

Creep of binary Fe-Al alloys with ultrafine lamellar microstructures

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Abstract

On the Al-rich side of the Fe-Al binary system, the eutectoid decomposition of Fe₅Al₈ into B2-ordered FeAl and triclinic FeAl₂ in the composition range of 55 – 65 at.% Al produces an ultrafine lamellar microstructure. The compression creep behavior of such two-phase intermetallic materials was investigated in the temperature range 600 – 800 °C under constant stress. In addition to the fully lamellar Fe-61Al alloy, Fe-58Al and Fe-62Al that included, pro-eutectoid FeAl and FeAl₂, respectively, were characterized in terms of their microstructure and creep response. For all microstructures, the strain rate as a function of time and strain exhibits a distinct minimum instead of a steady state creep regime. Microstructure instability, primarily in the vicinity of colony boundaries, is identified as the main reason for the increase in strain rate beyond the minimum. In contrast, lamellar coarsening is shown to be only a secondary factor influencing creep response for the conditions investigated. In comparison to single phase FeAl, the fully lamellar FeAl-FeAl₂ shows enhanced creep resistance while the presence of either pro-eutectoid phase leads to a relative deterioration of the creep resistance.

1 Introduction

Iron aluminides are of interest as an alternative to advanced steels intended for high temperature structural applications. Their excellent intrinsic oxidation resistance [1] [2] [3], lower density and material costs make them attractive candidates in potentially replacing steels in some of these applications [4] [5] [6] [7]. However, their widespread use is limited mainly due to (i) low ductility at room temperature [8] and (ii) reduced creep resistance at elevated temperatures beyond 600 °C. It has been shown, though, that the creep resistance of B2-ordered FeAl can be improved by classical physical metallurgy approaches such as solution strengthening with alloying elements like Ti, Zr, Mo [9] [10] [11], precipitation strengthening by addition of C or B which form carbides and borides respectively [9],[11], and dispersion strengthening, for instance, using Y₂O₃ nanoparticles [12]. Another strategy for improving creep resistance is to utilize a lamellar microstructure that may be obtained through a eutectic or eutectoid decomposition process. Thus, in the case of TiAl alloys, it has been shown that a fine, lamellar microstructure can substantially improve creep resistance [13].

A lamellar arrangement of two intermetallic phases has been observed in the Fe-Al-system as well in the composition range 55 – 65 at.% Al based on the eutectoid decomposition of the high-temperature ϵ -phase Fe₅Al₈ (space group: $I\bar{4}3m$, no. 217; Pearson symbol: $cI52$) [14] into B2-ordered FeAl ($Pm\bar{3}m$, no. 221; $cP2$) and triclinic FeAl₂ ($P\bar{1}$, no. 2; $aP19$) [15]. The eutectoid decomposition occurs at 60.9 at.% Al and 1095 °C [16]. The mechanical properties at high temperature of such in-situ composites have not been investigated in detail so far. In the present study, compression creep experiments of eutectoid and off-eutectoid binary compositions were carried out in the temperature range 600 – 800 °C; the deformed microstructures were examined and correlated with the observed mechanical response.

2 Experimental

The materials were produced by arc melting Al (99.99 %) and Fe (99.99 %) bulk elements. The chamber was purged with Ar and evacuated multiple times in an effort to reduce the oxygen partial pressure, and the final pressure in the chamber was maintained at 60 kPa. Additionally, a

lump of Zr metal was melted in the chamber prior to melting the Fe-Al alloys to further reduce residual oxygen. The Fe-Al buttons were flipped and re-melted five times to ensure homogenization. Final rods with dimensions of 14 mm diameter and 170 mm length were achieved by drop casting into a water-cooled Cu mold.

Three alloy compositions corresponding to Fe-58Al, Fe-61Al and Fe-62Al (at.%) as well as single phase FeAl as stoichiometric Fe-50Al were produced. Fe-61Al is expected to result in a fully lamellar microstructure according to the published binary phase diagram [16] whereas Fe-58Al and Fe-62Al contain a significant fraction of pro-eutectoid FeAl and FeAl₂ respectively. Additionally, for comparison single-phase FeAl₂ was prepared by levitation melting; a subsequent long-term heat-treatment is necessary to produce this single-phase upon arc melting because FeAl₂ does not form congruently from the melt and instead a non-equilibrium microstructure results in the as-cast state [16]. The as-cast microstructures were investigated using scanning electron microscopy (SEM). The samples were prepared by standard metallographic procedures including mechanical grinding followed by polishing with 1 μm diamond suspension. Final polishing was performed using a non-crystallizing oxide polishing suspension OP-S provided by Buehler, Illinois, USA. The average lamellar spacing was measured in the as-cast state for all three alloys and in the eutectoid alloy after exposure to 700 °C for various times, and after creep testing. The experimental approach to determine lamellar spacing is described elsewhere [17]. The volume fraction of the pro-eutectoid phase was evaluated utilizing imaging analysis software (AnalySis).

Cylindrical samples with a diameter of 5 mm and a height of 8 mm were electrical discharge machined for creep experiments. The samples were isothermally crept in compression, either at a constant true stress in vacuum, or at constant load in air. Please, note that until the minimum is reached due to the small amount of strain no difference can be observed between constant true stress vs. constant load and even after 10 % of strain no significant influence was found in the present case. The tests were carried out in the temperature range between 600 – 800 °C and the applied stress was varied from 50 to 175 MPa.

3 Results and Discussion

3.1 Initial microstructure

In the as-cast state, all three alloys exhibit a lamellar arrangement of FeAl and FeAl₂ and include morphological irregularities like terminations and branches (Figure 1a-c). In the off-eutectoid Fe-58Al and Fe-62Al alloys, in addition to the lamellar morphology composed of FeAl and FeAl₂, pro-eutectoid FeAl and FeAl₂ are observed respectively. Average lamellar spacing and the associated standard deviations were determined in the as-cast state for the three alloys and are summarized in Table 1. The experimentally measured volume fractions of the pro-eutectoid phases are listed as well in Table 1 and match reasonably well with the values calculated using the binary phase diagram in [16].

Table 1: Microstructure data in the as-cast state

Composition	lamellar spacing / nm	Pro-eutectoid phase	Measured volume fraction of pro-eutectoid particles / vol%	Calculated volume fraction of pro-eutectoid particles / vol%
Fe-58Al	400 ± 50	FeAl	31 ± 4	30
Fe-61Al	380 ± 40	–	–	–
Fe-62Al	400 ± 90	FeAl ₂	33 ± 5	28

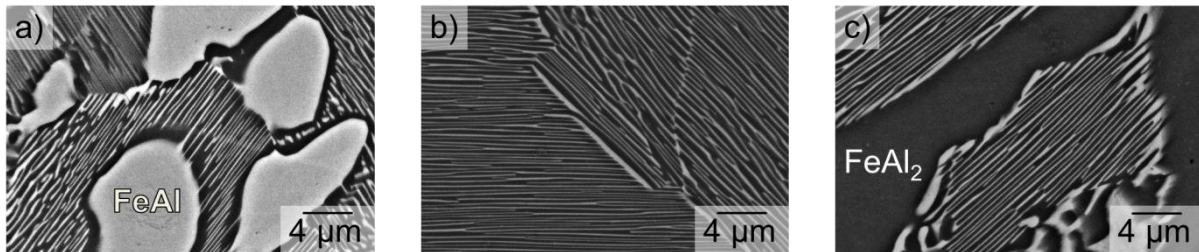


Figure 1: Backscattered electron images of the investigated alloys in the as-cast state: a) Fe-58Al, b) fully lamellar Fe-61Al and c) Fe-62Al.

3.2 Creep behavior of the fully lamellar material

The creep response at 700 °C and nominal constant stress of 100 MPa for the eutectoid alloy with the lamellar microstructure is shown in Figure 2a, b. The data are shown as logarithm of strain rate versus strain in Figure 2a, and as logarithm of strain rate versus time in Figure 2b. The resulting creep curve does not exhibit a “classical” steady-state regime of constant strain rate over a measurable period of strain or time. Instead, a characteristic minimum occurs, which is dependent on temperature and stress (these relations are presented later in Figure 8 and 9). After passing through the minimum, the strain rate markedly increases with increasing time and strain at 700 °C (Figure 2a, b).

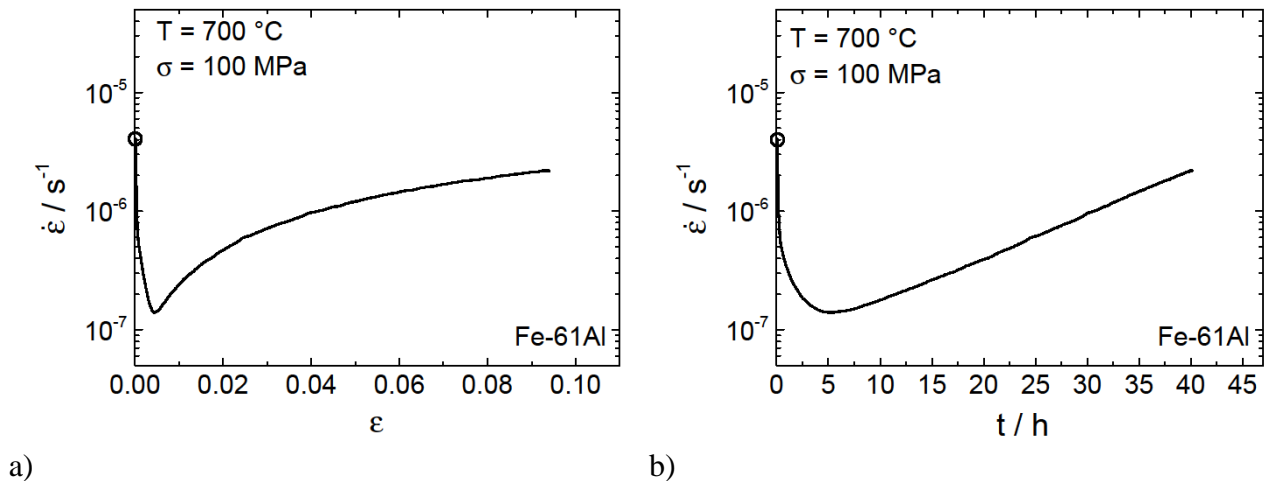


Figure 2: Creep response of a lamellar Fe-61Al alloy at 700 °C and an applied stress of 100 MPa: a) log strain rate vs. strain and b) log strain rate vs. time. The circles indicate the initial strain rate.

The increase in the creep rate beyond the minimum for the eutectoid alloy presented above is thought to be a result of microstructural changes along the lines of previous reports in the literature [18], [19]. We note here that a similar creep response was also observed for the off-eutectoid alloys. Common to all three alloys is the presence of a rather large fraction of the lamellar microstructure and changes associated with it are believed to be responsible for the increase in strain rate beyond the minimum. In general, the strength and creep resistance at high temperature of lamellar materials strongly depend on the lamellar spacing [20], [21] and on the

lamellar orientation with respect to the loading axis [22]. Lamellar orientation to the loading axis is of minor importance in the present case since a polycrystalline, non-textured as-cast material is investigated here. An increasing lamellar spacing can lead to a decrease in high temperature strength and creep resistance by at least three distinct microscopic mechanisms [21]: a) lamellar coarsening enables a longer dislocation glide distance within a lamella, b) the shear stress to bow out dislocations trapped at the interface decreases with increasing lamellar spacing [23], and c) the ability to operate Frank-Read sources is easier in coarser lamellae. Coarsening of a lamellar microstructure can be continuous or discontinuous depending on the test temperature and applied stress. For example, in the case of fully lamellar Ti-Al alloys, discontinuous coarsening of the lamellar microstructure is observed at lower stress levels [24], whereas at higher stresses, deformation-induced spheroidization occurs [25]. In both cases, the deterioration in the lamellar microstructure is reported to be the major reason for an increase in creep rate.

Thus, the evolution of lamellar spacing as a function of time and strain was determined by measuring the average lamellar spacing λ after various times of thermal exposure and mechanical loading, respectively. An increase in average lamellar spacing reveals that coarsening takes place during creep (Table 2).

Table 2: Evolution of average lamellar spacing at 700 °C at an applied stress of 100 MPa for the fully lamellar Fe-61Al alloy

condition	time / h	strain	lamellar spacing λ / nm
as-cast	–	–	380 ± 40
T = 700 °C σ = 100 MPa	5	0.01	390 ± 70
	30	0.05	520 ± 70
	50	0.20	680 ± 110

In order to separate the effect of thermal and mechanical loading on the coarsening behavior, a heat treatment at 700 °C without applied stress was carried out as well. The resulting change in average lamellar spacing as a consequence of thermal exposure alone is plotted as a function of time using filled symbols in Figure 3. In order to compare the long term (up to 5000 h) coarsening behavior due to thermal exposure with the data of the creep experiment (open

symbols in Figure 3) a double logarithmic plot was used. The coarsening behavior of lamellar structures has been studied in [19-21] and is based on a reduction of total interfacial energy by one or more of several mechanisms including: a) spheroidization [28], [29], b) cylinderization [28], [29], c) thermal grooving [30] and/or d) termination/migration [31]. A recent detailed study of the coarsening of lamellar FeAl and FeAl₂ confirmed that the process is volume diffusion-controlled and follows a classical ripening law as

$$\lambda^3 = \lambda_0^3 + kt \quad [32], \quad (1)$$

where λ is the average lamellar spacing, λ_0 the initial average lamellar spacing and k a constant. The superposition of stress on the coarsening response does not appear to influence the observed trend for times up to 50 h as shown by the open symbols in Figure 3. Hence, we conclude that in the regimes of temperature and stress examined in this study, coarsening of the lamellae is mainly a consequence of thermal exposure during creep.

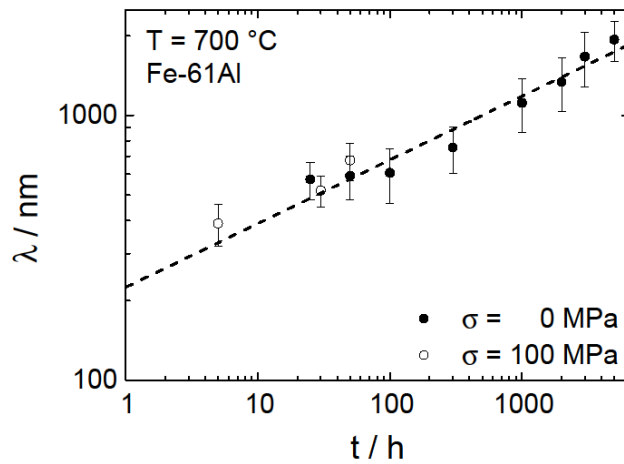


Figure 3: Change in average lamellar spacing with time at 700°C with (open symbols) and without (filled symbols) an applied stress of 100 MPa for the fully lamellar Fe-61Al. The dashed line is added to guide the eyes and helps to visualize that the coarsening during creep is comparable to the only heat treated material.

To investigate the effect of increasing lamellar spacing on creep behavior, a previously heat-treated sample (700 °C for 300 h) with a coarser average lamellar spacing of 760 ± 150 nm was tested. Figure 4a compares the creep behavior of this sample with that for the as-cast material

with a finer average lamellar spacing of 380 ± 40 nm. A careful examination of Figure 4a confirms that in the early stages of the experiment (roughly within the first percent of strain), the heat-treated microstructure creeps at a faster rate than does the as-cast material which has roughly half the former average lamellar spacing; however, at larger strains (and beyond the minimum in creep rate in Figure 4a), the creep rates converge and the effect of lamellar spacing on creep rate gradually diminishes. This suggests that changes in average lamellar spacing due to coarsening are unlikely to be the primary cause for the degradation in creep response beyond the minimum creep rate noted in Figure 4a.

An additional experiment was conducted to more vividly demonstrate that increases in average lamellar spacing have only marginal (if any) influence on the accelerated creep response beyond the minimum creep rate by using a single specimen that was initially in the as-cast state. In doing so, inevitable variations of the initial microstructures in different specimens can be excluded. Here the sample was first loaded at 700 °C and 100 MPa and allowed to reach the minimum strain rate of $2 \cdot 10^{-7} \text{ s}^{-1}$ (Figure 4b) which is in good agreement with the minimum strain rate previously observed in the as-cast condition in Figure 2a. The sample was then unloaded and allowed to isothermally coarsen for 20 h at this test temperature (note that the total plastic strain at the minimum strain rate is small and will not affect the coarsening rate appreciably). Following that, it was reloaded to 100 MPa and strain evolution was again monitored as a function of time. The results are shown for this second step in Figure 4b as well. A second minimum is noted beyond which the strain rate increases again. The occurrence of a minimum in the second loading step is puzzling. It suggests that during the isothermal coarsening, the structure must have recovered and subsequent loading enables it to harden. However, considering that the measured global plastic strain is less than one percent at the minimum strain rate (Figure 4a), this is unlikely unless there is significant plastic strain partitioning between the two phases - i.e. one phase plastically deforms in the early stages while the other is elastic and the plastically deforming phase recovers during the intermittent anneal; this hypothesis remains to be verified.

Returning to Figure 4b, a dashed line is incorporated that depicts what should have happened if the initial loading had not been interrupted at the minimum strain rate and creep had continued in accordance with Figure 2b. The 20 h exposure to 700 °C following the initial loading should result in an average lamellar spacing of the order of ~ 520 nm (Figure 3) as compared to 379 nm for the as-cast condition. If coarsening was the decisive factor controlling strain rate evolution

beyond the minimum, reloading should result in a strain rate evolution that is coincident with this extrapolation.

The second minimum, however, is observed at $4 \cdot 10^{-7} \text{ s}^{-1}$ which is significantly lower than the value predicted by the dashed curve (about $1.5 \cdot 10^{-6} \text{ s}^{-1}$). Subsequent increase in strain rate with time beyond this minimum parallels the extrapolated dashed line. For these reasons, the change in lamellar spacing cannot be the primary reason for the experimentally observed increase in strain rate beyond the minimum strain rate in Figures 2a, b as well as in Figure 4a.

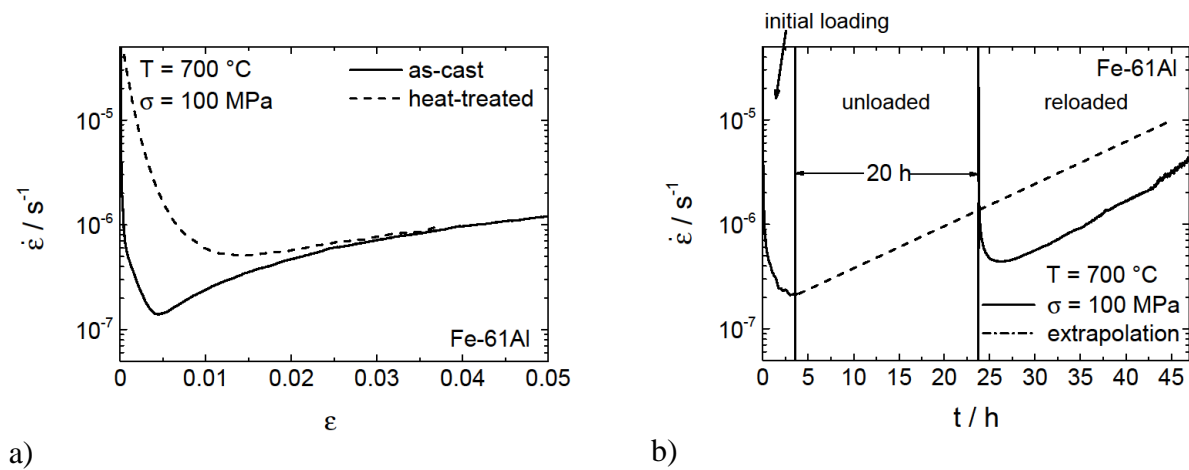


Figure 4: The effect of changing lamellar spacing on creep response under constant true stress: a) comparison of an as-cast specimen (solid line) with a specimen initially heat-treated at 700 °C for 300 h (dashed line) to alter the lamellar spacing; b) a single specimen that was initially in the as-cast state and was crept to the minimum, then unloaded and in-situ coarsened for 20 h and then subsequently reloaded.

Since lamellar coarsening has been ruled out as the primary cause of the increased creep rate beyond the minimum in Figure 4a and 4b, it is necessary to consider other aspects of microstructural degradation. Previous work on the creep of lamellar TiAl has shown that the fine structure of colony boundaries can control creep resistance. Specifically, smooth colony boundaries can lead to colony boundary sliding [33] and crack growth [34] whereas interlocking boundaries have been shown to promote a distinct steady-state regime [35] and, thus, to enhance creep resistance [33]. In the lamellar Fe-Al alloys examined in this study, the microstructure in

the vicinity of colony boundaries in the as-cast state reveals various “irregularities” relative to the periodicity exhibited within the colonies; in some instances, the minor phase in the lamellar structure displays terminations short of reaching the boundary (Figure 5b), leaving a 1 – 5 μm region of single phase layer of the majority phase, while at other boundaries, the lamellar morphology in the adjacent colonies extends right up to the colony boundary but the lamellae themselves are mismatched across the boundary (Figure 5c). Furthermore, along some colony boundaries but not others, FeAl precipitates are present as stringers (Figure 5b).

Microstructure degradation during creep in these colony boundary regions is accelerated compared to the interior due to stress incompatibilities, elastic anisotropy and lamella terminations. Thus, subsequent to creep exposure of 50 h at 700 °C at a constant stress of 100 MPa (this corresponds to a global plastic strain of about 0.2), the scale of microstructure and microstructural morphology in the vicinity of the colony boundaries has significantly changed as compared to the colony interior and as depicted in Figure 5d (white arrows indicate one such region). There now exists a region of 10 – 20 μm width in the vicinity of colony boundaries where the lamellar structure is no longer evident but is replaced by enlarged single-phase regions of FeAl and FeAl₂. If we argue that this microstructural degradation in the vicinity of colony boundary contributes to the enhanced creep rate, then it follows that FeAl₂ likely contributes to high temperature creep deformation, implying that this phase with its triclinic crystal structure experiences plastic flow at 700 °C. Anyway, it would be unrealistic to observe a plastic strain of 0.2 without any plastic flow of the FeAl₂ phase that constitutes to about 70 vol% in the present large colony size material. In a detailed crystallographic study presented elsewhere [36], it was shown that the (114)[$\bar{1}\bar{1}0$], ($\bar{1}\bar{1}3$)[$\bar{1}\bar{1}0$] and (221)[$\bar{1}\bar{1}0$] of FeAl₂ exhibit similar atomic arrangement to the { $\bar{1}\bar{1}0$ }{001} slip system in B2 FeAl [37] and it seems not unrealistic to assume that slip occurs on these systems in FeAl₂ at elevated temperatures; however, this remains to be verified. Hence, we hypothesize that one of the two phases deforms plastically in the early state of creep whereas the minimum in creep rate could signal the onset of plastic deformation of the second phase; plastic co-deformation of the two phases can subsequently lead to a progressive loss in microstructure stability by dynamic recovery/recrystallization of the individual phases, more so in the vicinity of colony boundaries but to a lesser extent within the colony themselves leading to an increase in strain rate. If this is the case, then, it implies that changes in creep

mechanism during the course of the constant stress experiment can cause significant difficulties in the interpretation of the experimentally determined activation energy for creep.

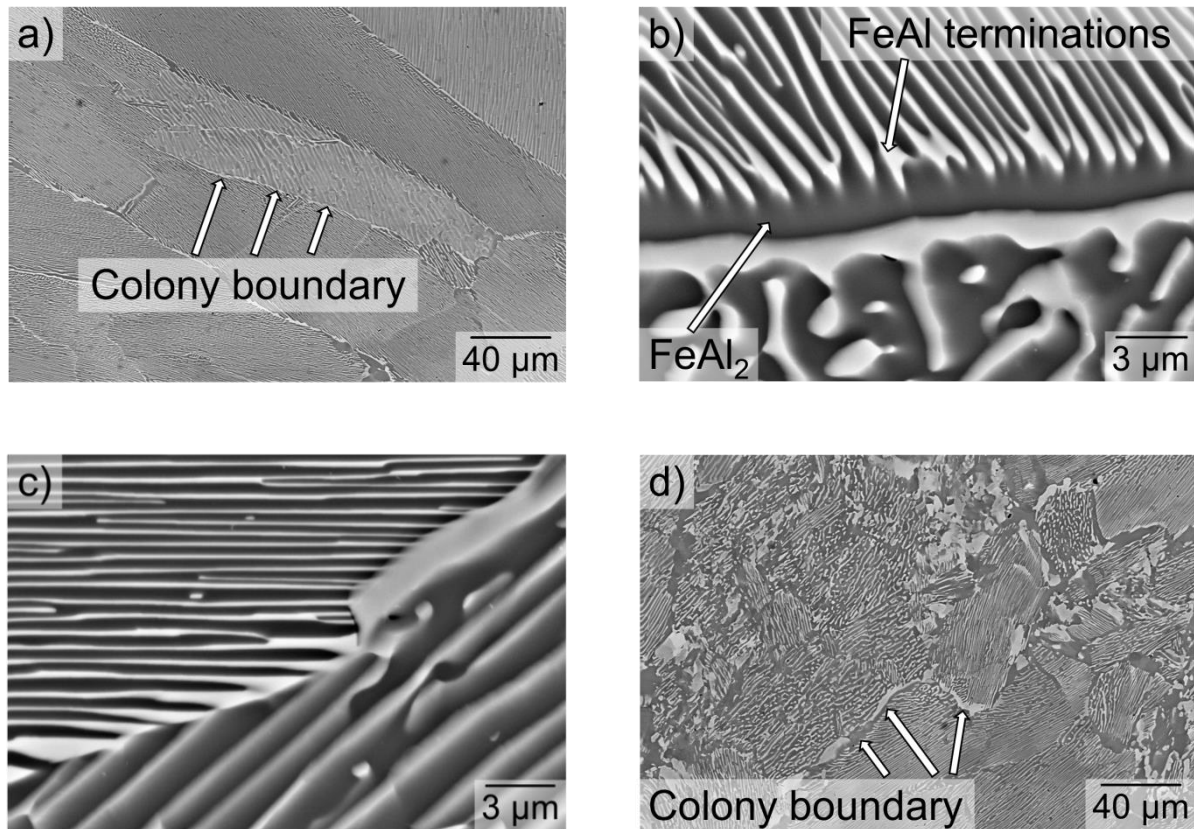


Figure 5: BSE micrograph of the fully lamellar Fe-61Al alloy in the (a-c) as-cast condition: (a) general microstructure, and (b,c) higher magnification images showing FeAl lamellae terminating short of the colony boundary in (b) and FeAl lamellae mismatch across colony boundary in (c). d) Microstructure coarsening at colony boundaries after 50 h of creep at 700 °C and 100 MPa.

3.3 A comparison of the creep response of Fe-58Al, Fe-61Al and Fe-62Al

The effect of the presence of pro-eutectoid FeAl or FeAl₂ in a lamellar FeAl/FeAl₂ matrix on creep response is illustrated in Figure 6 along with the behavior of the eutectoid alloy. Again, the comparison is made at 700 °C and 100 MPa and all creep curves show a pronounced minimum in creep rate. The fully lamellar alloy exhibits the lowest minimum strain rate of the three alloys. It appears that the presence of the globular pro-eutectoid FeAl or FeAl₂ phases (and a corresponding reduced amount of the lamellar eutectoid) leads to a deterioration in creep resistance. Further, beyond the strain rate minimum, the increase in creep rate with strain is affected by the presence and type of pro-eutectoid phase. Whereas the increase for the fully

lamellar Fe-61Al alloy and the aluminum-lean Fe-58Al alloy run almost parallel to each other, the aluminum-rich Fe-62Al exhibits a more gradual change intersecting the curve for the Fe-61Al alloy at a strain of ~ 0.04 (Figure 6) and thereafter improved creep resistance relative to the eutectoid alloy is noted. This response may be related to the fact that FeAl₂ is more creep resistant than FeAl (see Figure 7).

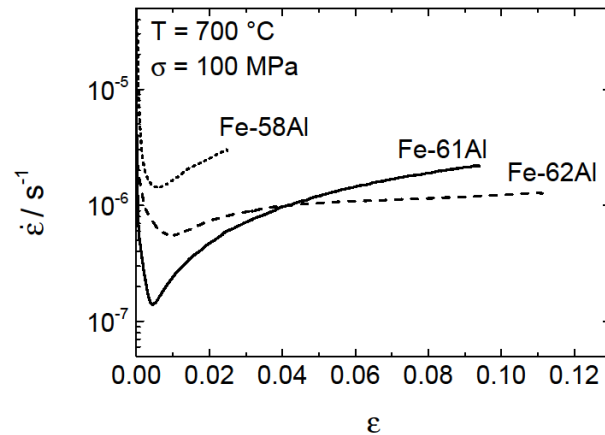


Figure 6: A comparison of the creep response of Fe-58Al, Fe-61-Al and Fe-62Al alloys at 700 °C and an applied true stress of 100 MPa.

To further explore this line of thinking, creep tests under similar conditions (100 MPa; 700 °C) were performed on single phase stoichiometric FeAl and single phase FeAl₂ specimens and the results are compared in Figure 7 against the response of the fully lamellar Fe-61Al eutectoid alloy. In Figure 7, the minimum strain rate for FeAl₂ (dashed line) is comparable to the one for the fully lamellar material (continuous curve). Post-deformation examination of the crept specimen showed that on a macroscopic scale, the specimens had deformed homogeneously despite the presence of minor levels of porosity in these specimens. The creep resistance of FeAl₂ is clearly superior to that of stoichiometric FeAl (dotted line) (note: The composition of FeAl in equilibrium with FeAl₂ at 700 °C is only slightly Al-rich compared to stoichiometric FeAl) but the presence of a fine lamellar structure is morphologically beneficial for creep in that the creep of the weaker phase (FeAl) is constrained by the more creep-resistant FeAl₂ phase in the early stages of creep. In this sense, a fully lamellar microstructure provides optimum creep resistance as was previously observed for TiAl as well [13]. However, for longer exposure times, and beyond the creep rate minimum when lamellae degradation, particularly in the vicinity of the

colony boundaries commences, the volume fraction of the more creep-resistant FeAl_2 phase (proeutectoid globules) can become rate-controlling as seen in Figure 6.

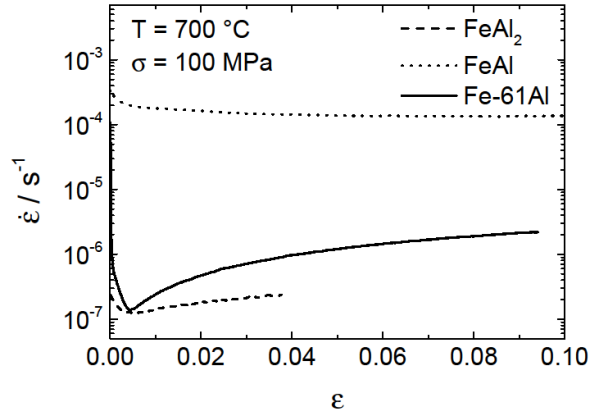


Figure 7: Creep response of single-phase stoichiometric FeAl and FeAl_2 compared to fully lamellar, two-phase Fe-61Al at $700\text{ }^\circ\text{C}$ and an applied true stress of 100 MPa .

3.4 Rate controlling creep mechanism and activation energy

Based on the above results, the creep response of these three two-phase Fe-Al alloys can be separated into two parts: 1) creep until the minimum strain rate which was reached at a strain less than one percent, and 2) creep beyond the minimum and up to strains as large as 20 percent that was reached in about 50 hours, at which point, the eutectoid lamellar morphology in the vicinity of colony boundaries appeared to have deteriorated (Figure 5d). To correctly interpret the measured creep exponents and activation energies from these experiments, it is important to understand the dominant deformation mode(s) through the duration of such experiments. For example, a) both phases may contribute to the creep deformation from the beginning and it is the degradation of the lamellar morphology that is responsible for the increase of the strain rate beyond the minimum; b) only one of the two phases deforms and the second phase does not contribute directly to creep throughout the entire experiment, and the degradation of the lamellae causes the increase of the strain rate beyond the minimum, or c) the second phase starts to creep-deform beyond the minimum causing the increase in the strain rate and subsequently, lamellar degradation enters the picture as well.

Since the creep strain at which the minimum creep rate occurs is less than one percent, it is conceivable that this relatively small global plastic strain can be accommodated predominantly

within one phase with little or no permanent deformation of the second phase. This implies that cases b) and c) described above are possible. The FeAl phase is significantly less creep-resistant than the FeAl₂ phase as presented in Figure 7. If it is assumed that until the strain rate minimum is reached, the plastic deformation is only supported by FeAl, then a change in the stress exponent as well as in the activation energy would be expected beyond the minimum.

We now examine the minimum strain rate (\leq one percent of plastic strain) and the strain rate at a plastic strain of 0.04. The second value was chosen since the increase in the strain rate beyond the minimum diminishes after a strain in the range of 0.02 to 0.04 (Figure 7), and thereafter, to a first approximation, it is viewed as approaching a steady-state regime. The stress dependence of the minimum strain rate (filled circles in Figure 8) as well as the strain rate at a strain of 0.04 (open circles in Figure 8) at 700 °C for different imposed stress levels is summarized in a Norton plot as a double logarithmic representation of minimum strain rate versus stress (Figure 8). The power law stress exponent was determined according to:

$$\dot{\epsilon} = A \cdot \sigma^n \quad (2)$$

where $\dot{\epsilon}$ is the strain rate, A is a temperature-dependent constant and σ is the applied stress. The resulting stress exponent n is 3.0 for the minimum creep rate and 3.3 for the strain rate at a strain of 0.04. A stress exponent between 3 and 5 in a single-phase alloy typically signifies that creep (rate) is controlled by thermally activated dislocation glide and climb [38], [39]. However, in this study, we are dealing with a two-phase lamellar microstructure where it is uncertain if both phases are experiencing permanent deformation or if one phase deforms only elastically, and also whether the response remains the same at the minimum strain rate and at the strain rate at a strain of 0.04. It is also important to know the creep characteristics of the individual phases and how they work together in a fine lamellar structure that includes a high density of interfaces.

In previous studies on FeAl single-phase material, a stress exponent of 3.5 was obtained for deformation in the temperature range of 600 °C – 1000 °C and at applied stresses between 50 and 200 MPa [40]. The operative slip systems in this temperature range were determined to be $\{1\bar{1}0\}\{001\}$ and $\{100\}\{001\}$ [37]. The stress exponent obtained in this study for single-phase FeAl₂ (open rhombi in Figure 8) is 3.1 and also indicative of dislocation glide/climb dominated creep. As both of these phases that constitute the eutectoid microstructure deform by dislocation-mediated creep under the imposed temperature and stress regime, it is difficult from the stress

exponent value alone to determine whether one or both phases is/are responsible for creep at the minimum strain rate or at the strain rate at a strain of 0.04.

When creep is climb-controlled, vacancy diffusion kinetics influence the creep rate, and thus, an examination of the activation energies could provide additional information on the operating mechanism. In order to determine the activation energy, creep experiments were carried out at different temperatures at a constant stress level of 100 MPa. The minimum strain rates obtained are plotted as a function of inverse temperature in Figure 9 for the eutectoid and off-eutectoid alloys together with the strain rates determined at a plastic strain of 0.04 for the eutectoid composition. The values for the activation energy obtained from the slopes of these Arrhenius plots for the fully lamellar Fe-61Al alloy are (321 ± 26) kJ/mol and (338 ± 21) kJ/mol for the minimum strain rate and the strain rate at a strain of 0.04, respectively. Both these values are above those reported in the literature by Eggersmann et al. [41], [42] for self-diffusion of Fe (265 kJ/mol), as well as for diffusion of In and Zn in B2-ordered Fe-50Al (258 kJ/mol); In and Zn are the usual substitutes for Al due to the lack of suitable Al tracers [42]. The values provided by Eggersmann [41], [42] are also supported by Nakamura et al. [43] for off-stoichiometric Fe-48Al of 250 kJ/mol for Fe- and 249 kJ/mol for Al-diffusion using single phase diffusion couples. Additionally, the coarsening behavior of a fully lamellar FeAl/FeAl₂ material has been characterized with an activation energy of 265 kJ/mol [32]. Thus, while coarsening itself occurs during creep, it does not appear to be the rate determining factor. The additional stress that is applied in creep activates other processes which are responsible for creep deformation. In contrast to the above, reported values in the literature for activation energy for creep of B2-ordered Fe-(39-49)Al of 460 kJ/mol by Whittenberger [44] and 395 kJ/mol by Jimenez & Frommeyer [40] are significantly higher than the values for self-diffusion but no clear explanation has been provided for this remarkable difference. It is worth noting that the diffusion data were collected in the temperature range of 500 to 1200 °C whereas creep data were only generated between 600 to 800 °C and therefore it is not clear whether the degree of disorder and/or the types of complex aggregates of point defects may have influenced the activation energy. Thus, according to the phase diagram from Kubaschewski [45] a transition from $\alpha_2(l)$ to $\alpha_2(h)$ occurs at ~ 800 °C in stoichiometric B2, a phenomenon that was originally reported by Köster and Gödecke [46]. Other studies [47], [48] have examined the nature of point defects and their mobility in FeAl alloys and claim that for Al concentration greater than 35 at.% in FeAl,

triple defect aggregates have to be considered in the $\alpha_2(l)$ region whereas in the $\alpha_2(h)$ regime additional di-vacancies are formed.

The activation energy for creep of single-phase FeAl₂ as well as the activation energies for self-diffusion of Fe and Al in FeAl₂ are not readily available in literature. For this reason, the activation energy for creep of single phase FeAl₂ material was characterized in this investigation in the temperature range of 650 °C to 800 °C at a nominal stress level of 100 MPa and found to be 309 ± 83 kJ/mol (see Figure 9b). This value matches well the values obtained for the dual phase material, confirming that the creep behavior is primarily determined by the more creep resistant FeAl₂ phase.

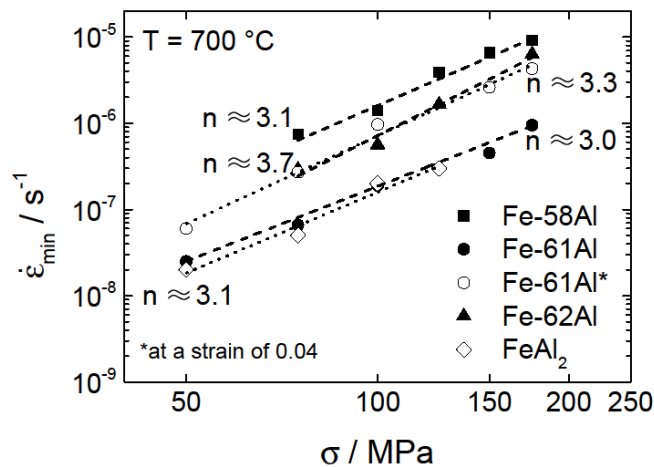


Figure 8: Stress dependence of the minimum strain rate at 700 °C for the three two-phase alloys and for single-phase FeAl₂. In addition, the stress dependence of the strain rate at a strain of 0.04 is also included for the Fe-61Al alloy (open circles). The stress exponent n is in the range 3 to 4 in all cases.

Returning to Figure 8, the minimum strain rate for different stress levels can be used to compare the creep behavior at 700 °C of the three Fe-Al alloys and the single-phase FeAl₂ material against each other. The stress exponent is in the range of 3 to 4 for all cases and indicates dislocation-mediated deformation. Observations that can be made from Figure 8 for all stress levels examined include: (i) the Fe-58Al alloy containing FeAl as the pro-eutectoid phase is the least

creep resistant, (ii) Fe-62Al containing FeAl₂ as the pro-eutectoid phase is better but not as good as the fully lamellar Fe-61Al alloy, and (iii) single phase FeAl₂ is superior in creep response (as measured using the minimum creep rate) relative to the off-eutectoid alloys but the response of the fully lamellar alloy virtually overlaps the behavior of single phase FeAl₂. By assuming that both phases are deforming when the minimum is reached, the fact that minimum creep rate of the fully lamellar and the single phase FeAl₂ match well under similar testing conditions and that the activation energy for creep for single phase FeAl₂ is in good agreement with that obtained for the two-phase fully lamellar microstructure, we conclude creep under these conditions is dominated by the FeAl₂ phase. With respect to activation energy for creep, it can be seen in Figure 9 that for all examined alloys at a stress of 100 MPa and in the temperature interval of 600 and 800 °C, the activation energies lie in a narrow range between 309 – 321 kJ/mol.

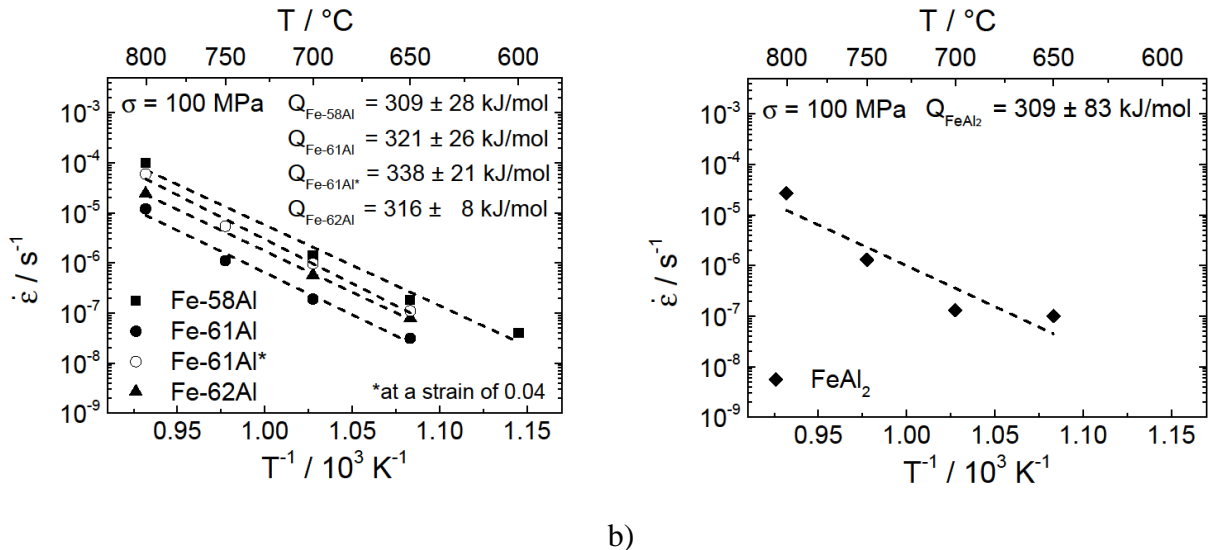


Figure 9: Arrhenius plot for the three alloys showing the variation in minimum strain rate with test temperature at a fixed stress level of 100 MPa. From these data, activation energy for creep was calculated. a) For all investigated two-phase alloys; the variation of strain rate at a strain of 0.04 with temperature for the eutectoid alloy is also included; b) for the FeAl₂ single phase.

4 Conclusions

The compressive creep behavior of a fully lamellar eutectoid Fe-61Al alloy as well as off-eutectoid Fe-58Al and Fe-62Al alloys that contain pro-eutectoid FeAl and FeAl₂, respectively, was examined at elevated temperatures. The main findings include:

- Creep rate as a function of time/strain exhibits a distinct minimum; a steady state creep regime does not occur.
- Lamellar coarsening observed during creep is primarily attributed to thermal exposure; the contribution from imposed mechanical loads is secondary. Lamellar coarsening within the colony only plays a minor role in affecting the creep response beyond the observed minimum of the creep rate.
- The morphological instability of the lamellar microstructure, particularly in the vicinity of colony boundaries during creep is identified as the main reason for the observed increase in strain rate beyond the minimum.
- The presence of a pro-eutectoid phase being FeAl or FeAl₂ leads to a deterioration in the creep resistance. By comparing the creep response of the FeAl/FeAl₂ lamellar eutectoid microstructure with that for single phase FeAl and FeAl₂, it is shown that the lamellar arrangement significantly enhances the creep resistance at least until the minimum creep rate is attained.
- The stress exponent at 700 °C for these two-phase alloys was determined to be 3 – 4 in all cases examined; while in a single-phase solid solution, this would signify creep controlled by dislocation glide and climb, a mechanism-based explanation for this observation in these multiphase lamellar alloys requires detailed microscopy analysis of the deformed structures and this work is in progress.
- The activation energy of 309 – 321 kJ/mol is higher than those reported for self-diffusion in FeAl but lower than the ones reported for creep of stoichiometric and near stoichiometric FeAl.
- The results from the Norton plot and the experimentally determined activation energy strongly indicate that beyond the strain rate minimum both phases deforms and contribute to the creep behavior but FeAl₂ is the rate controlling phase.

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