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High temperature oxidation protection in body-centered cubic superalloys

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ABSTRACT

Materials from the class of metallic-intermetallic, body-centered cubic (BCC) superalloys with microstructures composed of A2, B2 and $L2_1$ phases are candidates for high temperature application due to high solidus/solvus temperatures and the expectedly good creep resistance. However, experience with their base systems indicates substantial problems that need to be tackled when high temperature oxidation resistance is concerned. This is especially the case for refractory element-containing alloys where catastrophic oxidation at rather low temperatures can occur. This seems a particular challenge for alloy development as no predictive simulation capabilities and metallurgical mitigation strategies are available to systematically suppress critical oxidation behavior in complex alloys – while maintaining other relevant properties. The present article briefly reviews the possibilities to obtain known protective scales in relevant BCC superalloy systems. It concludes on the challenges that need to be addressed to empower novel BCC superalloys to provide protection against high temperature oxidation.

Introduction

The development of Ni-based superalloys is pivotal to advancements in high-temperature applications [1–3], such as those in the aerospace and power plant industries. These superalloys are outstanding because of their excellent resistance to mechanical and chemical degradation. The mechanical resistance of complex/modern Ni-based superalloys is largely due to precipitation strengthening by coherent Ni_3Al-based $L1_2$ (Cu_3Au prototype) precipitates in a Ni-rich A1 matrix (Cu prototype).

Furthermore, the inherent characteristics of Ni make it an ideal base metal [2]. These characteristics include low diffusivity at the melting point, structural stability and absence of phase transformations in the A1 matrix, ductility and toughness. This is accompanied by lower density and cost among other candidate base elements like for example Co [4–6] and Pt [7–9]. However, when it comes to chemical degradation, particularly oxidation at high temperatures, Ni seems not the optimal choice, as B1 NiO (NaCl prototype) does not provide an effective barrier to oxidation [10]. Instead, the alloying elements that contribute to the formation of the two-phase A1-$L1_2$ microstructures, Al and Cr, also promote the development of protective $D5_1$ Al_2O_3 or Cr_2O_3 (both corundum prototype) scales, respectively, which exhibit parabolic growth laws combined with low growth rates ($n = 2$ in Eq. (1)).

The engineering application in combustion environments at temperatures $> 1500^\circ$C requires additional measures as the solidus and liquidus temperatures of the alloys are exceeded. These measures, like cooling and thermal barrier coatings, restrict the gains in efficiency improvement [11].

As an alternative, materials that possess higher melting/solidus temperatures have evolved. Many of them are based on or contain large fractions of refractory elements or intermetallic phases and exhibit an A2 (W prototype, like Mo [12,13], Nb [13,14], Ta [13,15], W [16,17], Cr [18–21], etc.) or B2 crystal structure (CsCl prototype, like NiAl [20, 22–24], HfRu [25,26], ZrRu [27,28], TaMoCrTiAl [29,30], non-equimolar TiZrHfVNbAl [31] etc.), respectively. Despite the obvious benefit of higher melting/solidus temperatures, resistance against creep deformation is a concern due to their non-close-packed crystal structure with enhanced diffusivity at elevated temperature. Thus, the field of refractory body-centered cubic (BCC) superalloys [32] has emerged trying to transfer the microstructure of A1-$L1_2$ superalloys to BCC-based alloys.

With respect to the resistance against high temperature oxidation and corrosion, this novel concept poses substantial challenges different to experience in Ni-based superalloys. In the present article, we cover these challenges related to promoting protective oxide scale formation, specific problems induced by the relevant base alloying systems and the conclusions to the community in developing novel BCC superalloys with

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the required high temperature oxidation resistance. Currently existing gaps in detailed experimental investigations, the required consideration of evaporating species as well as the urgent need for the development of predictive simulation capabilities are stressed. To restrict the presentation to the outlined alloy development scheme, we focus on the oxidation resistance of materials with microstructures primarily consisting of A2-B2, but also A2-$L2_1$ ($L2_1$ is $AlCu_2Mn$ prototype) and A2-B2-$L2_1$ that form via a solid-state transformation as well as their constituting phases, A2, B2 and $L2_1$. However, the presentation is not limited to refractory-element based systems and covers (i) B2 and $L1_2$ strengthened (Fe,Ni)-based alloys [33–43], (ii) B2 NiAl strengthened (Cr,Fe)-based alloys [20,21], (iii) B2 TiFe strengthened, Ti- or (W,Ti)-based alloys [17,44,45], (iv) B2 Ta-rich matrix alloys with A2 (Ti,Zr,Al,Mo,Nb) [46] or refractory-element based high entropy A2 + B2/B2 + A2 superalloys [32,47]. The more complex subject of general high temperature corrosion is addressed in the outlook.

Protective scales

Oxidation resistance requires several key conditions to be met [48]. There must be a high driving force to preferentially form the desired oxide over compounds with the other constituting elements of an alloy. This can be achieved by surpassing a critical concentration of the passivating element. Additionally, fast initial formation of the covering oxide film followed by the slow, diffusion-controlled growth kinetics during the steady-state oxidation are essential to enable the formation of protective scales. Simultaneously, there needs to be a sufficiently fast diffusion of the passivating element in the substrate to allow a constant supply of the subscale region. The oxidation kinetics might be tracked via monitoring the area-specific mass change of specimens $\frac{\Delta m}{A}$ as a function of exposure time t:

$$\left(\frac{\Delta m}{A}\right)^n = k_{m,n}\, t \tag{1}$$

Ideally, kinetics should then be parabolic, $n = 2$, or with even larger growth exponents, $n > 2$, to facilitate long-term oxidation resistance. While the diffusion through the scale in this case continuously slows down, positive linear oxidation kinetics with $n = 1$ indicate porous scale formation with persistent O ingress to the substrate/scale interface. Negative or discontinuous mass changes indicate unrestricted contact of substrate surface to the atmosphere by the formation of evaporating species or spallation. The volume and thermal expansion of the oxides should approximately match the corresponding properties of the base alloy. This compatibility allows for the stress-lean growth of compact and dense oxide, even under thermal cycling conditions which is application relevant in most cases. Thus, in general thermodynamic, kinetic and mechanical considerations are relevant for the development of passivating alloys. This seems specifically challenging when the base alloy system does not exhibit intrinsic protectiveness. Strictly formulated, there are currently no consistent predictive capabilities/methodologies to convert complex alloying systems reliably into oxidation resistant.

Three types of oxides are commonly accepted to form protective scales [13], namely Al_2O_3 [1,2,48], Cr_2O_3 [1,2,48] and SiO_2 [12], and are, thus, typically targeted in alloy development as they usually fulfil the aforementioned requirements. The experimental ranges of oxidation rate constants $k_{m,n=2}$ according to Eq. (1) for these oxides are depicted in Fig. 1. These rate constants are low compared to other oxides and, thus, allow for protection. However, in case of complications like evaporation, it is also required to track scale thickness as a function of time to differentiate uptake by scale growth and loss by other mechanisms:

$$d^n = k_{d,n}\, t \tag{2}$$

The $k_{d,n=2}$ at 1000°C (indicated as dotted line in Fig. 1) for SiO_2, Al_2O_3 and Cr_2O_3 are in the order of 10^{-18}, 10^{-17}–10^{-16} and 10^{-16}–10^{-13} m^2/s, respectively. When engineering application is considered, experimental oxidation and growth rates should ideally be in the ranges indicated.

Apart from the commonly accepted protective oxides, other complex solid solution oxides were recently identified to exhibit some of the required characteristics named above, like C4 $(Cr,Ta,Ti)O_2$ (rutile prototype) on refractory high entropy alloys from the Ta-Mo-Nb-Cr-Ti-Al-Si system [15,30,49–51]. Furthermore, investigations confirm the ability of Fe-, Ni- and Co-based alloys to form such complex oxides causing a significant improvement over chromia-forming alternatives [52]. In conclusion, four alloy development routes are feasible if the addition of

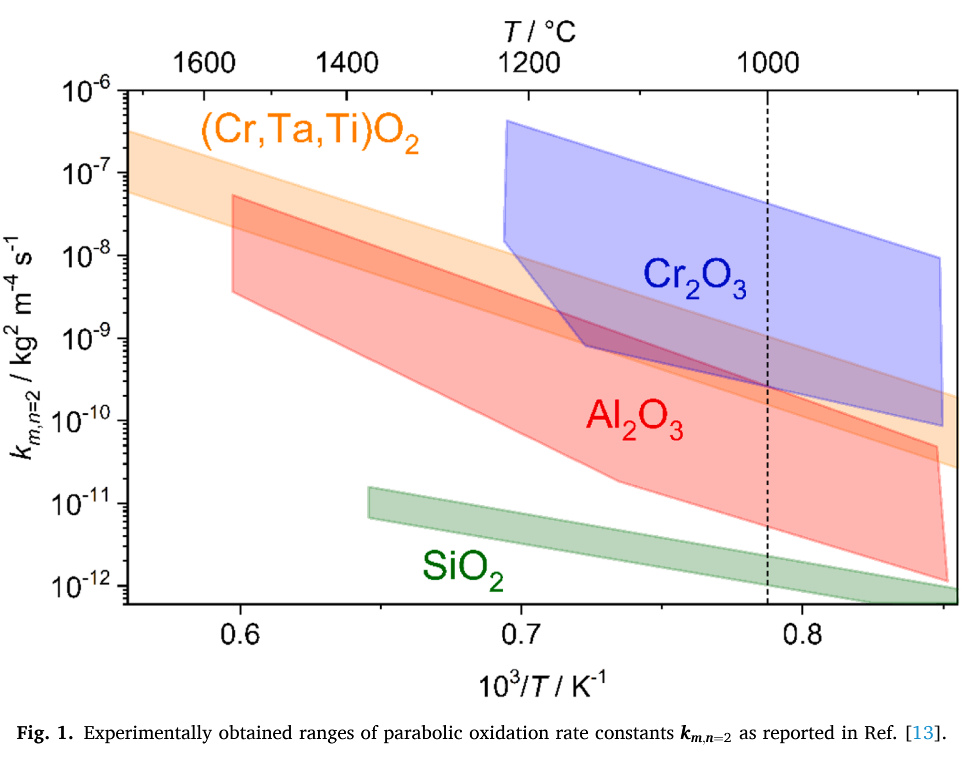

Fig. 1. Experimentally obtained ranges of parabolic oxidation rate constants $k_{m,n=2}$ as reported in Ref. [13].

the required elements to form passivating scales are compatible with the BCC superalloy paradigm which seems questionable for substantial amounts of Si to form SiO_2.

Relevant BCC superalloy systems

Al_2O_3 and Cr_2O_3 are the relevant oxide scales formed on Ni-based superalloys [1,2,48]. When highest application temperatures in flowing gas are anticipated, Cr_2O_3 is avoided as it reacts to volatile CrO_3 at the scale surface above 1000°C [53] and loses protectiveness. However, this does not exclude Cr from being a crucial alloying element, as the combined alloying of Cr and Al to Ni has been found synergistic [1,48]. The selective oxidation of Cr initially prevents internal corrosion of Al and allows for the subsequent formation of a continuous Al_2O_3 layer beneath Cr_2O_3 and NiO + $Ni(Cr,Al)_2O_4$ in the following. In essence, much lower Al contents are required in ternary Ni-Al-Cr and multinary Ni-Al-Cr-based alloys to promote protectiveness by Al_2O_3 in comparison to binary Ni-Al alloys. A similar trend is found for BCC, ferritic Fe-Cr-Al alloys which are commercially used for heating elements, albeit at higher Cr levels [48]. However, these alloys do not compete with Ni-based superalloys' creep resistance even in dispersion-strengthened conditions. Considering lower density and costs, B2 and $L1_2$ strengthened Fe-based superalloys [33–42] are interesting in this sense for intermediate application temperatures when derived from existing Fe-Cr-Al solid solutions.

When higher Al contents in Ni and Fe are considered, the B2 intermetallic compounds NiAl and FeAl are obtained which attracted substantial research interest not only as strengthening phases in BCC superalloys but as base compounds for high temperature alloys [22–24, 54–56]. In single-phase condition, the formation of Al_2O_3 was verified but in different crystallographic modifications [22–24]. The anticipated growth of dense, thin and slowly growing $D5_1$ α-Al_2O_3 was only obtained at temperatures above about 950°C. At lower temperatures, the θ-Al_2O_3 (no Strukturbericht designation, β-Ga_2O_3 prototype [57]) and $D5_7$ γ-Al_2O_3 (γ-Fe_2O_3 prototype [57]) polymorphs are found that grow at substantially higher growth rates. Specifically, θ-Al_2O_3 exhibits non-dense, needle- and platelet-shaped morphology. The Al depletion beneath the scale by oxidation to Al_2O_3 leads to an opposite Ni/Fe concentration gradient and inward diffusion of Ni/Fe. This causes void formation below the oxide. The addition of Cr, for example to generate two-phase, eutectic NiAl and Cr composite microstructures [22–24] does not lead to an improvement of the oxidation behavior in contrast to what is observed for Ni-Al-Cr solid solutions [48]. With respect to the oxidation behavior of BCC superalloys with B2 precipitates, solute partitioning between A2 and B2, the volume fractions of the corresponding B2 phases as well as their sizes might be decisive.

Recently, Cr-based superalloys with B2 NiAl precipitates were introduced [20,21]. Fe addition in this system enhances the maximum solubility of Ni and Al in the BCC (Cr,Fe) solid solution and, thus, allows for tailoring of the B2 volume fraction beyond the about 5 vol.% investigated in Ref. [20]. In the case of Cr-based alloys, Cr_2O_3 scales are formed [53]. Apart from the mentioned limitations beyond 1000°C [53], spallation and nitridation are particular problems in these cases. A comparably large difference in volume between Cr and Cr_2O_3 as well as differences in thermal expansion are accounted for this. N ingress into the substrate can lead to the formation of nitrides in the substrate and close to the scale/substrate interface. Improvements were obtained by additions of Si [18], Si+Mo as well as Si and further elements [19,58, 59]. A recent study on concentrated BCC Cr-Mo-Si solid solutions indicates that SiO_2 assisted the formation of Cr_2O_3 with Mo enrichment in the subscale region within the substrate [60]. In this case, Cr_2O_3 provides oxidation protection while Mo enrichment suppresses nitridation of the bulk alloy. Remarkably, the single-phase BCC solid solution alloys exhibit compression ductility at room temperature and the activation of deformation twinning. The application of the alloying principle to form B2 precipitates [20] in Si-containing, high Mo-containing Cr alloys [60] seems a logical future developmental direction.

SiO_2 is predominantly investigated as protective oxide on refractory-metal containing alloys, particularly based on Mo [12] and Nb [14]. Fundamental issues here are the catastrophic behavior of refractory elements by the formation of either voluminous or volatile oxides [61]. As silicide phases have proven high temperature oxidation resistance, metallic-intermetallic materials were developed. However, most of the development focuses on high temperature oxidation resistance while only a few high refractory-element containing alloys withstand catastrophic oxidation at intermediate temperatures [62,63]. With respect to BCC superalloys, this is particularly relevant for B2 TiFe strengthened, Ti- or (W,Ti)-based alloys as recently suggested in Refs. [45,44,17]. W suffers from porous and fast-growing WO_3 that evaporates beyond 1000°C [61,64–66]. There are currently no design strategies to mitigate the catastrophic oxidation of W in air. In the case of Ti-based variants with B2 TiFe [45], an experimental high throughput study indicated non-protective, predominant TiO_2 even when Al and Cr are added. As Ta forms porous and fast-growing Ta_2O_5, B2 Ta-rich matrix alloys with A2 (Ti,Zr,Al,Mo,Nb) precipitates suggested in the pioneering BCC superalloy work by Naka and Khan [46] will be challenging.

Refractory high entropy alloy (RHEA) or compositionally complex alloy (RCCA) concepts exhibit the largest compositional degrees of freedom among the present alloying concepts. Indeed, several solid state transformed systems with A2 + B2/B2 + A2 microstructures were recently identified in literature [32]. Latest developments address the significant improvement of solvus temperatures [26–28] which seems required to meet creep resistance targets [67]. The oxidation behavior of actual RHEAs and RCCAs might be categorized as following [13]: (i) Some alloys primarily suffer from severe O solubility in the substrate, leading to extensive crack formation in the O-rich subscale region, for example in alloys with high Nb or Ta contents. (ii) Similarly detrimental is the fast formation of oxide mixtures with porous or lamellar morphology, often resulting from repetitive exfoliation of the substrate. Two categories of alloys exist that allow for reasonable or even very good oxidation resistance. (iii) Rutile-type $(Cr,Ta,Ti)O_2$ features relatively protective oxide scales, see Fig. 1, originating from the reaction between Cr_2O_3 and Ta_2O_5. Since Ta_2O_5 remains monomorphic up to 1350°C, this Ta-containing rutile is more beneficial compared to for example Nb-containing $(Cr,Nb,Ti)O_2$ derived from polymorphic Nb_2O_5. Finally, (iv) alloys exist that form protective $D5_1$ Al_2O_3 scales, which can be potentially supported by reactive elements like Si or Hf.

Conclusions and challenges

While the search for ultimate oxidation protection might fail and even be unnecessary as coatings are required anyway, reasonable emergency resilience deems mandatory for cases of coating failure. However, general metallurgical design rules on systematic development (which for example exist for tailoring mechanical strength) or predictive simulation capabilities to induce oxidation resistance are still limited or not existing and are to be developed. Hence, several major aspects are identified that need to be addressed in future work:

- Specifically, the refractory-metal based systems suffer from evaporating oxides, spallation or a combination thereof. The physical relevance of mass change is then limited as uptake by internal oxidation/scale growth might be outbalanced by evaporation/ spallation. Investigations must be accompanied by detailed tracking of scale growth. The partially erratic nature of these phenomena requires statistically relevant assessments.
- From an engineering perspective, transient and long-term oxidation under static and cyclic conditions are to be systemically investigated, not only at maximum application temperature but also at intermediate, critical temperatures if catastrophic oxidation or pesting is possible.

- Efforts to develop consistent and predictive simulation capabilities need to be undertaken covering the complex superposition of thermodynamic, kinetic, chemical and mechanical aspects of oxidation. This should be assisted by machine learning strategies that are currently developed [68–72]. The shortcomings of mass change data as input data need to be addressed and corresponding design rules should be extracted from the computer assisted searches. As large datasets are required, high-throughput experiments (like in Ref. [45]) and simulations need to be performed to feed these analyses. Furthermore, even results on unprotective alloys should become publicly available, specifically if other design criteria are met like mechanical properties.
- As the research is still in the status to identify suitable BCC superalloy systems, assessment of the impact of microstructural variations is still illusive which needs to be addressed for promising base systems.
- Finally, oxidation resistance in static gas environments is one aspect of high temperature corrosion which is usually quite different from corrosion in moving gases as well as in hydrogen-containing, humid or even more complex combustion atmospheres.

CRediT authorship contribution statement

Alexander Kauffmann: Writing – review & editing, Writing – original draft, Visualization, Data curation, Conceptualization. **Bronislava Gorr:** Writing – review & editing, Writing – original draft, Visualization, Formal analysis. **Martin Heilmaier:** Writing – review & editing, Writing – original draft, Supervision, Conceptualization.

Conflict of interest

The authors declare no conflict of interest.

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