced internal stress field around coherent precipitates in the absence of an external load. (b) Superposition of misfit strain and external compressive stress, resulting in an anisotropic elastic strain energy distribution. (c) Formation of N-type rafts, where precipitates elongate perpendicular to the loading direction to reduce the total elastic energy.

Figure 10. Determination of the precipitate solvus temperature, T_{solv} , of TMT-8Cr-10Al. (a–d) SEM-BSE micrographs after annealing at 1040, 1050, 1060, and 1070 °C for 95 h followed by water quenching, showing the progressive dissolution of the precipitates with increasing temperature. (e) Quantitative evolution of the volume fraction of precipitates, determined from image-based area fraction measurements of the corresponding microstructures in (a–d). (f) Schematic illustration of the difference between T_{ord} and T_{solv} in the TMT-8Cr-10Al alloy.

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Figure 29. TEM results of the specimen after creep at 1030 °C under 100 MPa. (a) TEM-SADP along the [001] zone axis (ZA) showing fundamental A2/B2 spots and weak B2 superlattice peaks. (b) TEM-DF micrograph obtained using a (100) B2 superlattice peak reveals bright-contrast B2 precipitates. (c) Low-magnification, two-beam TEM-BF micrograph providing an overview of the crept microstructure with few marked curved dislocations. (d) A relatively high-magnification, two-beam TEM-BF micrograph showing a curved dislocation segment (black arrow) interacting with a B2 precipitate, consistent with local climb-assisted bypass. The blue and red dashed lines indicate the projected screw and edge orientations, respectively; the solid white line indicates the trace of the $(\bar{1}01)$ plane. Adapted from Ref. [114].

Figure 30. TEM results of the specimen after creep at 1030 °C under 125 MPa. (a) TEM-SADP along the [001] ZA showing fundamental A2/B2 spots and weak B2 superlattice peaks. (b) TEM-DF micrograph obtained using a (100) B2 superlattice peak. (c) Low-magnification, two-beam TEM-BF micrograph taken along the [113] ZA, showing the deformed microstructure. (d) Two-beam TEM-BF micrograph acquired using $g = [1\bar{1}0]$, showing dislocation climb (pink arrows) and superpartial-mediated cutting of a B2 precipitate (ellipse). (e) Same area as in (d), imaged under reversed diffraction vector, $-g = [\bar{1}10]$, revealing the jogged climb segment (black arrow) and contrast reversal of superpartials within B2. (f) Paired dislocations (yellow arrow) near precipitates, indicative of earlier or forthcoming cutting events. (g) Long dislocation segments within A2 channels (red arrow), with corresponding trace analysis marked by solid lines. (h) and (i) Two-beam TEM-BF. Adapted from Ref. [114].

Figure 31. TEM results of the specimen after creep at 1030 °C under 150 MPa. (a) TEM-SADP along a [001] ZA showcasing fundamental A2/B2 spots and weak B2 superlattice peaks. (b) TEM-DF micrograph taken using a [100] B2 superlattice reflection. (c) Low-magnification, two-beam TEM-BF micrograph showing long dislocation segments within A2 channels, many of which exhibit abrupt changes in line direction (yellow arrows). (d, e) Representative dislocation configuration imaged along the [001] and [113] ZA, respectively. The trace analysis in (d), together with the schematic representation in (f), indicates that adjacent

dislocation segments are compatible with different $\{110\}$ and $\{112\}$ planes for the assigned $1/2\langle 111 \rangle$ -type Burgers vector. (g) TEM-BF micrograph acquired close to the $[113]$ zone axis, showing local dislocation-precipitate interactions, including configurations compatible with climb-assisted bypass. Adapted from Ref. [114].

Figure 32. A2/B2 interface in the specimen after creep at 1030 °C under 125 MPa to a strain of 3.1%. (a) Low-magnification STEM-HAADF micrograph showing an elongated, rafted B2 precipitate in the A2 matrix. (b₁, c₁ and d₁) Atomic-resolution STEM-HAADF micrographs of different sections of the precipitate-matrix interface, as indicated in (a). (b₂, c₂ and d₂) Corresponding IFFT images obtained using the (110) spots from the FFT patterns. One representative FFT pattern corresponding to (b₁) is shown in (b₃). Adapted from Ref. [114].

Figure 33. SEM-BSE micrographs showing the grain structure of the Re- and Ru-modified alloys in the homogenized and furnace-cooled condition. (a) 1.5Re, (b) 2.5Re, (c) 5Re, (d) 1.5Ru, (e) 2.5Ru and (f) 5Ru. All alloys exhibit coarse equiaxed grains with sizes on the order of several hundred micrometers. The dark spots visible in the micrographs correspond to pores formed during sample preparation.

Figure 34. SEM-BSE micrographs of homogenized and furnace-cooled Re- and Ru-modified alloys. (a–c) Re-modified alloys with increasing Re content, and (d–f) Ru-modified alloys with increasing Ru content. All samples display a two-phase microstructure comprising a disordered A2 matrix and ordered B2 precipitates at the nanoscale. Green and purple labels denote the Re- and Ru-modified alloy series, respectively, and the same color coding is used throughout this chapter.

Figure 35. XRD patterns of powder samples of the base TMT-8Cr-10Al alloy and the Re- and Ru-modified alloys in the homogenized and furnace-cooled condition. (a) Re-modified alloys and (b) Ru-modified alloys. All peaks correspond to a BCC lattice and are denoted as A2/B2 reflections, since the fundamental reflections of the disordered A2 matrix and the ordered B2 phase overlap.

Figure 36. Dependence of the ordering temperature, T_{ord} , and the solvus temperature, T_{solv} , on alloying element content for (a) Re-modified and (b) Ru-modified alloys. The T_{ord} values were determined from DSC measurements by extrapolating the peak temperatures obtained at different heating rates to a nominal heating rate of 0 K/min. The T_{solv} values were determined independently by systematic isothermal annealing experiments followed by water quenching. Error bars indicate an estimated uncertainty of ± 5 K. Lines are guides to the eye.

Figure 37. Schematic section along the approximate tie line illustrating the miscibility gap and ordering reaction. The T_{solv} is defined as the temperature above which the secondary phase completely dissolves $A2' + A2'' \rightarrow A2$, while the T_{ord} is defined as the temperature at which

long-range B2 order within the secondary phase disappears, corresponding to the B2 $\rightarrow$ A2'' order-disorder transition. The data for the base alloy were obtained from Refs. [35,145]. Temperature uncertainties are conservatively estimated; the compositions are obtained at 1000 °C and rounded to 1 at.%. Schematic compositions at temperatures higher than 1000 °C are estimated based on the phase fractions obtained after quenching.

Figure 38. SEM-BSE micrographs showing the microstructural evolution of the Re-modified alloys after isothermal annealing at 1000 °C for (a–c) 10 h, (d–f) 100 h, and (g–i) 1000 h.

Figure 39. SEM-BSE micrographs showing the microstructural evolution of the Ru-modified alloys after isothermal annealing at 1000 °C for (a–c) 10 h, (d–f) 100 h, and (g–i) 1000 h. The red arrows indicate isolated A2 regions that are spatially separated from the interconnected B2 network.

Figure 40. TEM characterization of the 5Re- and 5Ru-modified alloys after isothermal heat treatment at 1000 °C for 1000 h. (a, d) TEM-BF images acquired with the selected-area aperture inserted, indicating the regions used for diffraction analysis. (b, e) TEM-SAED patterns obtained along the $\langle 001 \rangle$ zone axis. The bright fundamental A2 spots, such as $\{110\}$ and $\{200\}$, are labeled in white. The weak $\{100\}$ superlattice spot associated with B2 ordering is highlighted by a yellow circle and selected for TEM-DF imaging. (c, f) TEM-DF images formed using the $\{100\}$ superlattice spot, revealing the spatial distribution of B2-type ordered domains in both modified alloys.

Figure 41. (a, c) FIB-prepared cuboids of 5Re and 5Ru alloys aged at 1000 °C for 1000 h, showing three orthogonal surfaces (top, lateral, front) prepared for multi-directional SEM-BSE imaging. (b, d) Quasi-3D representation of the precipitate morphologies in 5Re and 5Ru alloys generated from SEM-BSE images acquired on the corresponding three surfaces.

Figure 42. APT results of the (a–c) 2.5Re and (d–f) 2.5Ru alloys aged at 1000 °C for 100 h. (a, d) 3D reconstructions showing Ti isoconcentration surfaces at 35 at.% delineating the B2 phase. (b, c) and (e, f) 1D cylindrical concentration profiles across representative A2/B2 interfaces for the 2.5Re and 2.5Ru alloys, respectively, illustrating elemental partitioning between the matrix and precipitates.

Figure 43. Evolution of characteristic length parameters during isothermal aging at 1000 °C for TMT-8Cr-10Al base alloy and Re-, Ru-, and Zr-modified alloys. The mean minor axis $\bar{b}_p$, mean major axis $\bar{a}_p$, and mean equivalent radius $\bar{r}_{eq}$ are used for (a–c) Re-modified alloys, (d) Ru-modified alloys, (e, f) TMT-8Cr-10Al, and (g, h) 1Zr alloy. The y-axes are cube-scaled but labelled with the corresponding characteristic length in nm. Linear fits were applied to $\bar{b}_p^3$, $\bar{a}_p^3$ and $\bar{r}_{eq}^3$ as a function of aging time to determine the coarsening-rate constant κ , as indicated in each panel. For the Ru-modified alloys, only the $\bar{b}_p$ -based coarsening rate is shown because

partial interconnection of the plate-like precipitates prevents reproducible measurement of the full particle length.

Figure 44. Coarsening kinetics of B2 precipitates in the investigated A2+B2 RHEAs. (a) Coarsening rate constant κ for the base alloy and Zr-, Re-, and Ru-modified alloys, obtained from the linear fits of $\bar{b}_p^3$ as a function of aging time. The inset compares κ values derived from different characteristic lengths ($\bar{a}_p$, $\bar{b}_p$ and $\bar{r}_{eq}$). (b) Dependence of the $\bar{b}_p$ -based κ on the normalized temperature T/T_{solv} for the investigated RHEA system. (c–d) Comparison of κ for the present RHEA alloys with literature data for Ni-based and Cr-based superalloys as a function of T/T_{solv} and $T/T_{solidus}$, respectively. Dashed lines are guides to the eye.

Figure A1. Trace analysis and schematic representation of the dislocation configuration shown in Figure 29d in the main text for two possible slip planes. (a) $(\bar{1}01)^{1/2}[\bar{1}\bar{1}1]$ and (b) $(101)^{1/2}[\bar{1}\bar{1}1]$. In both cases, the projected line direction indicates mixed edge-screw character, which is compatible with possible climb-assisted bypass of B2 precipitates. The projected screw and edge orientations are indicated by blue and red dashed lines, respectively. (c) Corresponding TEM-SAED pattern along the $[001]$ zone axis and stereographic projection showing the relevant plane traces. Because the two possible traces overlap in the $[001]$ projection, a unique slip-plane assignment is not possible from this projection alone. Adapted from Ref. [114].

Figure A2. TEM-BF two-beam micrographs of the specimen after creep at 1030 °C under 125 MPa, showing pronounced dislocation-precipitate interactions. Jogged or cusp-like dislocation configurations are marked by yellow arrows. Several paired dislocation configurations are observed near B2 precipitates, providing additional examples of dislocation arrangements associated with the interactions shown in Figure 30 in the main text. The colored trace overlays indicate representative projected traces of relevant $\{110\}$ and $\{112\}$ planes for the $[001]$ zone-axis projection. Adapted from Ref. [114].

Figure A3. Color-filtered overlay generated using the cropped sections of Figures 30d–e in the main text for better visualization of the paired dislocation cutting through B2. The images were acquired under opposite diffraction vectors, $g = [\bar{1}\bar{1}0]$ and $-g = [\bar{1}\bar{1}0]$. The color filtering is used only for visualization. Adapted from Ref. [114].

Figure A4. Trace analysis and diffraction-contrast analysis of dislocation configurations in the specimen crept at 1030 °C under 150 MPa. (a) Two-beam TEM-BF micrograph acquired close to the $[001]$ zone axis, with colored trace overlays showing relevant $\{110\}$ and $\{112\}$ plane traces. (b) Two-beam TEM-BF images acquired close to the $[001]$ zone axis under different diffraction conditions, showing visibility and invisibility of selected dislocation segments. (c) Two-beam TEM-BF images acquired close to the $[113]$ zone axis under different diffraction conditions. The visibility changes are used together with the $g \cdot b = 0$ criterion to constrain the

Burgers vector to $\frac{1}{2}[\bar{1}11]$. Combined with the trace analysis, these observations support the interpretation that the dislocation segments shown in Figures 31d–f are geometrically compatible with different $\{110\}$ and $\{112\}$ planes. The color filtering is used only for visualization. Adapted from Ref. [114].

Figure A5. Two-beam TEM-BF micrographs taken along the $[113]$ zone axis from the specimen crept at 1030 °C under 150 MPa. The same dislocation features are marked by arrows of the same color in images recorded under different diffraction vectors. The change in apparent contrast with diffraction condition confirms that the observed line contrast is diffraction-controlled and supports the use of diffraction-contrast analysis together with trace analysis for the local dislocation-precipitate interactions shown in Figure 31g. Adapted from Ref. [114].

Figure A6. Schematic representing an active slip system in BCC illustrating evidence of cross-slip. Adapted from Ref. [114].

Figure A7. Differential scanning calorimetry (DSC) curves of Re- and Ru-modified alloys measured at heating rates of 10, 20, and 40 K/min. The λ -shaped endothermic peaks indicate the ordering-disordering transition of the B2 phase. The peak temperature shifts with heating rate due to kinetic effects and the overlap of ordering and phase separation processes. The extracted peak temperatures were used to determine the ordering temperature, as shown in Figure A8.

Figure A8. Peak temperatures extracted from DSC measurements as a function of heating rate for (a) Re- and (b) Ru-modified alloys. The peak temperature increases with heating rate due to kinetic effects. Linear fits were applied to the data, and extrapolation to zero heating rate was used to determine the T_{ord} of the B2 phase.

Figure A9. Determination of the T_{solv} for Re-, Ru-, and Zr-modified alloys based on microstructural observations. Representative microstructures are shown for each alloy at temperatures below and above the T_{solv} . The disappearance of precipitates indicates that T_{solv} has been exceeded, resulting in a single-phase A2 microstructure. The estimated T_{solv} values are: 1.5Re and 1.5Ru (1075 ± 5 °C), 2.5Re and 2.5Ru (1085 ± 5 °C), 5Re (1135 ± 5 °C), 5Ru (1115 ± 5 °C), and 1Zr (1065 ± 5 °C).

Figure A10. Segmentation sensitivity analysis for the determination of B2 volume fraction in 5Re after isothermal heat treatment at 1000 °C for 1000 h. (a) Original SEM-BSE micrograph. (b–c) Segmented image and binary mask obtained using the upper-bound threshold. (d–e) Segmented image and binary mask obtained using the intermediate threshold. (f–g) Segmented image and binary mask obtained using the low-bound threshold. The corresponding B2 volume fractions are 29%, 25%, and 21%, respectively. The small variation demonstrates the limited influence of threshold selection on the quantitative phase fraction.

Figure A11. Segmentation sensitivity analysis for the determination of B2 volume fraction in 5Ru after isothermal heat treatment at 1000 °C for 1000 h. (a) Original SEM-BSE micrograph. (b–c) Segmented image and binary mask obtained using the upper-bound threshold. (d–e) Segmented image and binary mask obtained using the intermediate threshold. (f–g) Segmented image and binary mask obtained using the low-bound threshold. The corresponding B2 volume fractions are 29%, 25%, and 21%, respectively. The small variation demonstrates the limited influence of threshold selection on the quantitative phase fraction.

Figure A12. TEM-based determination of B2 precipitate area fraction in the 5Re alloy aged at 1000 °C for 1000 h. (a) Original TEM micrograph. (b, d, f) Manually annotated precipitate regions (highlighted in red) to compensate for limited contrast between precipitates and matrix. (c, e, g) Corresponding binary masks used for quantitative analysis. Three segmentation levels were applied, yielding B2 area fractions of 30%, 25%, and 20%, respectively.

Figure A13. TEM-based determination of B2 precipitate area fraction in the 5Ru alloy aged at 1000 °C for 1000 h. (a) Original TEM micrograph. (b, d, f) Manually annotated precipitate regions (highlighted in red) to compensate for limited contrast between precipitates and matrix. (c, e, g) Corresponding binary masks used for quantitative analysis. Three segmentation levels were applied, yielding B2 area fractions of 30%, 25%, and 20%, respectively.

Figure A14. (a) Creep strain rate as a function of strain for the 5Re-modified alloy tested at 1080 °C and 125 MPa. A minimum creep rate of $2.66 \times 10^{-8} \text{ s}^{-1}$ is observed after the primary stage. (b) Corresponding microstructure after creep exposure for 115 h, showing predominantly spherical B2 precipitates. The absence of directional coarsening (rafting) indicates a reduced lattice misfit between the A2 matrix and B2 precipitates.

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Table 1. Nominal target compositions, experimentally measured compositions, and interstitial impurity contents of the alloys investigated in this work. The concentrations of O and N were determined by CGHE.

Table 2. Desired and experimental composition of the monolithic A2 alloy (at.%).

Table 3. $\dot{\epsilon}_{\min}$ of TMT-8Cr-10Al measured at 125 MPa for different temperatures. Values represent averages of repeated tests where available; otherwise, representative values from individual tests are reported.

Table 4. Quantitative characterization of B2 precipitates in Re- and Ru-modified alloys after isothermal aging at 1000 °C for different durations. The table summarizes the mean major axis ($\bar{a}_p$), mean minor axis ($\bar{b}_p$), mean equivalent particle radius ($\bar{r}_{eq}$), B2 area fraction, circularity, and number density of precipitates obtained from SEM-BSE image analysis. For the Ru-modified alloys, $\bar{a}_p$ and $\bar{r}_{eq}$ are not reported because partial interconnection of the plate-like precipitates prevents reproducible assignment of the full precipitate length to individual particles.

Table 5. Average compositions of the A2 matrix and B2 precipitates measured by atom probe tomography after aging at 1000 °C for 100 h. The partitioning coefficients k_i and composition differences ΔC_i between the two phases are also listed. Data for the base alloy are taken from Ref. [27].

Table A1. Parameters used for the semi-quantitative estimate of the critical shear stress for B2 precipitate cutting at RT and 1030 °C using the Hütner-Reppich model [114].

Table A2. Microstructural parameters and estimated Orowan stresses for TMT-8Cr-10Al after creep at 1030 °C [114].

Table A3. Chemical composition of the B2 site occupancies predicted by CALPHAD at room temperature (RT) and measured by APT for specimens aged at 1000 °C for 100 h.

Table A4. Estimated lattice misfit values for the investigated alloys, calculated from the A2 and B2 phase compositions obtained by APT after aging at 1000 °C for 100 h. The calculation is based on the atomic size data reported in the Frey database [34] and should be regarded as an approximate reference, since the database provides atomic radii at 0 K.

12 Appendix

A1. Trace Analysis and Supplementary TEM Observations for Chapter 5

The slip-plane assignment of dislocation segments observed in Chapter 5 was performed by trace analysis using the known crystallographic zone axis of the TEM micrographs and the cube-on-cube orientation relationship between the A2 matrix and B2 precipitates established in Section 4.4. The purpose of this analysis was not to uniquely determine every individual slip system, but to identify the slip-plane families that are geometrically compatible with the observed projected dislocation traces.

For a given candidate slip plane (hkl), the trace direction observed in a TEM projection corresponds to the intersection between the slip plane and the image plane normal to the electron-beam direction. If the electron-beam direction is denoted by the zone-axis vector $\mathbf{z}$, and the normal vector of the candidate slip plane is denoted by $\mathbf{n}_{hkl}$, the projected trace direction $\mathbf{t}_{hkl}$ is given by:

$$\mathbf{t}_{hkl} = \mathbf{n}_{hkl} \times \mathbf{z} \quad (\text{A.1})$$

The calculated trace directions for possible BCC slip planes were projected into the corresponding TEM image plane and compared with the experimentally observed dislocation-line traces. Candidate slip planes were restricted to BCC-type slip systems containing $1/2\langle 111 \rangle$ Burgers vectors, primarily $\{110\}\langle 111 \rangle$ and $\{112\}\langle 111 \rangle$, as introduced in Section 2.1.2. A candidate plane was considered geometrically possible only when the Burgers vector lies within the plane, i.e.:

$$hu + kv + lw = 0 \quad (\text{A.2})$$

where $[uvw]$ is the Burgers-vector direction and (hkl) is the candidate slip plane.

For selected dislocations, the Burgers vector was further constrained using diffraction-contrast analysis under different diffraction vectors g . The condition:

$$g \cdot b = 0 \quad (\text{A.3})$$

was used as the primary invisibility criterion. Because residual contrast may remain for mixed dislocations and because the exact dislocation line direction is affected by projection, the Burgers-vector assignment is treated as consistent with, rather than as a unique proof of, the proposed slip system.

In the [001] projection, several {110} and {112} traces overlap or lie close to each other in projection. Therefore, some slip-plane assignments cannot be uniquely distinguished from a single projection. For this reason, the main text uses the wording “geometrically compatible with slip on {110} or {112} planes” rather than assigning a unique individual slip plane. Where two zone axes were available, such as the [001] and [113] projections used for Figures 31d–g, the combined projection geometry provides stronger evidence that adjacent dislocation segments lie on different crystallographic planes. This supports the interpretation of repeated glide-plane changes or cross-slip-like activity of screw or mixed dislocations.

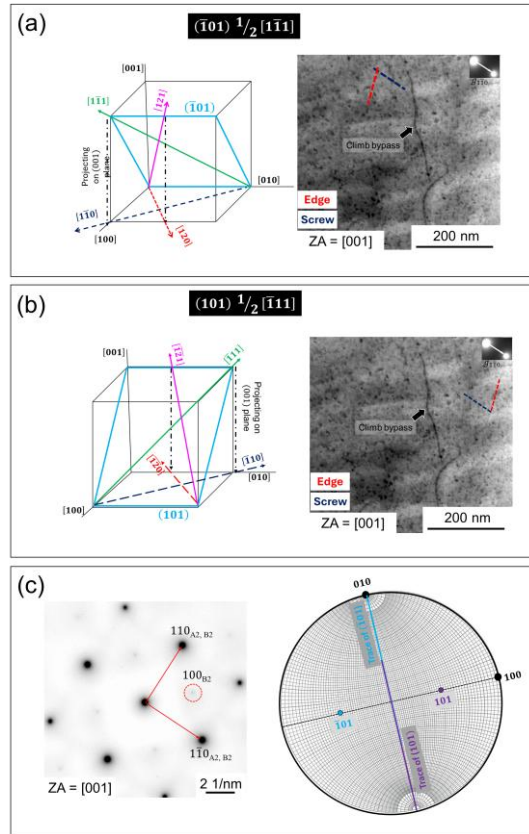


Figure A1. Trace analysis and schematic representation of the dislocation configuration shown in Figure 29d in the main text for two possible slip planes. (a) $(\bar{1}01) \frac{1}{2} [1\bar{1}1]$ and (b) $(101) \frac{1}{2} [\bar{1}11]$. In both cases, the projected line direction indicates mixed edge-screw character, which is compatible with possible climb-assisted bypass of B2 precipitates. The projected screw and edge orientations are indicated by blue and red dashed lines, respectively. (c) Corresponding TEM-SAED pattern along the [001] zone axis and stereographic projection showing the relevant plane traces. Because the two possible traces overlap in the [001] projection, a unique slip-plane assignment is not possible from this projection alone. Adapted from Ref. [114].

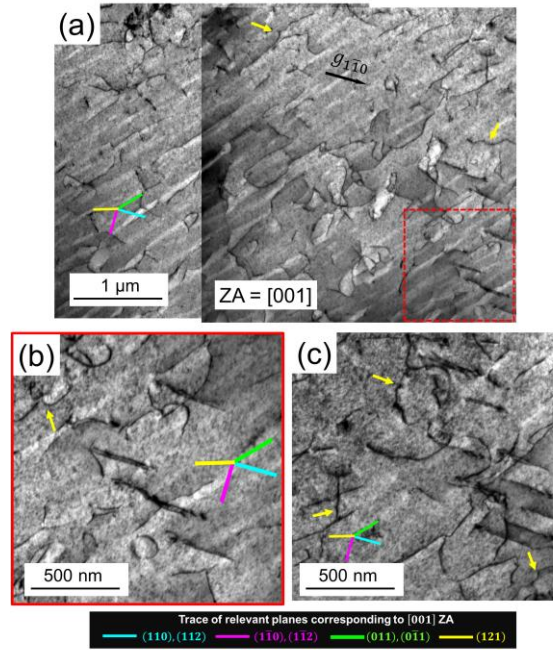


Figure A2. TEM-BF two-beam micrographs of the specimen after creep at 1030 °C under 125 MPa, showing pronounced dislocation-precipitate interactions. Jogged or cusp-like dislocation configurations are marked by yellow arrows. Several paired dislocation configurations are observed near B2 precipitates, providing additional examples of dislocation arrangements associated with the interactions shown in Figure 30 in the main text. The colored trace overlays indicate representative projected traces of relevant $\{110\}$ and $\{112\}$ planes for the $[001]$ zone-axis projection. Adapted from Ref. [114].

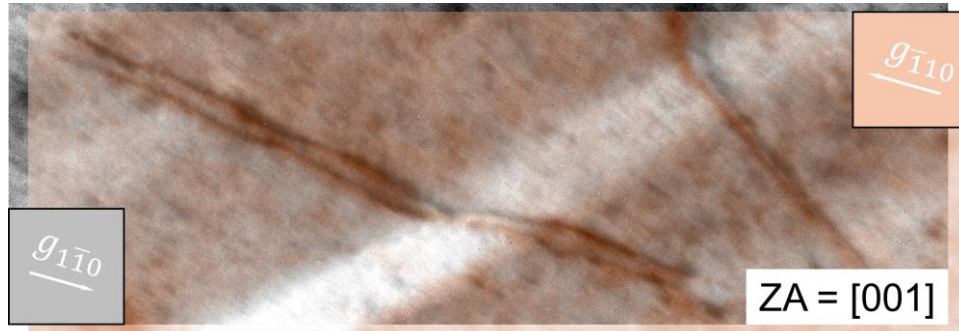


Figure A3. Color-filtered overlay generated using the cropped sections of Figures 30d-e in the main text for better visualization of the paired dislocation cutting through B2. The images were acquired under opposite diffraction vectors, $g = [1\bar{1}0]$ and $-g = [\bar{1}10]$. The color filtering is used only for visualization. Adapted from Ref. [114].

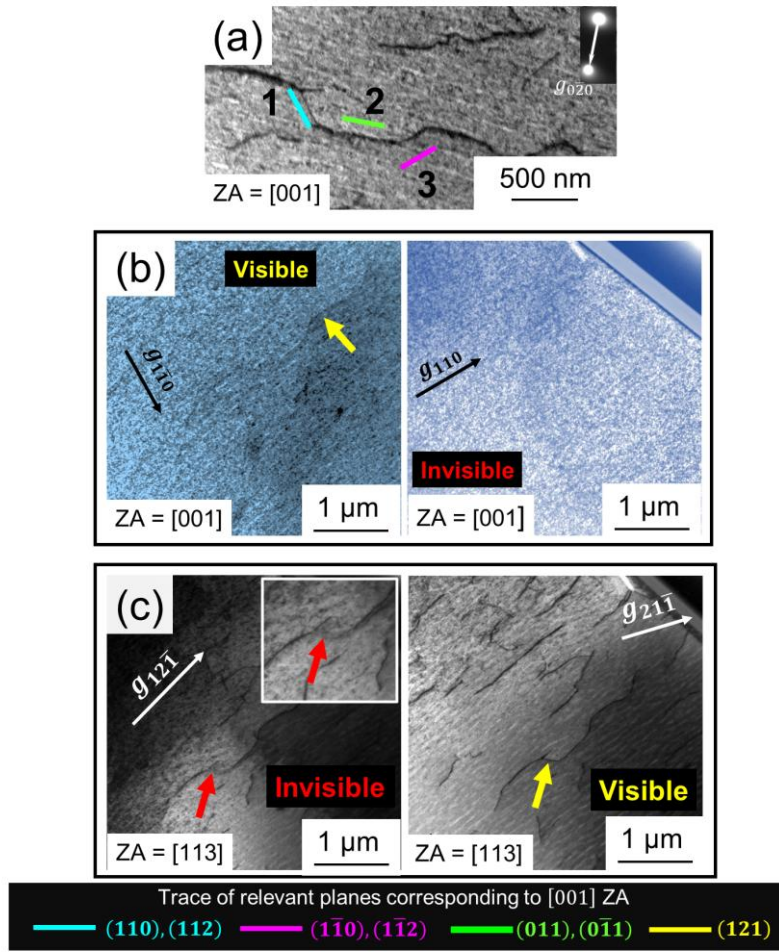
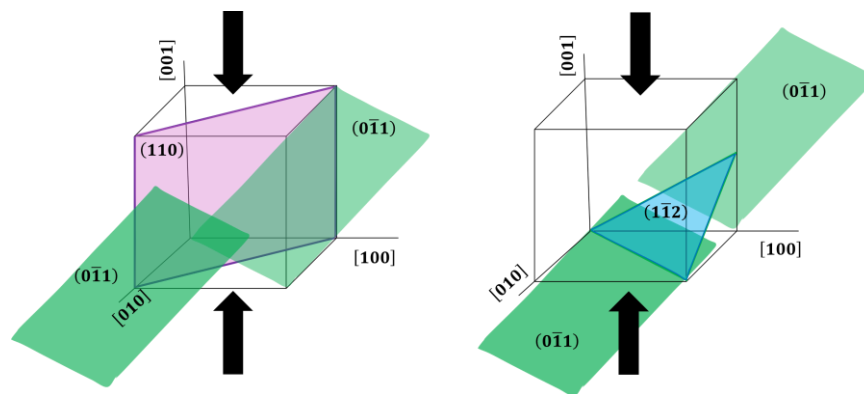
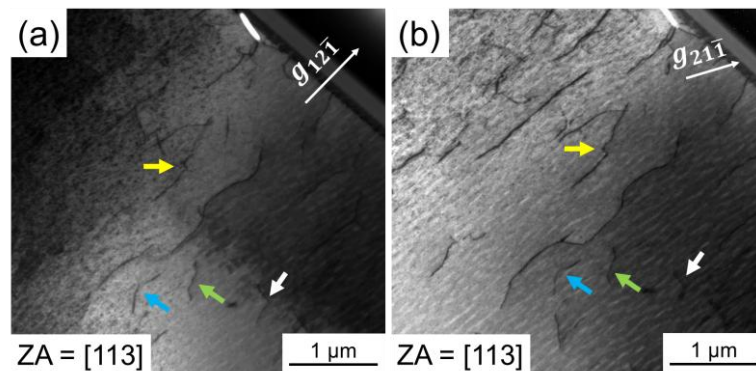


Figure A4. Trace analysis and diffraction-contrast analysis of dislocation configurations in the specimen crept at 1030 °C under 150 MPa. (a) Two-beam TEM-BF micrograph acquired close to the $[001]$ zone axis, with colored trace overlays showing relevant $\{110\}$ and $\{112\}$ plane traces. (b) Two-beam TEM-BF images acquired close to the $[001]$ zone axis under different diffraction conditions, showing visibility and invisibility of selected dislocation segments. (c) Two-beam TEM-BF images acquired close to the $[113]$ zone axis under different diffraction conditions. The visibility changes are used together with the $g \cdot b = 0$ criterion to constrain the Burgers vector to $\frac{1}{2}[\bar{1}11]$. Combined with the trace analysis, these observations support the interpretation that the dislocation segments shown in Figures 31d–f are geometrically compatible with different $\{110\}$ and $\{112\}$ planes. The color filtering is used only for visualization. Adapted from Ref. [114].



A2. Parameters Used for the Semi-Quantitative Stress Estimates in Section 5.6

The interpretation of dislocation-precipitate interactions in Section 5.6 is supported by two semi-quantitative estimates. First, the critical shear stress for B2 precipitate cutting was evaluated using the Hühner-Reppich model introduced in Section 2.2.4. The parameters used for this estimate are summarized in Table A1. Second, the Orowan stress was estimated using the measured channel widths of the rafted B2 precipitate microstructure. The corresponding microstructural parameters and calculated stresses are summarized in Table A2. These estimates are intended to support the mechanistic interpretation of the TEM observations and

should be regarded as semi-quantitative because several high-temperature material parameters of the present alloy are not directly available.

Table A1. Parameters used for the semi-quantitative estimate of the critical shear stress for B2 precipitate cutting at RT and 1030 °C using the Hütner-Reppich model [114].

Parameter	Symbol	Room Temperature	1030 °C	Reference
Adjustable Parameter	w	0.22	0.22	[81,119]
Empirical Constant	C_{SL}	1	1	[81,119]
Shear Modulus	G	80 GPa	60 GPa (75% retention)	[129]
Line Tension	T_L	3.14 nN	2.35 nN	[82,119]
APB Energy	γ_{APB}	150 mJ/m ²	~90 mJ/m ² (60% retention)	[81,119]
Volume Fraction	f	26%	26%	[81,119]
Precipitate Radius	r	65 nm	65 nm	[81,119]
Burgers Vector	b	0.28 nm	0.28 nm	[81,119]
Taylor Factor	M	3.06	3.06	[81,119]

Table A2. Microstructural parameters and estimated Orowan stresses for TMT-8Cr-10Al after creep at 1030 °C [114].

Applied Stress	f (%)	r (nm)	L (nm)	Λ (nm)	τ_{Orowan} (MPa)	σ_{Orowan} (MPa)
100 MPa	21%	65	260	130	~52	~159
125 MPa	26%	65	237	100	~67	~206
150 MPa	18%	40	175	95	~71	~217

The channel width λ was estimated as $L - 2r$, where L is the center-to-center spacing and r is the mean precipitate radius.

A3. Supplementary Results for Chapter 6

DSC measurements were used to determine the ordering temperature, T_{ord} , of the B2 phase in the Re- and Ru-modified alloys. The thermal response is shown in Figure A7 as the temperature derivative of the enthalpy, dH/dT . For all investigated alloys, a characteristic λ -shaped endothermic peak is observed. This peak is associated with the order-disorder transition of the B2 phase. The peak temperature shifts systematically with heating rate,

which is attributed to kinetic effects during heating and to the coupling between ordering and compositional redistribution.

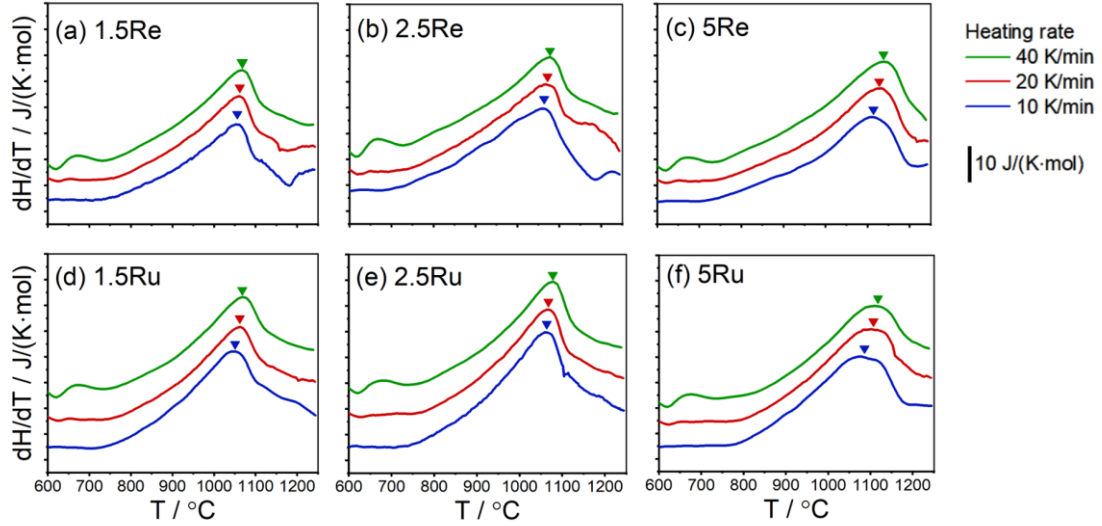


Figure A7. Differential scanning calorimetry (DSC) curves of Re- and Ru-modified alloys measured at heating rates of 10, 20, and 40 K/min. The λ -shaped endothermic peaks indicate the ordering-disordering transition of the B2 phase. The peak temperature shifts with heating rate due to kinetic effects and the overlap of ordering and phase separation processes. The extracted peak temperatures were used to determine the ordering temperature, as shown in Figure A8.

The peak temperatures extracted from the DSC curves were analyzed as a function of heating rate, as shown in Figure A8. A nearly linear dependence is observed for all investigated alloys. To obtain the thermodynamic transition temperature, linear fits were applied and extrapolated to zero heating rate. The intercept of each fitted line is defined as the T_{ord} of the B2 phase. This procedure minimizes kinetic effects and enables a consistent comparison between different alloy compositions. It should be emphasized that the T_{ord} determined from DSC is fundamentally different from the solvus temperature, which is independently obtained from microstructural observations after heat treatment, as shown in Figure A9.

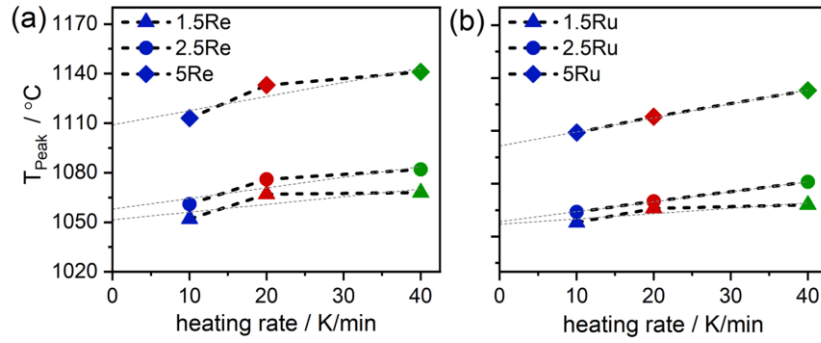


Figure A8. Peak temperatures extracted from DSC measurements as a function of heating rate for (a) Re- and (b) Ru-modified alloys. The peak temperature increases with heating rate due to kinetic effects. Linear fits were applied to the data, and extrapolation to zero heating rate was used to determine the T_{ord} of the B2 phase.

The T_{solv} was estimated by identifying the temperature at which precipitates are no longer observed after heat treatment at selected temperatures for 95 h. Microstructures below the T_{solv} exhibit a clear two-phase microstructure, while those above the T_{solv} show a single-phase A2 matrix. The transition temperature was determined within an uncertainty of ± 5 °C.

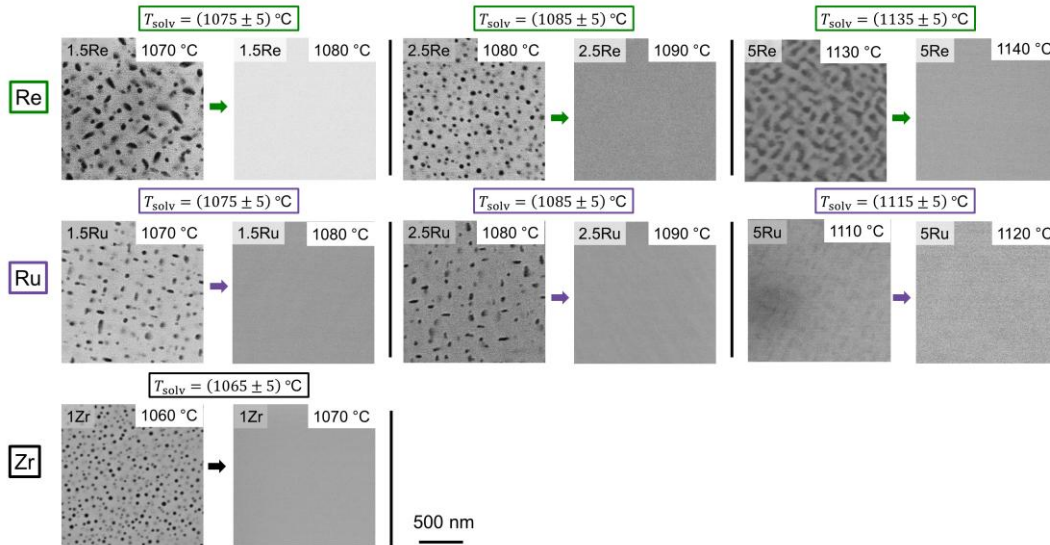


Figure A9. Determination of the T_{solv} for Re-, Ru-, and Zr-modified alloys based on microstructural observations. Representative microstructures are shown for each alloy at temperatures below and above the T_{solv} . The disappearance of precipitates indicates that T_{solv} has been exceeded, resulting in a single-phase A2 microstructure. The estimated T_{solv} values are: 1.5Re and 1.5Ru (1075 ± 5 °C), 2.5Re and 2.5Ru (1085 ± 5 °C), 5Re (1135 ± 5 °C), 5Ru (1115 ± 5 °C), and 1Zr (1065 ± 5 °C).

The B2 phase fraction was determined from SEM-BSE micrographs using ImageJ. In the investigated alloys, the B2 precipitates are nanoscale and densely distributed. Owing to the finite interaction volume of backscattered electrons and the projection nature of SEM imaging, signals from precipitates located at slightly different depths may partially overlap. Consequently, the precipitate-matrix interfaces appear as gradual gray-level transitions rather than perfectly sharp boundaries, rendering the segmentation procedure moderately sensitive to threshold selection.

To quantify this segmentation sensitivity, three different threshold levels within a physically reasonable range were applied to the same micrograph for each alloy, as illustrated in Figure A10 (5Re) and Figure A11 (5Ru). The selected threshold range was defined such that clearly identifiable precipitates were captured while avoiding artificial merging of neighboring features or misclassification of matrix regions. The procedure involved: (i) acquisition of SEM-BSE micrographs under identical imaging conditions; (ii) threshold-based segmentation using ImageJ; (iii) conversion to 8-bit binary masks; and (iv) determination of the precipitate area fraction using the “Area Fraction” measurement function after selecting the entire image area. For randomly distributed precipitates, the area fraction equals the volume fraction. Therefore, the measured area fraction was taken as the B2 volume fraction. This procedure was applied consistently to all investigated compositions. For the representative 5Re and 5Ru samples (Figure A10 and A11), the three threshold settings yielded B2 volume fractions of 29%, 25%, and 21%, resulting in a mean value of 25% with an uncertainty of $\pm 4\%$ (standard deviation).

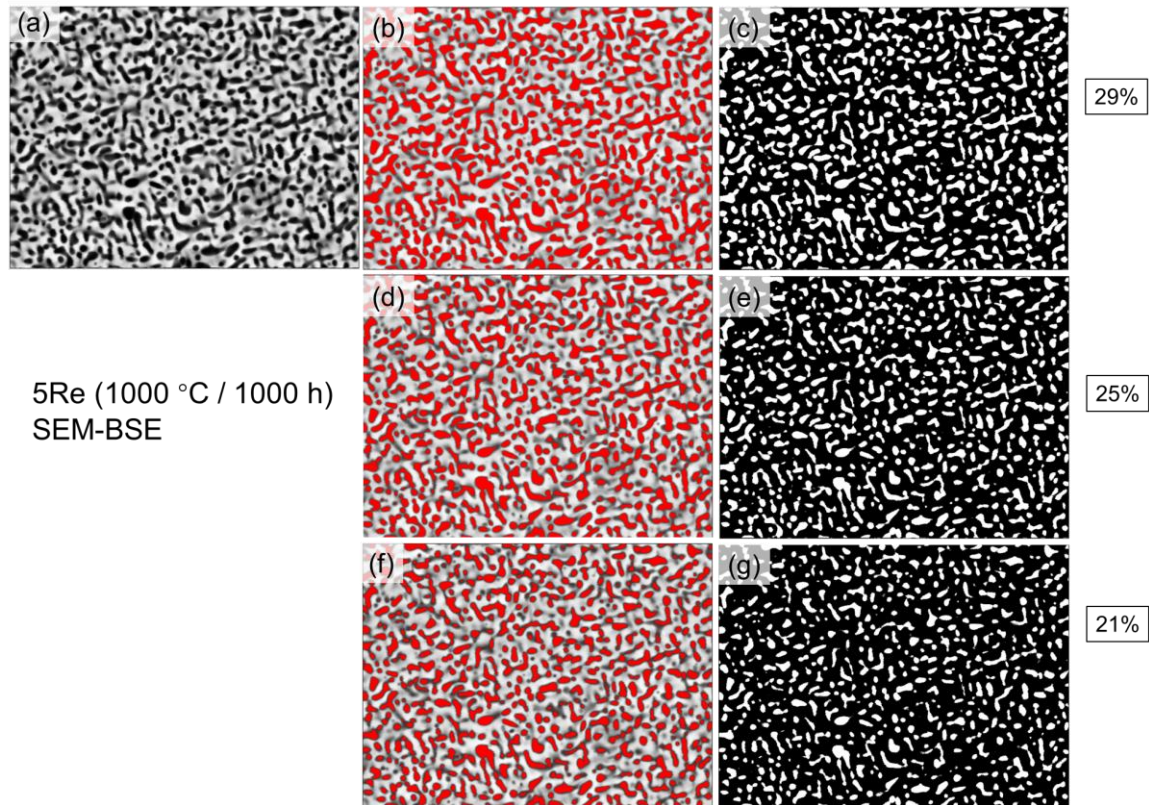


Figure A10. Segmentation sensitivity analysis for the determination of B2 volume fraction in 5Re after isothermal heat treatment at 1000 °C for 1000 h. (a) Original SEM-BSE micrograph. (b–c) Segmented image and binary mask obtained using the upper-bound threshold. (d–e) Segmented image and binary mask obtained using the intermediate threshold. (f–g) Segmented image and binary mask obtained using the low-bound threshold. The corresponding B2 volume fractions are 29%, 25%, and 21%, respectively. The small variation demonstrates the limited influence of threshold selection on the quantitative phase fraction.

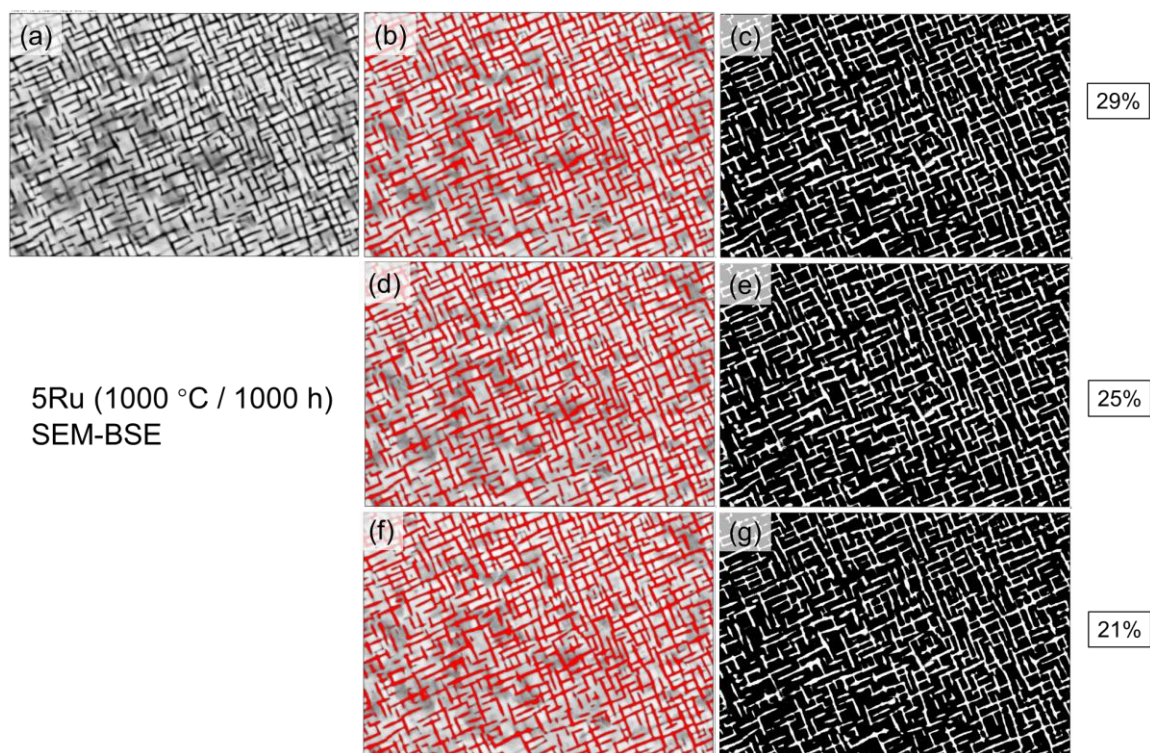


Figure A11. Segmentation sensitivity analysis for the determination of B2 volume fraction in 5Ru after isothermal heat treatment at 1000 °C for 1000 h. (a) Original SEM-BSE micrograph. (b–c) Segmented image and binary mask obtained using the upper-bound threshold. (d–e) Segmented image and binary mask obtained using the intermediate threshold. (f–g) Segmented image and binary mask obtained using the low-bound threshold. The corresponding B2 volume fractions are 29%, 25%, and 21%, respectively. The small variation demonstrates the limited influence of threshold selection on the quantitative phase fraction.

To further validate the reliability of the measured B2 phase fraction, TEM analysis was additionally performed. Due to the inherently limited contrast in TEM images, the distinction between B2 precipitates and the matrix is less pronounced compared to SEM-BSE imaging. As a result, conventional threshold-based segmentation becomes unreliable and highly sensitive to local contrast variations. To address this issue, the precipitate regions were manually annotated based on careful visual identification of morphology and contrast features, as shown in Figure A12 (5Re) and Figure A13 (5Ru). The annotated regions were subsequently converted into binary masks, from which the area fraction was calculated using the same ImageJ workflow as described for SEM-BSE analysis.

To evaluate the uncertainty associated with manual identification, three independent segmentation levels were generated for each TEM micrograph by slightly varying the inclusion criteria of ambiguous regions. The resulting B2 area fractions for the representative

5Re sample are 30%, 25%, and 20%, respectively, which are in good agreement with the values obtained from SEM-BSE analysis. This consistency confirms that, despite the limited contrast in TEM images, the combined approach of manual annotation and statistical evaluation provides a reliable estimation of the B2 phase fraction. Similar results were obtained for the 5Ru alloy (Figure A13), further supporting the robustness of the analysis.

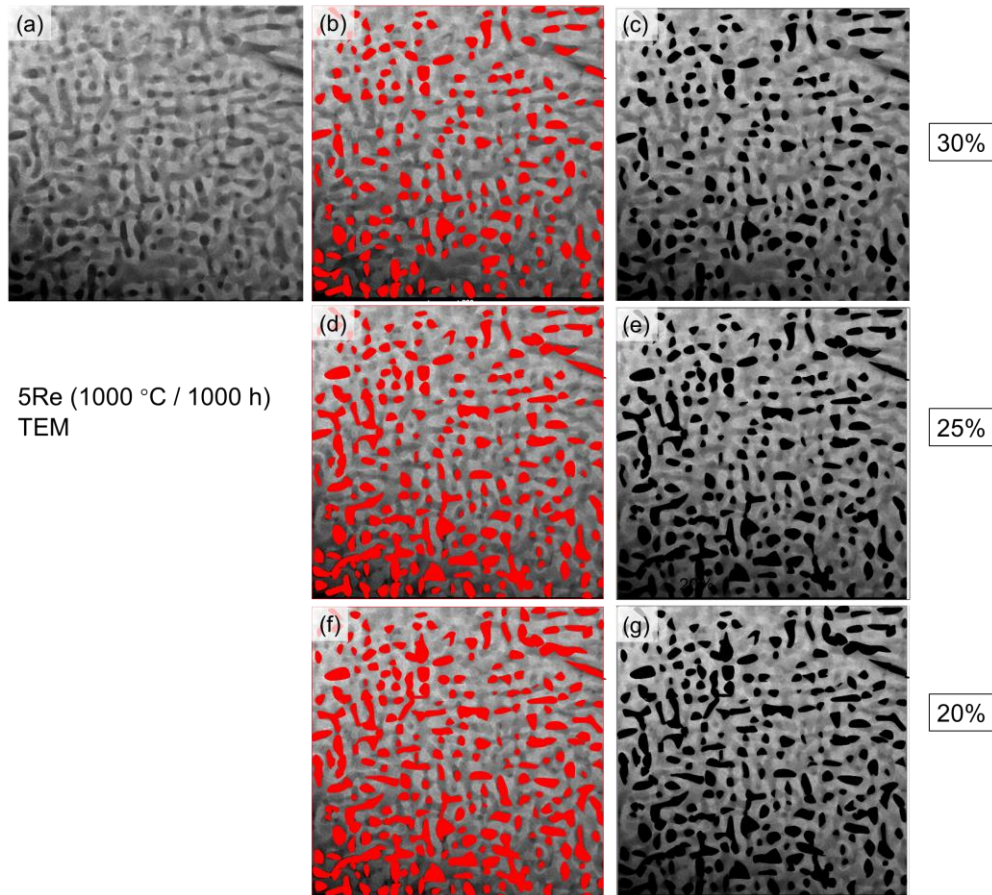


Figure A12. TEM-based determination of B2 precipitate area fraction in the 5Re alloy aged at 1000 °C for 1000 h. (a) Original TEM micrograph. (b, d, f) Manually annotated precipitate regions (highlighted in red) to compensate for limited contrast between precipitates and matrix. (c, e, g) Corresponding binary masks used for quantitative analysis. Three segmentation levels were applied, yielding B2 area fractions of 30%, 25%, and 20%, respectively.

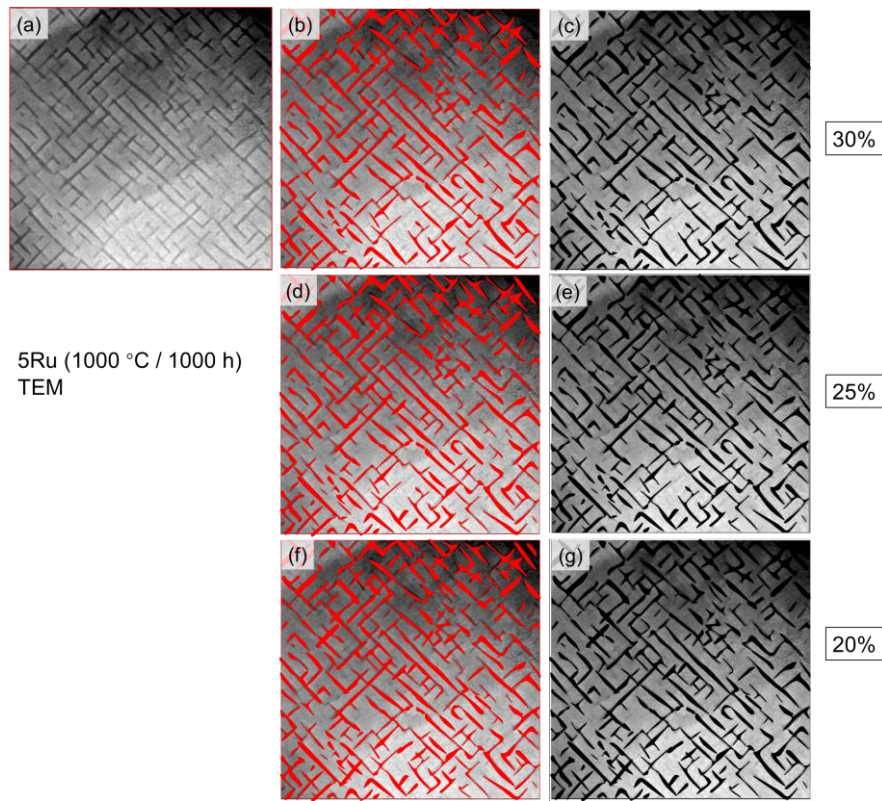


Figure A13. TEM-based determination of B2 precipitate area fraction in the 5Ru alloy aged at 1000 °C for 1000 h. (a) Original TEM micrograph. (b, d, f) Manually annotated precipitate regions (highlighted in red) to compensate for limited contrast between precipitates and matrix. (c, e, g) Corresponding binary masks used for quantitative analysis. Three segmentation levels were applied, yielding B2 area fractions of 30%, 25%, and 20%, respectively.

The creep behavior and corresponding microstructure of the 5Re-modified alloy provide indirect evidence for a reduced lattice misfit between the A2 matrix and B2 precipitates. As shown in Figure A14a, the alloy exhibits a minimum creep rate after the primary stage. The microstructure after creep exposure in Figure A14b reveals predominantly spherical precipitates without indication of directional coarsening. In precipitation-strengthened alloys, rafting is typically driven by elastic interactions arising from lattice misfit under applied stress. The absence of pronounced rafting therefore suggests a reduced elastic driving force for directional coarsening. This observation is consistent with the decreasing lattice-misfit trend with Re addition discussed in Section 6.7.

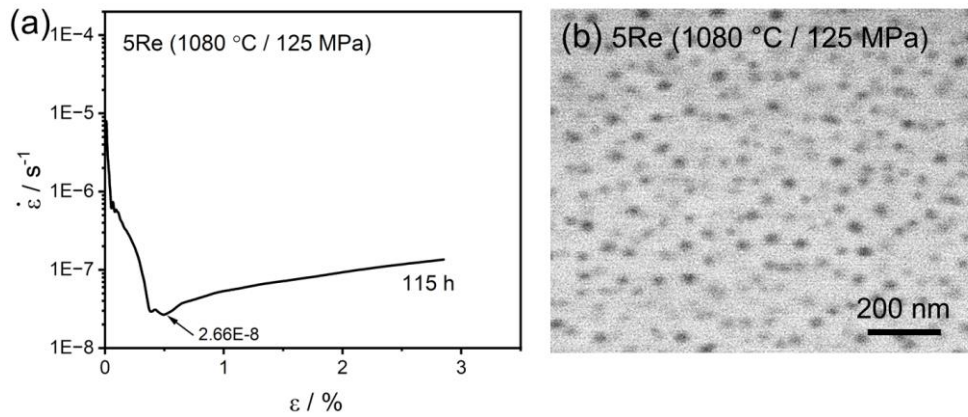


Figure A14. (a) Creep strain rate as a function of strain for the 5Re-modified alloy tested at 1080 °C and 125 MPa. A minimum creep rate of $2.66 \times 10^{-8} \text{ s}^{-1}$ is observed after the primary stage. (b) Corresponding microstructure after creep exposure for 115 h, showing predominantly spherical B2 precipitates. The absence of directional coarsening (rafting) indicates a reduced lattice misfit between the A2 matrix and B2 precipitates.

Table A3. Chemical composition of the B2 site occupancies predicted by CALPHAD at room temperature (RT) and measured by APT for specimens aged at 1000 °C for 100 h.

	at.%	Ta	Mo	Ti	Cr	Al	Ru	Zr	f
alloy	TMT-8Cr-10Al	27.3	27.3	27.3	8.0	10.0			-
A2	1000°C/100h	30.4 ± 0.4	31.6 ± 0.5	25.1 ± 0.4	7.1 ± 0.2	5.7 ± 0.3			73.0
B2		14.9 ± 0.3	21.8 ± 0.3	38.4 ± 0.4	6.6 ± 0.2	18.2 ± 0.3			27.0
A2	Pandata	43.6	42.5	13.9	0.0	0.0	-	-	62.7
B2		0.0	1.8	49.9	21.5	26.8			37.3
B2, site 1	Pandata	0.0	0.2	99.8	0.0	0.0			-
B2, site 2		0.0	3.4	0.0	42.9	53.7			-
alloy	1.5Ru	26.8	26.8	26.8	8.0	10.0	1.5		-
A2	1000°C/100h								-

B2								
A2		42.8	42.3	14.9	0.0	0.0	0.0	62.6
B2		0.0	0.9	46.9	21.4	26.8	4.0	37.4
B2, site 1	Pandat	0.0	0	92.0	0.0	0.0	8.0	-
B2, site 2		0.0	1.8	1.9	42.8	53.5	0.0	-
alloy	2.5Ru	26.5	26.5	26.5	8.0	10.0	2.5	-
A2	1000°C/100h	31.8 ± 2.3	32.4 ± 2.3	21.7 ± 2.0	5.9 ± 1.2	4.2 ± 1.0	2.9 ± 0.8	66.3
B2		16.5 ± 1.3	21.5 ± 1.4	36.1 ± 1.6	6.5 ± 0.8	14.9 ± 1.2	3.4 ± 0.6	33.7
A2		42.9	42.5	14.6	0.0	0.0	0.0	- 61.8
B2		0.0	0.6	45.8	20.9	26.2	6.5	38.2
B2, site 1	Pandat	0.0	0.0	86.9	0.0	0.0	13.1	-
B2, site 2		0.0	1.2	4.7	41.7	52.3	0	-
alloy	5Ru	25.7	25.7	25.7	8.0	10.0	5	-
A2	1000°C/100h	32.7 ± 2.5	33.0 ± 2.5	20.6 ± 2.2	6.1 ± 1.3	3.5 ± 1.0	3.8 ± 1.0	63.5
B2		18.3 ± 1.7	22.6 ± 1.9	34.8 ± 2.1	6.1 ± 1.1	12.3 ± 1.5	5.6 ± 1.0	36.5
A2		39.5	39.2	15.4	5.2	0.7	0.0	- 64.3
B2		0.8	1.3	44.1	13.1	26.7	14.0	35.7
B2, site 1	Pandat	0.1	0.0	71.8	0.1	0.0	28.0	-
B2, site 2		1.5	2.6	16.4	26.2	53.4	0	-
alloy	TMT-8Cr- 10Al-12r	27.0	27.0	27.0	8.0	10.0	1.0	-

A2	1000°C/100h	33.4	32.1	24.0	5.6	4.7	0.3	68.4
		± 0.5	± 0.5	± 0.4	± 0.2	± 0.7	± 0.1	
B2	1000°C/100h	13.2	21.7	38.8	4.9	19.9	1.4	31.6
		± 0.6	± 0.7	± 0.8	± 0.4	± 0.7	± 0.2	
A2	1000°C/100h	43.8	43.4	12.8	0.0	0.0	0.0	61.7
B2		0.0	0.6	49.8	20.9	26.1	2.6	
B2, site 1	Pandat	0.0	1.1	0.0	41.8	52.2	5.0	-
B2, site 2	Pandat	0.0	0.2	99.6	0.0	0.0	0.2	

To obtain an approximate assessment of the lattice misfit between the A2 matrix and B2 precipitates, the phase compositions measured by APT for samples aged at 1000 °C for 100 h were used for calculation based on the atomic size data from the Frey database [34]. The resulting values are summarized in Table A4. It should be noted that these values are only intended as a qualitative reference, since the Frey database provides atomic radii at 0 K and therefore does not fully represent the atomic dimensions at the experimental temperatures relevant to the present work. Consequently, the calculated lattice misfit values should not be interpreted as exact quantitative values, but rather as an approximate indication of the relative trend among the investigated alloys. Despite this limitation, the calculation suggests that Re additions tend to reduce the magnitude of the negative lattice misfit compared to the base alloy, whereas Ru additions lead to a slight increase in the magnitude of the lattice misfit. This trend is consistent with the experimentally observed tendency towards more spherical precipitate morphologies in the Re-modified alloys and more plate-like morphologies in the Ru-modified alloys.

Table A4. Estimated lattice misfit values for the investigated alloys, calculated from the A2 and B2 phase compositions obtained by APT after aging at 1000 °C for 100 h. The calculation is based on the atomic size data reported in the Frey database [34] and should be regarded as an approximate reference, since the database provides atomic radii at 0 K.

Alloy	δ (%)	δ^2 (%)
TMT-8Cr-10Al	$(+ 0.6 \pm 0.4)$	0.3600
(TEM, published data [98])		
TMT-8Cr-10Al	-0.16	0.0271
2.5Re	-0.16	0.0264

5Re	-0.18	0.0335
2.5Ru	-0.22	0.0501
5Ru	-0.25	0.0619
1Zr	-0.13	0.0157

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