Evolution of microstructures during creep in TiAl-base intermetallics with a different Nb content

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Abstract. An evolution of microstructure has been investigated with increasing compression creep strain in intermetallic alloys Ti-48Al-2Cr-2Nb-1B (alloy Nb2) and Ti-46Al-7Nb-0.6Cr-0.2Ni-0.1Si (alloy Nb7) loaded to 350 MPa at 973 K. Scanning electron microscopy and transmission electron microscopy analysis focused on individual deformation modes and on the phase stability of the alloys. Dislocation densities and deformation twinning characteristics were systematically evaluated for strains up to 0.38. Results of these quantitative studies showed that the evolution of total and non-zero \(<c>-component dislocation densities with strain was similar for all the investigated materials in spite of relevant differences in the initial \(\gamma/\alpha_2\) microstructures. However, the spacing between deformation twins seemed to scale with the creep strength of the two alloys. These results are discussed in terms of the fundamental deformation modes and their contributions to the strain accumulation kinetics during high temperature creep in TiAl-base intermetallics with a different Nb content.

1. Introduction

TiAl-base intermetallic alloys of the 2nd and 3rd generation are attractive engineering materials for gas turbine and car engine applications [1, 2], but they also represent interesting systems for basic high temperature creep studies [3, 4]. A transition from 2nd to 3rd generation alloys was based on new alloying strategies, which promoted materials with higher Nb content [5]. While the positive influence of Nb on room temperature mechanical properties was soon appreciated [6, 7], the influence of Nb alloying on high temperature strength has not yet been fully understood [4, 8]. There seems to be a general agreement in that the additions of Nb exceeding 5 at% cause an increase of creep strength. Controversies still exist regarding the nature of the strengthening mechanism [4]. Since the solid solution contribution is less important in the creep regime [9], the strengthening effect due to Nb additions was suggested to reflect either a decrease of the stacking fault energy or a limited diffusivity of Nb atoms and/or better thermal stability of \(\gamma/\gamma\) and \(\gamma/\alpha_2\) lamellar microstructures [4]. However, the investigations performed so far did not address the evolution of deformation structures with strain in terms of dislocation densities and deformation twinning characteristics and their potential dependence on the Nb content. Therefore, in the present study we focus on two alloys with different Nb content (Ti-48Al-2Cr-2Nb-B and Ti-46Al-7Nb-0.6Cr-0.2Ni-0.1Si) and investigate their high temperature creep and associated microstructural changes in detail. The quantitative assessment of dislocation structures is based on a transmission electron microscopy (TEM) technique in which dislocations with non-zero component of the Burgers vector into the \(<c>-axis of the \(L_1_0\) lattice (superdislocations and twinning dislocations) are systematically displayed with a reflection condition \(g = (002)\) [10 – 13].
2. Materials and experiments

The Ti-48Al-2Cr-2Nb-1B (at%) alloy (Nb2) was plasma melted at the IRC Birmingham. The alloy was isothermally forged at 1423 K and a strain rate of $5 \times 10^{-3}$ s$^{-1}$ with a height reduction of 70%. Parts of the resulting pancake received three different heat treatments in order to generate different prior-to-creep microstructures: (i) annealing at 1473 K for 4 hours followed by furnace cooling – the resulting microstructure was equiaxed with a low $\alpha_2$ volume fraction of 1.2% (ELA), (ii) annealing at 1658 K for 1 hour followed by furnace cooling – the resulting microstructure was nearly lamellar (NL) with 4.8 vol.% of $\alpha_2$ and (iii) after the heat treatment under (i), the alloy was additionally annealed at 1523 K for 1 hour which resulted in Widmanstätten-like distributed $\alpha_2$ precipitates that occupied high volume fraction of 17.9 % in equiaxed $\gamma$ grains (alloy version EHA). Further details on the heat treatments and prior-to-creep microstructures of the Nb2 alloy were published elsewhere [10].

The Ti-46Al-7Nb-0.6Cr-0.2Ni-0.1Si (at%) alloy (Nb7) was melted, cast and delivered by Flowserve Dayton in a form of a cylindrical rod the length and diameter of which were 1200 mm and 70 mm, respectively. The rod was HIPped after casting and no additional heat treatment was applied. The volume fraction of the $\alpha_2$ phase prior-to-creep was close to 5%. Further information on the processing of the Nb7 alloy can be found in [11].
Compression creep tests were performed in purified argon at 973 K and under the constant applied stress of 350 MPa. The tests were interrupted at pre-selected strains and the corresponding microstructures were investigated. Pre-deformed compression cylinders were cut axially; one surface of the axial cut was electrolytically polished and observed by scanning electron microscopy (SEM). As an example, the SEM image in Figure 1 was taken using back scattered electrons (BSE) and documents the prior-to-creep microstructure of the alloy Nb7, which consists of equiaxed $\gamma$ (L1$_0$ crystal lattice) and $\alpha_2$ (D0$_{19}$ crystal lattice) grains surrounded by $\gamma/\gamma$ and $\gamma/\alpha_2$ lamellar colonies. Thin foils cut from the other half of the pre-deformed cylinders were investigated using (scanning) transmission electron microscopy ((S)TEM). Further details on the creep testing and the quantitative assessment of microstructures were published elsewhere [3, 11, 12].

3. Results
A stress of 350 MPa was applied to the compression cylinders at 973 K. The corresponding creep data are plotted in Figure 2. Creep curves obtained for the alloy Nb2 (three microstructural states ELA, NL and EHA) and the alloy Nb7 (as-cast and HIPped state) are compared in Figure 2a. While the creep

Figure 3. STEM HAADF micrographs of the alloy Nb7 illustrating the microstructural evolution with increasing compression creep strain at 973 K and 350 MPa: (a) $\varepsilon = 0$ (prior-to-creep), (b) $\varepsilon = 0.027$ (at the minimum creep rate) and (c) $\varepsilon = 0.380$ (at the terminal compression strain).
rates of the alloy Nb2 tested in versions NL and EHA are rather similar throughout the investigated strain range, the microstructural version ELA creeps clearly slower. The alloy Nb7 then exhibits even smaller creep rates at all strains. Figure 2b summarizes the interrupted creep experiments performed for the alloy Nb7 and indicates the creep strain levels at which the individual tests were terminated (0.6, 2.7, 5.1 and 38%) in order to analyse corresponding deformation structures.

Examples of high angular annular dark field (HAADF) STEM images are presented in Figures 3a (prior-to-creep microstructure of the alloy Nb7), 3b (after 2.7% of compression strain) and 3c (at the terminal compression strain 38%). The STEM images in Figs. 3a and b clearly show that, while the total dislocation density in the γ-phase increases considerably, the γ/γ and γ/α2 lamellar interfaces are quite stable and the corresponding interlamellar spacing does not change markedly during primary creep. The total dislocation density (Figure 4a) and the density of dislocations with non-zero <c>-component of the Burgers vector (Figure 4b) seem to evolve similarly with increasing compression creep strain in all investigated material states in spite of relevant differences in their γ/α2 prior-to-creep microstructures. On the other hand, the distance λ between deformation twins is generally higher for the ELA version of the alloy Nb2 and for the alloy Nb7, as documented in Figure 4c. Since the spacing λ characterizes the intensity of twinning in the individual pre-strained materials, the plots in Figure 4c suggest that the NL and EHA versions of the alloy Nb2 exhibit correspondingly higher twinning activity throughout the investigated range of strain.

![Figure 4](image_url)

**Figure 4.** Characteristics of the microstructure after creep in the alloys Nb2 and Nb7 and their evolution with compression creep strain: (a) overall dislocation density, (b) density of dislocations with non-zero <c>-component of Burgers vector and (c) spacing between deformation twins.
4. Discussion

The creep curves presented in Figure 2a show that the creep strain accumulation kinetics is similar for microstructural modifications EHA and ELA of the alloy Nb2 and for the alloy Nb7. In all the three cases, the creep rate decreases by several orders of magnitude in a rather narrow strain range before a minimum creep rate is attained at creep strains of about 3% or less. During this primary stage the total and \(<c>\)-component dislocation densities grow by at least one order of magnitude, see Figures 4a and 4b. We note that the \(<c>\)-component dislocation density represents approximately one fourth of the total density at comparable strains. On further straining, the hardening-type kinetics changes to an accelerating stage during which creep rate increases and seems to seek for a new steady value, see the curves recorded for material states Nb2-EHA, Nb2-ELA and Nb7. Thus the alloys with considerably different prior-to-creep \(\gamma/\alpha_2\) microstructures follow the similar type of accelerating stage. Moreover, corresponding \(\gamma/\alpha_2\) microstructures exhibited no significant changes up to strains well behind the range where the creep rate reaches a minimum. Therefore, we conclude that expected microstructural instabilities like coarsening of \(\gamma\) and \(\alpha_2\) lamellae [4] cannot account for the creep behaviour observed in the present study. Furthermore, similar acceleration stages have also been observed during creep of Al-rich binary alloys that fully consisted of equiaxed \(\gamma\) grains and did not contain any \(\alpha_2\) phase either in the form of lamellae or particles [12]. In the present study, creep experiments were performed at a relatively low temperature of 973 K and, while some recrystallization was detected in the Nb7 specimen subjected to the pre-deformation of 38%, no evidence of recrystallization was found for the other Nb7 microstructures pre-deformed to lower strain levels. These results combined with a fact that strains, at which creep rates exhibit a minimum, are small (between 2 and 3%) strongly support the conclusion that the dynamic recrystallization can be excluded as a reason of the softening during the acceleration stages.

In our earlier studies [11, 12] we have shown that the creep strain accumulation kinetics of the type shown in Figure 2 for the Nb2 alloy modifications EHA and ELA and for the alloy Nb7 can be reasonably well accounted for when combinations of individual deformation modes operating in the \(L_{10}\) \(\gamma\)-TiAl lattice are taken into account. Mecking and co-workers [13] pointed out that a proper partitioning of the overall strain accumulated in \(\gamma\)-TiAl intermetallics among contributions due to ordinary dislocations, superdislocations and deformation twinning is necessary to fulfil the von Mises criterion. This principle is still important in the creep regime in order to avoid excessive internal stress concentrations. As the critical resolved shear stress for the ordinary slip is small relative to the other two deformation modes, a stress redistribution during primary creep is needed in a majority of cases to promote a sufficient activity of deformation twinning and to activate sources of \(<c>\)-component dislocations [11]. Figure 4c shows that a clear difference in the twinning activity exists when the NL modification of the alloy Nb2 is compared to the modification Nb2-ELA or the alloy Nb7. As a consequence, the NL modification of the alloy Nb2 exhibits a different type of creep strain accumulation kinetics (Figure 2a) and the last two material states (Nb2-ELA and Nb7) creep at low creep rates. A contribution to the superior creep strength of the alloy Nb7 may also be due to a drop of diffusion coefficients associated with the Nb alloying [4]. The low twinning activity in the alloy Nb7 is rather unexpected based on the low stacking fault energy argument [4] and needs a further investigation.

5. Conclusions

Creep strength of the TiAl base alloys Nb2 and Nb7 tested at 973 K and 350 MPa increases in a sequence Nb2 - NL, Nb2 - EHA, Nb2 - ELA and Nb7 - as cast. The evolution of the total and \(<c>\)-dislocation densities with increasing compression creep strain is similar for all the four investigated material states. Stronger alloy versions exhibit less twinning activity and may thus carry higher internal stresses due to the incompatibility of the plastic strain. Results of this study suggest that neither the overall content of the \(\alpha_2\) phase nor its evolution with the increasing creep strain could account for the observed variations of the creep strength.
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