Abstract—The creep deformation behaviour and creep microstructures of a dual phase TiAl/Ti₃Al alloy Ti₄₈Al₂Cr (at. %) with a fully transformed lamellar structure have been investigated at 800°C in tension creep. The creep curves show no steady state creep rate, rather a minimum creep rate was obtained followed by a steep increase in the creep rate. The application of a power-law creep equation of the type \( \dot{\epsilon} = A\sigma^n \) leads to a stress exponent \( n = 7.6 \). The deformation microstructures show no deformation twins and essentially consist of \( \frac{1}{2}[110] \) dislocations; recovered structures were observed in the fine \( \gamma \)-grains at colony boundaries whereas long and curved \( \frac{1}{2}[110] \) segments were found in the \( \gamma \)-lamellae. These microstructural observations suggest a dislocation controlled creep; it ensues that the stress exponent obtained using power-law creep does not reflect the operating creep mechanism. This is attributed to early nucleation and growth of voids and cavities at colony boundaries which do not allow steady-state creep to be established, an underlying condition for the applicability of the power-law equation above. The nucleation and growth of void and cavities are thought to arise as a result of stress concentration at colony boundaries which in turn results from the dependence of deformation behaviour on the orientation of the lamellar colonies with respect to the tensile axis. The loss of creep strength is found to be primarily due to growth and coalescence of these voids and cavities.

Résumé—Le comportement de déformation par fluage d’un alliage biphase d’aluminiures de titane TiAl/Ti₃Al ayant pour composition Ti₄₈Al₂Cr et une structure lamellaire complètement transformée a été étudié en traction à 800°C. Nous présentons de même les microstructures associées à ce type de déformation. Les courbes de fluage montrent un régime transitoire assez marqué, suivi d’un minimum de vitesse de fluage avant l’établissement d’un régime tertiaire où une augmentation rapide de la vitesse de fluage se produit. Nous n’avons pas observé de régime permanent. L’application d’une loi de puissance de type \( \dot{\epsilon} = A\sigma^n \) conduit à un exposant de contrainte \( n = 7.6 \). Les microstructures de fluage ne montrent pas de macles de déformation et comprennent des dislocations de vecteur de Burger \( \frac{1}{2}[110] \); des structures de restauration ont été observées dans des fins grains equiaxes de \( \gamma \)-TiAl présent les joints de grains des colonies lamellaires; les lamelles de \( \gamma \)-TiAl comportent de longs segments de dislocations \( \frac{1}{2}[110] \) mixtes. Ces observations montrent que le fluage est contrôlé par les mouvements des dislocations; il s’en suit que l’exposant de contrainte trouvé en utilisant la loi de puissance ci-dessus ne rend pas compte du mécanisme de fluage. Ceci est attribué à la nucléation et à la croissance prétexturées de pores et de cavités aux joints de grains des colonies lamellaires, ce qui ne permet pas l’établissement d’un régime permanent, qui est une condition de base pour l’application de la loi ci-dessus. Nous pensons que la nucléation et la croissance de ces pores et cavités se produisent en raison de l’accumulation de contraintes aux joints de grains des colonies lamellaires; ces contraintes elles mêmes parviennent du fait que la déformation des colonies lamellaires soit une fonction de leur orientation par rapport à l’axe de traction. La détérioration de la résistance au fluage est due essentiellement à la croissance et à la coalescence de ces pores et cavités.

Zusammenfassung—Das Kriechverformungsverhalten einer zweiphasigen TiAl/Ti₃Al Legierung Ti₄₈Al₂Cr (in at. %) im volltransformierten lamellaren Zustand wurde im Zugversuch bei 800°C untersucht. Eine mikroskopische Untersuchung der Mikrostrukturen vor und nach den Kriechversuchen wurde ebenso durchgeführt. Die Kriechkurven zeigen einen ausgeprägten Primärkriechbereich; darauf folgt ein Bereich minimaler Kriechrate bevor tertiäres Kriechen einsetzt. Stationäres Kriechen konnte nicht beobachtet. Die Beschreibung der Ergebnisse durch ein Potenzgesetz vom Typ \( \dot{\epsilon} = A\sigma^n \) ergibt einen Spannungsexponenten \( n = 7.6 \). Die nach dem Kriechvorgang untersuchten Mikrostrukturen zeigen keine Verformungszwillinge; sie bestehen im wesentlichen aus einfachen Versetzungen \( \frac{1}{2}[110] \). In den feinen \( \gamma \)-TiAl Körnern an Lamellenkoloniegrenzen deuten sich Erholungsstrukturen an; in den \( \gamma \)-Lamellen wurden längere gekrümmte \( \frac{1}{2}[110] \) Versetzungssegmente beobachtet. Diese mikrostrukturellen Ergebnisse weisen auf einen Versetzungskriechmechanismus hin und zeigen, daß der oben ermittelte Spannungsexponent den Kriechmechanismus nicht widerspiegelt. Der hohe Spannungsexponent folgt einem Versetzungskriechmechanismus. Die in früheren Kriechstadien einsetzende Keimbildung und Wachstum von Poren und Hohlräumen an den Grenzen der Lamellenkolonien das Einsetzen des stationären Kriechens, eine Voraussetzung für die Anwendung des Potenzgesetzes, verhindern. Solche Poren und Hohlräume entstehen infolge der Bildung
1. INTRODUCTION

Among low-density intermetallic alloys under development for high temperature structural applications, γ-TiAl-base alloys have been, and continue to be, of major fundamental and engineering interest. Because of the brittleness of these alloys at temperatures below ~700°C, much of the research activity has focused essentially on the optimization of room temperature ductility and fracture toughness via suitable alloying additions and microstructure control, e.g. see Refs [1–3].

TiAl-base alloys of engineering interest have Al contents in the range 47–48 at.% and contain ternary/quaternary alloying additions of Cr, Nb, . . . , etc. [1–3]; they are based on a dual phase microstructure consisting of a TiAl matrix and small amounts (~8%) of the ordered (D0₁₉) h.c.p. Ti₃Al phase. The properties of these alloys have been shown to strongly depend on processed microstructures which can be controlled by suitable sequences of thermomechanical treatment and subsequent heat treatment in the appropriate phase field (e.g. see Ref. [1]).

While satisfactory room temperature tensile ductilities could be achieved via alloy optimization and microstructure control, the high temperature properties, in particular creep strength, are still unsatisfactory for these alloys to be considered for high temperature structural applications, e.g. Ref. [3]. Surprisingly, the issue of creep in near γ-TiAl base alloys has been addressed only marginally and, in contrast to the profuse literature on microstructure and mechanical properties of these alloys a very limited number of publications [3–11] were concerned with the creep properties of these alloys and studies of creep microstructures and mechanisms are still lacking.

In recent work [12, 13], we reported the creep deformation properties and the microstructures associated with creep of a near γ-TiAl alloy Ti–48 at.% Al–2 at.% Cr in the cast and hot isostatically pressed condition with a microstructure consisting of large lamellar colonies and equiaxed single phase γ-TiAl grains at the colony boundaries. In contrast to other reported work [4–6], no steady state creep, rather a minimum creep rate followed by a steep increase in the creep rate, was observed, due to the early onset of microstructural instabilities in the single phase γ-grains [13]. Assuming a power-law dependence of the minimum creep rate on stress, a stress exponent of 7.6 was obtained, which did not agree with the microstructural observations which, unambiguously, supported a dislocation creep mechanism [13]. These results, together with those compiled from the literature, were discussed in terms of the inadequacy of the power-law formalism to interpret the creep data of these alloys, since the constancy of the deformation microstructure, which underlies this formalism, was not satisfied.

In order to investigate the effects of heat treatment and microstructure on the creep deformation behaviour of the near γ-TiAl alloy Ti–48 at.% Al–2 at.% Cr, specimens were homogenized in the α phase field at 1400°C; the microstructure thus obtained consists of large lamellar colony grains, and is devoid of equiaxed γ-TiAl grains. In the present work, the results of this investigation are reported and discussed in comparison to previous work.

2. EXPERIMENTAL PROCEDURES

The Ti–48Al–2Cr (at.%) alloy was produced by Böhler Corporation, Kapfenberg (Austria) via ingot casting and subsequent vacuum arc remelting. The main impurities were O: ~600 wt ppm, N: 100 wt ppm and C: ~110 wt ppm. The ingots were then hot isostatically pressed at 1185°C and 150 MPa to achieve full compaction. In order to produce a fully transformed lamellar structure, specimens were homogenized in the disordered α phase field at 1400°C for 2 h in vacuum and slowly cooled to 1000°C for an aging treatment of 4 h. Cylindrical tensile specimens with gauge sections of 4 mm dia. × 20 m length were prepared by spark erosion. The specimens were mechanically ground and subsequently electrolytically polished before testing to eliminate surface damage. The creep tests were conducted in air at 800°C under constant load in the stress range from 150 to 260 MPa. The creep strains were monitored using an inductive extensometer.

Polarized light microscopy (LM), scanning electron microscopy (SEM) using the back-scattered (BS) electron imaging mode and transmission electron microscopy (TEM) were used to investigate creep microstructures. Thin foil specimens for TEM studies were thinned electrolytically in a twin-jet polishing machine using a 5% perchloric acid solution in methanol at −50°C with V = 15 V and I = 10 mA.

3. RESULTS

3.1. Starting microstructure

Figure 1 shows a polarized light micrograph (a) and a BS-SEM micrograph (b) of the starting microstructure which consists of coarse lamellar grains; colonies of coarse and interwoven lamellae were frequently observed and are shown in Fig. 1(a). The coarse single phase γ grains observed in the HIP
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Ti₃Al phases. This is illustrated in Fig. 1(b), where the α₃ laths are bright in contrast (because of their higher Ti content). Such a lamellar structure results from the solid state phase transformation of the primary disordered α grains. The fine γ grains surrounding the lamellar colonies are proeutectoid γ-TiAl which form at α grain boundaries during cooling. (See [1, 2] for a review of phase transformations, microstructural development and phase relationships in TiAl-base alloys.)

TEM investigations of the starting dislocation substructures essentially show sessile configurations of \( \frac{1}{2} <112> \) and \( \frac{1}{2} <110> \) dislocations, Fig. 2. These configurations are similar to the sessile "tree-like" structures described by Greenberg et al. [14] in single phase γ grains deformed at 400°C; they were observed in all grains and lamellae investigated here, and are

Fig. 1. (a) Polarized light micrograph of the starting microstructure. Arrows indicate fine γ grains at colony boundaries. (b) SEM-BS micrograph of the starting microstructure. Notice the very fine interlamellar spacing, and the existence of fine γ grains at colony boundaries (arrows).

structure [12, 13] were eliminated by the homogenization treatment; only very fine γ grains at colony boundaries were observed. The lamellar structure consists of alternating laths of the γ-TiAl and α₃-

Fig. 2. Bright field (BF) micrographs of the starting dislocation structure under two different diffraction conditions: (a) \( g = 022 \), the beam direction \( B \) is close to [111]; (b): \( g = 220, B = [111] \). The segments S1 and S2 are \( \frac{1}{2} <112> \) superdislocations; the segments designed P are [110] dislocations.
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analogous with the starting dislocation structures observed in the hot isostatically pressed condition [13]. Such configurations are thought to arise as a result of phase transformations and disregistry of interphase and domain boundaries [15]; they may act as obstacles to moving dislocations, owing to their sessile character [13].

3.2. Creep behaviour

The evolution of the creep strain rate $\dot{\varepsilon}$ vs strain $\varepsilon$ for different initial stresses is shown in Fig. 3. The creep test at 150 MPa was interrupted at a creep strain of $\varepsilon = 0.03$ to allow microstructural investigations near the minimum creep regime to be performed. As can be seen, the creep curves exhibit a well pronounced primary creep regime followed by a minimum creep range. The strain rate then increases slightly but continuously with strain up to $\varepsilon = 0.05$ before catastrophic acceleration occurs followed by creep failure. In all the specimens tested necking was not observed which suggests that the increase in the creep strain rate is due to microstructural instabilities. This behaviour does not reflect a steady-state creep and contrasts with the results of Hayes and London [4], and Huang and Kim [6], who report the existence of a steady-state regime, and indicates that, under the conditions of stress and temperature investigated here, microstructural instabilities become dominant in controlling the creep rate at the end of the primary creep regime.

A similar creep behaviour and creep strain rates have been reported for the hot isostatically pressed (HIP) condition under similar stress and temperature conditions [13]. However, the creep fracture strains, which range from 0.05 to 0.08, Fig. 3(a), are quite lower than those of the HIP specimens, where creep fracture strains up to 0.20 have been found [13].

The stress dependence of the minimum creep rate is usually described by a power law according to $\dot{\varepsilon}_m = A\sigma^n$, where $A$ is a constant depending on temperature, $\sigma$ the applied stress and $n$ the stress exponent. This equation yields a linear relation between $\sigma$ and $\dot{\varepsilon}_m$ in a double logarithmic plot, as demonstrated in Fig 3(b). The slope of this curve yields a stress exponent of $n = 7.6$. This value equals that reported for the HIP condition [13], and is close to the values reported by Takahashi and Oikawa [7] on a Ti-50 at.% Al alloy with a near lamellar microstructure, and by Wheeler et al. [8] on a Ti-48Al-2Cr-2Nb (in at.%) alloy with a duplex microstructure. (See Ref. [13] for a compilation of the creep data of γ-TiAl base alloys.) Such a value is quite high in comparison to the values usually found in f.c.c. metals and alloys for dislocation creep, e.g. in the range 3-5 [16], and does not agree with the microstructures observed in creep specimens presented below.

3.3. Microstructures associated with creep

3.3.1. Minimum creep regime. The creep microstructures of the specimen deformed at an initial stress of 150 MPa to a creep strain of $\varepsilon = 0.03$, corresponding to the minimum creep regime, Fig. 3(a), are shown in Fig. 4(a) which is a low magnification micrograph of the deformed specimen. Deformation twinning across the lamellae was rarely observed, in contrast to the HIP condition where this deformation mode substantially contributes to the overall deformation process [13]. The absence of twinning across the lamellae was...
Fig. 4. Caption on facing page.
also reported by Huang and Kim [6] in specimens with a fully transformed lamellar structure crept at 900°C. It seems that favorable nucleation sites for mechanical twins have been suppressed, or at least substantially reduced, by the homogenization treatment. Since such a treatment results in the elimination of interdendritic Al-rich γ-TiAl, it can be speculated that these two phenomena might be closely dependent. As described in [13], interdendritic γ-TiAl grains have been found to be the loci of high deformation stresses which probably induce deformation twinning in the lamellar structure, to relieve stress and maintain strain continuity. More studies are, however, needed in order to elucidate the absence of twinning deformation, which is an important deformation mode in γ-base alloys, e.g. [1, 2], during creep of fully transformed lamellar structures.

Figure 4(b, c) show an example of the dislocation structure obtained in the single phase γ grain of Fig. 4(a), these grains contain a high dislocation density, and evidence of recovery can be seen, Fig. 4(b). As deduced from diffraction contrast analysis using the \( \mathbf{g} \cdot \mathbf{b} = 0 \) criterion for the invisibility of dislocations, where \( \mathbf{g} \) is the reciprocal lattice vector and \( \mathbf{b} \) the Burgers vector, the dislocations are of the type \( \frac{1}{2}[100] \), \( [110] \) perfect dislocations and \( [010] \) superdislocations. With respect to the latter, it is not clear, at this stage, whether they belong to the starting microstructure or they contribute to the deformation process. The deformation structure of an adjacent grain is shown in Fig. 4(e, f). The dislocations are of the type \( \frac{1}{2}[110] \) and are lying on two different \( \{111\} \) planes; superdislocations were not observed in this grain. Figure 4(f) suggests a pinned morphology of the \( \frac{1}{2}[110] \) dislocations, which is probably due to intersection events between these dislocations and the \( \frac{1}{2}[101] \) dislocations. Such events are thought to control the creep strain rate in the primary creep regime. Furthermore, the high number of dislocation loops present in both grains, which suggests many dislocation interactions either between perfect dislocations gliding on intersecting \( \{111\} \) planes and/or as perfect dislocations cut through superdislocations, seems to support this hypothesis.

The dislocation structures of the γ lamellae show long and curved segments of perfect \( \frac{1}{2}[101] \) dislocations and, occasionally, superdislocations, Fig. 5(a, b). The overall dislocation density in the lamellar structure has been found to be low as compared to that reported in the HIP condition [13], and no recovery structures, e.g. subgrain formation, were observed.

3.3.2. Tertiary creep regime. The tertiary creep regime is characterized by a rapid increase in the creep strain rate as a result of microscopic instabilities. Figure 6(a, b) are respectively a polarized light micrograph and a BS-SEM micrograph of the specimen deformed to fracture at an initial stress of 260 MPa; the micrographs were taken just beneath the fracture surface. Cracks along colony boundaries can easily be seen; they are thought to result from cavity nucleation, growth and coalescence at colony boundaries where strain incompatibilities are high (see below for discussion). As observed by light and scanning microscopy, the existence of these cracks constitutes the main difference between crept and starting microstructures.

In contrast to the HIP condition [13], the homogenized specimens show neither dynamic recrystallization nor spheroidization of the \( \alpha_2 \)-laths in the stress range investigated. TEM observations of the same specimen reveals subgrain formation in the fine single phase γ grains, Fig. 7(a). The γ-lamellae contain a higher dislocation density in comparison to the minimum creep regime, Fig. 7(b). However, no change in the deformation mode was observed, e.g. deformation twins were rarely found. Again no dynamically recrystallized grains, which could have
been unresolved in the BS-SEM micrographs, were observed.

Therefore, we conclude that tertiary creep in the homogenized specimens is primarily controlled by cavity nucleation, growth and coalescence at colony boundaries.

Fig. 6. LM (a) and SEM-BS (b) micrographs of a homogenized specimen deformed to fracture; initial stress: 260 MPa; \( \varepsilon_f = 0.05 \). Notice cracks (a), and cavities (b) at colony boundaries (arrows).

3.3.3. Creep fracture. The fracture surface of the specimen deformed to fracture at an initial stress of 260 MPa is shown in Fig. 8. The intergranular, brittle character of creep fracture can easily be seen. The cavities and cracks at colony boundaries, e.g. Fig. 6(b), are thought to grow catastrophically during the final stage of tertiary creep thus leading to creep failure along these boundaries. A similar fracture mode has also been reported for lamellar structures during tensile testing at 900°C [2].

4. DISCUSSION

The results reported in this work suggest a quite complex creep deformation behaviour of the microstructure investigated. In the following, these results will be qualitatively discussed.
The well pronounced primary creep regime, which may be regarded as a serious drawback to technological application of these alloys, is probably due to the abundance of interfaces, and to the relatively high density of dislocations in the starting microstructure, which may serve as dislocation sources. Microstructural observations suggest that the creep rate at this stage is controlled by glide of $\{110\}$ dislocations, and by dislocation interactions, e.g. intersections of $\{110\}$ dislocations gliding on different $\{111\}$ planes, e.g. Fig. 4. Microstructure evolution leads to the formation of recovered structures in the fine $\gamma$ grains present at colony boundaries, and to the nucleation of voids and cavities as a result of stress concentration at these boundaries. The accumulation of stress at colony boundaries may arise as a result of the strong dependence of the yield stress on the orientation of the lamellae with respect to the tensile axis. As recent work [17] suggests, such a dependence gives rise to two deformation modes: an easy deformation mode, characterized by low flow stresses and high ductilities, "when the lamellar boundaries are oriented within $30^\circ$-70° from the tensile axis" where slip proceeds parallel to the lamellar boundaries, and a hard deformation mode, characterized by high flow stresses and low ductilities, where shear deformation has to occur across the lamellar boundaries. Stress buildup at colony boundaries has been reported to be responsible for brittle fracture during tensile testing of fully transformed structures with coarse and randomly oriented lamellar grains at room temperature and 900°C [2], where crack initiation has been found to follow some suitably oriented lamellar boundaries. The picture is quite similar during creep testing. The high stresses, which develop at colony boundaries, are accommodated by cavity nucleation and growth probably early in the creep life. In the case of the HIP condition, such stresses have been reported to be accommodated by plastic deformation and dynamic recrystallization of the equiaxed $\gamma$ grains surrounding the lamellar colonies [13]. On the basis of these observations, we surmise that the growth rate of voids at colony boundaries should at a certain time, depending on stress, balance the primary creep rate thus giving rise to a minimum creep rate, beyond which the creep rate is governed by void growth and coalescence. It becomes clear that, under these conditions of microstructural instabilities, the application of a power-law creep of the form $\dot{\varepsilon}_0 = A_0 \sigma^n \exp(-Q/RT)$, where $A_0$ is a material constant, $\sigma$ the applied stress, $Q$ the activation energy of the creep process, $R$ the gas constant and $T$ the absolute temperature, to describe the creep behaviour of the present alloy microstructure yields an apparently higher stress exponent. Indeed, this equation is only valid in the case of steady-state creep, where the rate of dislocation generation, i.e. hardening, is assumed to be balanced by the rate of dislocation annihilation, i.e. recovery. In this case, the dislocation configuration and density are assumed to be constant, and are included in the pre-exponential factor together with other structural parameters such as stacking fault energy, grain size, type and dispersion of second phase precipitates, etc., all supposed to be constant. In the case where these conditions are satisfied, a stress exponent in the range 4–5 and a $Q$ close to the activation energy of self-diffusion should be expected [16]. In our case, these constancy of $A_0$ is certainly not satisfied, as ensues from the above discussion and the microstructural investigations presented in the preceding section, which might explain the higher stress exponent obtained.

The stress exponent of 7.6 obtained in this work is quite similar to the stress exponents obtained by Takahashi and Oikawa [7], and Wheeler et al. [8] under similar conditions of stress and temperature. A stress exponent of 7.7 has also been obtained by Oikawa [9] on equiaxed $\gamma$-TiAl grains. Takahashi and Oikawa [7], and Oikawa [9] did not observe steady-state creep in their alloys, whereas Wheeler et al. did. However, in the absence of detailed microstructural studies, we can only speculate that microstructural instabilities, e.g. dynamic recrystallization, might have been affecting their systems thus giving rise to such high stress exponents. Dynamic recrystallization also takes place during creep testing of equiaxed $\gamma$-TiAl microstructures [10] which might also explain the stress exponent of 7.6 obtained by Oikawa [9]. However, values of $n \approx 4.5$ and $Q \approx 300$ kJ/g-atom (close the activation energy for self diffusion of Ti...
in single phase γ-TiAl, i.e. 290 kF/g-atom, recently reported [18] have been reported [4, 5] on lamellar structures with equiaxed γ-grains at colony boundaries, and would suggest a dislocation climb controlled mechanism and a true steady-state creep regime. Unfortunately, the absence of previous microstructural investigations does not allow an insight in the causes of these discrepancies. Nevertheless, such discrepancies attest to the complexity of the creep behaviour of the different microstructures of γ-TiAl base alloys; more studies, both macroscopic and microscopic, are thus needed in order to gain some insight in to the creep mechanisms in these alloys.

5. CONCLUDING REMARKS

The creep deformation and the microstructures associated with creep at 800°C of a dual-phase TiAl/Ti₃Al alloy Ti-48Al-2Cr (at.%) in the homogenized (fully transformed) condition have been presented. The creep behaviour is characterized by a pronounced primary creep regime followed by a minimum creep range before the onset of tertiary creep, and is similar to that of the cast and hot isostatically pressed (HIP) condition presented in previous work [13]. The minimum creep rates as a function of the applied stress are also close to that of the HIP condition, and the equal stress exponent of 7.6 was obtained. These results indicate that creep deformation in both microstructures is controlled by similar mechanisms. The deformation microstructures corresponding to the minimum creep regime consist essentially of ½ <110> dislocations; deformation twins were rarely observed. Therefore, a stress exponent of ~4.5 would have reflected more correctly the creep mechanism. It is thought that the early formation of voids and cavities at colony boundaries, as a result of stress concentration at these boundaries, leads to the absence of steady-state creep. The application of a power-law equation of the type \( \epsilon \) = \( A \sigma^m \) to interpret the reported yield stress points an apparently higher stress exponent, since the condition of constancy of microstructure, which underlies this equation, is not satisfied. Similar conclusions have been reported for the HIP condition, where the absence of steady-state creep was attributed to dynamic recrystallization of the equiaxed γ-grains surrounding the lamellar colonies. However, as it appears from a literature survey, the creep behaviour of γ-base alloys is quite complex, and discrepancies in reported results are common. More studies, both macroscopic and microscopic, under well controlled conditions, are needed for an adequate interpretation of the creep behaviour of the complex microstructures, which characterizes the γ-base alloys, to be approached.

REFERENCES