Diffusion creep of intermetallic TiAl alloys

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Abstract
The creep behaviour of γ-TiAl with L1₀ structure without second phases, γ-TiAl with precipitated particles of α₂-Ti₃Al with D₀₁₉ structure, and γ-TiAl with the H-phase Ti₂AlC has been studied at low stresses in the temperature range 900–1200°C. The obtained data allow the construction of creep deformation mechanism maps for the studied alloys which may be used for an extrapolation of the observed creep behaviour. At higher stresses dislocation creep occurs in all alloys, which is well described by the Dorn equation with stress exponents in the range 3–5. Extended Coble creep with threshold stress was observed only for the studied two-phase alloys. A strong temperature dependence of the threshold stress for Coble creep was found for the TiAl alloy with carbide particles.

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Keywords: Titanium aluminides; Based on TiAl; Multiphase intermetallics; Creep (properties and mechanisms); Diffusion

1. Introduction
Developments of new structural materials on the basis of intermetallic compounds have reached a high technological importance. Various intermetallic materials show advantageous creep and oxidation properties, which makes them candidates for high-temperature applications. However, there is still a lack of experimental data with respect to the long-term creep behaviour at high temperatures and low stresses with strains of the order of only 1% within several years, when diffusion creep is expected to be the rate-controlling deformation-mechanism.

In the present work the long-term creep behaviour of γ-TiAl with L1₀ structure has been studied. The low density, the high-temperature strength and the good oxidation resistance up to 800°C are the reasons for the high interest in γ-TiAl, which is considered in particular for applications as structural components in gas turbines [1]. The creep behaviour of γ-TiAl and γ-TiAl alloys at high temperatures was studied repeatedly – see e.g. Refs. [2–6]. However, in those studies the creep deformation occurred primarily by glide or climb of dislocations, and in some cases creep was coupled with dynamic recrystallisation. Only in few cases diffusion creep was observed in a small stress and temperature range – see Ref. [7].

Diffusion creep, i.e. the plastic deformation by stress-directed flow of vacancies, was first analysed theoretically by Nabarro [8] and Herring [9]. In tensile experiments, vacancies are created by the applied stress at grain boundaries perpendicular to the stress axis, they migrate under the action of the stress through the lattice and are absorbed at boundaries parallel to the stress axis. The resulting creep rate \( \dot{\varepsilon} \) is a linear function of the applied stress \( \sigma \) and follows the equation [9,10]

\[
\dot{\varepsilon} = A_{NH} \sigma \Omega D_L / d^2 k T
\]  

\( A_{NH} \): dimensionless factor which considers the grain geometry and is of the order of 10, \( \Omega \): atomic volume, \( D_L \): coefficient of lattice self-diffusion, \( d \): grain size, \( k \): Boltzmann’s constant, \( T \): temperature). This Nabarro–Herring creep only occurs at temperatures near the melting point. At lower temperatures, another diffusion creep mechanism is to be considered which was first described theoretically by Coble [11]. This diffusion creep process relies on the diffusional flow of vacancies along grain boundaries and is described by the equation [10,11]

\[
\dot{\varepsilon} = A_C \sigma \Omega w D_{GB} / d^3 k T
\]  

\( A_C \): dimensionless factor which considers the grain geometry and is of the order of 50, \( w \): grain boundary width, \( D_{GB} \): coefficient for grain boundary diffusion.)
is noted that the geometrical factors $A_{NH}$ and $A_C$ depend on the grain shape and grain size distribution as has been shown by model calculations [12]. $A_{NH} = 13.3$ and $A_C = 47.7$ was used in this study for estimating diffusion creep rates, which follows the example of Ref. [13].

In these theories, grain boundaries are supposed to act as perfect sources and sinks for vacancies. However, various experimental studies revealed an apparent threshold stress for diffusion creep even in pure metals [14–20] which cannot be explained by the above theories. Some models have been proposed for such a threshold stress for diffusion creep for alloys with particles [21–23] and for pure metals [13], but the models do not describe the observed stress and temperature dependence of the apparent threshold stresses completely.

The aim of the present work is to examine the creep of $\gamma$-TiAl in detail at very low stresses with respect to the occurrence of diffusion creep and eventual threshold stresses. The obtained data are to be compared with the predictions of the available theories and models.

2. Materials

In this work the creep behaviour of single-phase $\gamma$-TiAl and of two-phase ($\gamma + \alpha_2$)-TiAl with $D0_{19}$ structure as second phase has been investigated. In addition, the creep of a ternary TiAl–C alloy with precipitated carbide particles has been studied. All alloys were prepared by inductive levitation melting under argon and solidification in a cold Cu crucible. The alloy compositions, pre-treatments with prior annealing at various temperatures for 24 h and subsequent compression at 1200°C with $10^{-3}$ s$^{-1}$ rate for the TiAl alloys and hot extrusion for the TiAl–C alloy, resulting grain sizes and constituent phases are listed in Tables 1 and 2. The hot compression and extrusion was used for grain refinement by recrystallisation which should favour diffusion creep. The microstructures of the studied alloys are shown in Fig. 1.

3. Experimental

Rectangular specimens ($4 \times 4 \times 8$ mm$^3$) were cut by spark erosion for compression creep tests. The tests were performed by stepwise compressive loading to determine the creep rate-stress dependence for single specimens. The load was increased after a constant strain rate had been reached, i.e. after secondary creep was established. The specimens were compressed only up to about 5–10% to avoid excessive barrelling. All creep tests at temperatures up to 1000°C were performed in an argon atmosphere (99.995%) whereas testing at temperatures above 1000°C was carried out in vacuum ($2.0 \times 10^{-5}$ bar) because of excessive oxidation in the used argon atmosphere.

4. Results and discussion

4.1. Single-phase $\gamma$-TiAl

Fig. 2 shows the secondary creep rate vs. stress curve for the single-phase Ti–55Al alloy at 1000°C in the familiar double-logarithmic plot and a linear plot. It is clearly visible that there is a stronger stress dependence of the creep rate than expected according to a linear relationship, which rules out a large contribution of diffusion creep.

The strong stress dependence indicates dislocation creep, i.e. creep by movement of dislocations which is usually described by the Dorn equation [10] for secondary creep:

$$\dot{\varepsilon}_S = A_{SF} \frac{G(T)b}{kT} D_L \left( \frac{\sigma}{G(T)} \right)^n \quad (3)$$

($A_{SF}$: dimensionless structure factor, $G(T)$: shear modulus, $b$: Burgers vector, $D_L$: coefficient of lattice diffusion, $n$: stress exponent, which is in the range 3–5 for dislocation creep). The data in the double-logarithmic plot of Fig. 2a indicate slopes of the creep-rate-stress curves, i.e. stress exponents in the range 3.2–4.8, which indeed corresponds to dislocation creep. It is noted that

<table>
<thead>
<tr>
<th>Alloy composition</th>
<th>Pre-treatment</th>
<th>Constituent phases</th>
<th>Grain size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti (at.%)</td>
<td>Al (at.%)</td>
<td>Annealing temperature (°C)</td>
<td>Hot compression strain (%)</td>
</tr>
<tr>
<td>45</td>
<td>55</td>
<td>1000</td>
<td>49</td>
</tr>
<tr>
<td>45</td>
<td>55</td>
<td>1200</td>
<td>49</td>
</tr>
<tr>
<td>45</td>
<td>55</td>
<td>1350</td>
<td>49</td>
</tr>
<tr>
<td>49</td>
<td>51</td>
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<td>47</td>
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<td>49</td>
<td>51</td>
<td>1200</td>
<td>47</td>
</tr>
<tr>
<td>49</td>
<td>51</td>
<td>1350</td>
<td>47</td>
</tr>
</tbody>
</table>
dislocation creep and diffusion creep are independent deformation processes, which always act in parallel and contribute to the observed overall creep rate, i.e. the overall creep rate is the sum of the rates of dislocation creep and diffusion creep and is dominated by the faster process.

As to a quantitative description of the observed creep, the parameters in Eq. (3) can be estimated with the help of available data in the literature. The elastic shear modulus at creep temperature is estimated by using the linear temperature dependence of the shear modulus as found for two-phase (γ + α2)-TiAl [24]. With this and the values for \( G(293 \, \text{K}) = 71.4 \, \text{GPa} \) [25] and \( G(1173 \, \text{K}) = 58.4 \, \text{GPa} \) [26], \( G(1273 \, \text{K}) = 56.9 \, \text{GPa} \) is obtained. The length of the Burgers vector is \( 2.83 \times 10^{-10} \, \text{m} \) [26].

The effective lattice-diffusion coefficient for creep is chosen as that of Ti in TiAl, i.e. \( D_L = D_{0L} \cdot e^{-Q_L/RT} \), with \( D_{0L} = 1.53 \times 10^{-4} \, \text{m}^2/\text{s} \) and \( Q_L = 291 \, \text{kJ/mol} \) (\( R = \) gas constant) [27,28]. These values were derived from tracer-diffusion experiments of Ti in γ-TiAl. Here they are used for both kinds of atoms because interdiffusion experiments in γ-TiAl showed nearly no concentration dependence [28]. With the above data, the structure factor can be calculated for the studied alloys. Fig. 3 shows the obtained data for the structure factor of dislocation creep which obviously varies with the observed stress exponent according to the equation

\[
A_{\text{SF}} = A_{\text{SF0}} \cdot e^{a \sigma}
\]

with \( a = 7.4 \) and \( A_{\text{SF0}} = 5.6 \times 10^{-11} \).

Such a relationship was found for various metals, alloys and non-metallic solids in the past [29] — see Table 3.

The obtained parameters can be used to construct deformation-mechanism maps following the example of Ref. [10]. Such maps show the curves of constant creep rates in the different stress-temperature regions in which a particular deformation mechanism dominates. The calculation of the Coble creep rates necessitates the knowledge of the respective coefficient of grain boundary diffusion. The values for the deformation maps in the present study were chosen according to an estimate in Ref. [30]: \( wD_{\text{GB}} = wD_{\text{GB0}} \cdot e^{-Q_{\text{GB}}/RT} \), with \( wD_{\text{GB0}} = 3.0 \times 10^{-12} \, \text{m}^2/\text{s} \) and \( Q_{\text{GB}} = 202 \, \text{kJ/mol} \). This was in good agreement with former experimental creep data for other two-phase TiAl alloys [7]. It is noted that another general estimate for grain boundary diffusion in fcc alloys resulted in \( D_{\text{GB0}} = 0.3 \, \text{cm}^2/\text{s} \) and \( Q_{\text{GB}} = 9 \, RT_m \) (\( T_m = \) melting temperature) [31], which may be relevant for TiAl since the L1_0 structure is derived from the fcc lattice. This latter estimate gives a lower activation energy than the former estimate, but the resulting grain boundary diffusion coefficient is of the same order as the used one in the temperature range of interest. The atomic volume was determined as \( \Omega = 0.7 \cdot b^3 \) [32].

![Fig. 1. Optical micrographs of (a) Ti–51Al, (b) Ti–55Al and (c) Ti–52Al–4C after pre-treatment (Table 1Table 2) before creep.](image)
Fig. 4 shows the deformation-mechanism map for the single-phase Ti–55Al alloy with the smallest grain size \(d=45\ \mu\text{m}\). The stress exponent was chosen as \(n=4\). It can be seen that the experimental data for the Ti–55Al alloy are in the region of dislocation creep besides the rate at the lowest stress level which is just in the Coble creep region. This is not regarded as significant in view of the potential inaccuracy of the used grain boundary diffusion data and the error of the grain size determination. For coarser grained material, the border line between diffusion creep and dislocation creep moves to still lower stresses. Thus, the data for the other two single-phase alloys with larger grain sizes \((d=80\ \mu\text{m}, 100\ \mu\text{m})\) lie completely in the dislocation creep region. It is concluded that the observed creep behaviour of the single-phase alloys do not indicate any notable contribution of diffusion creep to the overall creep rate, which then is controlled only by dislocation creep. Diffusion...
creep can be expected only at still lower stresses. Then Coble creep is the rate-controlling mechanism in the studied temperature range whereas the Nabarro–Herring creep can be expected only at much higher temperatures near the melting point.

4.2. Two-phase \((\gamma + \alpha_2)\)-TiAl

Fig. 5 shows the creep rate vs. stress curve for the two-phase \((\gamma + \alpha_2)\)-Ti–51Al alloy at 1000°C in double-logarithmic and linear plots. It can be seen that there is indeed a linear stress dependence of the creep rate at low stresses, whereas at higher stresses it is stronger. The linear region extends to higher stresses in material with smaller grain size. This behaviour may be regarded as an indication of the dominance of diffusion creep in the low stress region and of dislocation creep at higher stresses and is to be checked in more detail.

The extrapolations to vanishing creep rates in the linear plot intersect the abscissa at values \(\sigma > 0\). This is not in agreement with Eqs. (1) and (2) for diffusion creep and thus indicates an apparent threshold stress. The observed apparent threshold stresses obviously increase with increasing grain size – see Table 4.

The stress exponents for creep in the high stress region are in the range 3.3–3.8 corresponding to dislocation creep. There is again an interrelation between the structure factor and the respective stress exponent according to Eq. (4), but with different parameters – see Table 3. In alloys with second phases, a threshold stress is often observed for dislocation creep [33]. This reflects the retarding action of particles on dislocation migration and results from the climb of dislocations around particles and detachment of dislocations from particles. The threshold stress is usually a fraction of the Orowan stress, which has been expressed by the equation [33]

\[
\sigma_{Or} = \frac{0.84 \cdot M}{2\pi \cdot (1 - \nu)^{1/2}} \frac{Gb}{l} \ln\left(\frac{E_0}{b}\right),
\]

where \(M\) is the Taylor factor for polycrystals, \(\nu\) the Poisson’s ratio, \(l\) the mean spacing between particles and \(r\) the mean particle radius. With \(M = 3\) (for untextured polycrystals [33]), \(\nu = 0.24\) [25], \(l = 1 \times 10^{-4}\) m for the considered alloys and \(r = 1.5 \times 10^{-5}\) m an Orowan stress \(\sigma_{Or} = 0.8\) MPa is calculated, which may be regarded as the upper limit for an eventual threshold stress for dislocation creep in the investigated TiAl alloys. As to the obtained experimental data for the studied two-phase alloys, threshold stresses for dislocation creep would be visible in the \(\dot{\varepsilon}^{1/n}\) vs. \(\sigma\) plot of Fig. 6. The data in the high-stress regime for dislocation creep are well

<table>
<thead>
<tr>
<th>Material</th>
<th>(A_{SF}), 1000°C for (n = 4)</th>
<th>(A_{SF})</th>
<th>(a)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single-phase (\gamma)-TiAl</td>
<td>401</td>
<td>5.6 \times 10^{-11}</td>
<td>7.4</td>
</tr>
<tr>
<td>Two-phase ((\gamma + \alpha_2))-TiAl</td>
<td>64.0</td>
<td>4.9 \times 10^{-10}</td>
<td>6.4</td>
</tr>
<tr>
<td>Pure metals [29]</td>
<td></td>
<td>5.7 \times 10^{-12}</td>
<td>8.5</td>
</tr>
<tr>
<td>Alloys [29]</td>
<td></td>
<td>6.7 \times 10^{-10}</td>
<td>7.0</td>
</tr>
<tr>
<td>Non-metallic solids [29]</td>
<td></td>
<td>1.9 \times 10^{-12}</td>
<td>8.8</td>
</tr>
</tbody>
</table>

Table 3

<table>
<thead>
<tr>
<th>Material</th>
<th>(A_{SF}), 1000°C for (n = 4)</th>
<th>(A_{SF})</th>
<th>(a)</th>
</tr>
</thead>
<tbody>
<tr>
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<td></td>
<td>1.9 \times 10^{-12}</td>
<td>8.8</td>
</tr>
</tbody>
</table>

Fig. 4. Deformation mechanism map for single-phase Ti–55Al (grain size = 45 μm) with experimentally observed creep rates as given in the legend (see text).
described by a straight line, which intersects the abscissa at stresses near zero. This means that the possible threshold stress for dislocation creep is negligibly small in agreement with the above estimate.

Deformation mechanism maps (Figs. 7–9) were calculated as before, which now take the threshold stress for diffusion creep into account and for which an averaged stress exponent $n = 3.5$ was chosen. It can be seen that there is an order-of-magnitude agreement between the measured creep rates and the calculated rates which rely in the dislocation creep field on the observed average structure factor and stress exponent for dislocation creep and in the Coble creep field on the chosen estimate for grain boundary diffusion, i.e. the calculated rates in the diffusion creep fields do not depend on experimental fit parameters. Then the order-of-magnitude agreement between measured and calculated creep rates in the Coble creep field means that the observed creep strain corresponds to what is expected for Coble creep, i.e. the observed creep rates with linear stress dependence may indeed be regarded as indications of Coble creep. Any other independent creep mechanism would only give rise to additional contributions of creep strain and thus would result in higher creep rates. It is noted that diffusion creep is coupled with accommodating grain boundary sliding, i.e. these two mechanisms act in mutual dependence, which, however, does not affect the validity of the constitutive equations for diffusion creep [34].

A further evidence for the action of Coble creep in the low-stress regime is the variation of creep rate with

Table 4
Threshold stresses and upper stress limit for diffusion creep in Ti–51Al with various grain sizes

<table>
<thead>
<tr>
<th>Grain size (μm)</th>
<th>Threshold stress (MPa)</th>
<th>Maximum stress for diffusion creep (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>$3.1 \pm 0.6$</td>
<td>16.9</td>
</tr>
<tr>
<td>50</td>
<td>$3.8 \pm 1.2$</td>
<td>12.5</td>
</tr>
<tr>
<td>60</td>
<td>$6.0 \pm 0.7$</td>
<td>11.0</td>
</tr>
</tbody>
</table>
increasing grain size as visible in Fig. 5b. With the data of Fig. 5b, Fig. 10 shows the creep rate at constant low stress as a function of grain size. It can be seen that the observed decrease of creep rate with increasing grain size indeed corresponds to the theoretical behaviour of Coble creep according to Eq. (2). In view of the observed stress dependence of the creep rate at low stresses, the order-of-magnitude agreement between experiment and theory and the strong grain-size dependence corresponding to theory, it is concluded that Coble creep is the rate-controlling mechanism at stresses below 20 MPa for the studied two-phase TiAl alloys at 1000°C.

4.3. TiAl–C with precipitated carbide particles

The studied TiAl–C alloy (Table 2) contains 16 vol% of the complex carbide Ti2AlC, which is known as H-phase [35,36]. Creep experiments between 900°C and 1200°C have shown that there is again a low-stress regime with approximately linear stress dependence of creep rate and a high-stress regime with much stronger stress dependence of creep rate (Fig. 11). However, a detailed quantitative analysis of the obtained experimental data has revealed that the observed creep rates in the low-stress regime, which may be controlled by diffusion creep, are significantly lower than expected theoretically when using the materials parameters of the preceding sections.

This discrepancy cannot be caused by the action of other creep mechanisms since diffusion creep strains are the lower limit of the total creep strain as mentioned before. Obviously, the used materials parameters for estimating the diffusion creep rates are not adequate for the TiAl–C alloys. Various sources of error have to be considered. First, the structure of the grain boundaries

![Graph](image_url)  
**Fig. 6.** $\varepsilon^{1/n}$ vs. $\sigma$ plot of secondary creep rate as a function of stress for two-phase Ti–51Al with various grain sizes (Table 1) at 1000°C.

![Graph](image_url)  
**Fig. 7.** Deformation mechanism map for two-phase Ti–51Al (grain size = 40 µm) with experimentally observed creep rates as given in the legend (see text).
and phase boundaries in the TiAl–C alloy may differ from that in the TiAl alloys, which affects the effective grain boundary diffusion coefficient in Eq. (2). In addition, it may affect the production and annihilation rate of vacancies, which is supposed to be sufficiently high for derivation of Eq. (2). Second, the grain size distribution may differ from that of the TiAl alloys, which affects the geometry factor $A_C$ in Eq. (2) according to Ref. [12]. The grain size distribution in the TiAl–C alloy was found to be rather inhomogeneous with ensembles of small grains surrounding large grains. In addition, some grain coarsening was observed at the higher temperatures. Consequently, the analysis of the observed creep behaviour with identification of the rate-controlling mechanisms necessitates knowledge of the accurate effective grain-boundary diffusion coefficient as well as a detailed characterisation of the grain size distribution.

An extrapolation to vanishing creep rates in the low-stress regime reveals a strongly temperature-dependent threshold stress for diffusion creep – see Table 5 and Fig. 12. Diffusion creep has been studied experimentally for various pure metals in the past and threshold stresses have been reported as summarised in Ref. [13]. Those threshold stresses showed a much weaker temperature dependence. In view of the observed strong temperature dependence of this threshold stress, the available models for the threshold stress for diffusion creep of single-phase alloys [13] and alloys with particles [21–23] seem to be inadequate for understanding the observed behaviour.

The $\varepsilon^{1/n}$ vs. $\sigma$ plot of the secondary creep data (Fig. 13) shows that there is practically no threshold stress for dislocation creep in the high-stress regime as in the case of the two-phase TiAl alloy. With the
obtained experimental data, the apparent activation energy for dislocation creep has been found as about 380 kJ/mol which is higher than that for bulk diffusion in $\gamma$-TiAl – see Section 4.1. Such a behaviour is not unusual for intermetallic alloys and is usually attributed to temperature-dependent changes in microstructure.

The temperature dependence of the factor $1 = RT G^{n}\nu$ is only small and can be neglected in this context.

5. Conclusions

The creep behaviour of $\gamma$-TiAl with L1\textsubscript{0} structure without second phases, $\gamma$-TiAl with precipitated particles of $\alpha_2$-Ti$_3$Al with D\textsubscript{0\textsubscript{19}} structure and of $\gamma$-TiAl with the H-phase Ti$_2$AlC has been studied at low stresses in the temperature range 900–1200°C. The obtained data

<table>
<thead>
<tr>
<th>Temperature</th>
<th>900°C</th>
<th>950°C</th>
<th>1000°C</th>
<th>1100°C</th>
<th>1150°C</th>
<th>1200°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\sigma_t$ (MPa)</td>
<td>27.5 ± 0.2</td>
<td>18.8 ± 0.2</td>
<td>6.6 ± 0.6</td>
<td>1.2 ± 0.3</td>
<td>0.7 ± 0.3</td>
<td>0.3 ± 0.1</td>
</tr>
</tbody>
</table>
allow the construction of creep deformation mechanism maps for the studied TiAl alloys which may be used for the interpolation and extrapolation of the observed creep behaviour. The following conclusions are drawn from the results.

**Dislocation creep:**
- At higher stresses (e.g. above the order of 10 MPa at 1000°C for the alloy Ti–51Al) dislocation creep occurs in all alloys, which is well described by the Dorn equation with stress exponents in the range 3–5.
- Threshold stresses for dislocation creep are negligible for all studied alloys.
- The correlation between the structure factor in the Dorn equation for dislocation creep and the stress exponent is similar to that for various other materials.
- The structure factors for dislocation creep of the two-phase alloys are markedly lower than that for the single-phase alloy.

**Diffusion creep:**
- Nabarro–Herring creep is expected only at temperatures near the melting point.
- Extended Coble creep without notable contributions of dislocation creep occurs in the studied stress–temperature range only in the two-phase TiAl alloys.
- The observed Coble creep rates indicate the presence of threshold stresses.
- The observed threshold stresses for creep of the TiAl–C alloy in the low-stress regime indicate strongly temperature-dependent threshold stresses, which are not accounted for by available models.
- An improved physical understanding of the observed behaviour necessitates accurate data for the diffusion in the grains and at the grain boundaries as well as a detailed characterisation of the grain size distribution.

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**References**