

# Steady-state creep behaviour of Ti<sub>3</sub>Al-base intermetallics

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The steady-state creep behaviour of Ti<sub>3</sub>Al and Ti<sub>3</sub>Al + 10 wt% Nb was studied in the temperature range 550 to 825° C and in the stress range 69 to 312 MN m<sup>-2</sup>. The temperature and stress dependences of the steady-state creep rates were determined for both intermetallics, and the activation energy and stress-exponent were measured. At temperatures above 700° C, the stress dependence of the steady-state creep rate indicated two distinct creep regimes: at stresses above 138 MN m<sup>-2</sup>, the creep was controlled most probably by dislocation climb; at stresses below 138 MN m<sup>-2</sup>, a transition regime with a lower stress-exponent value was obtained.

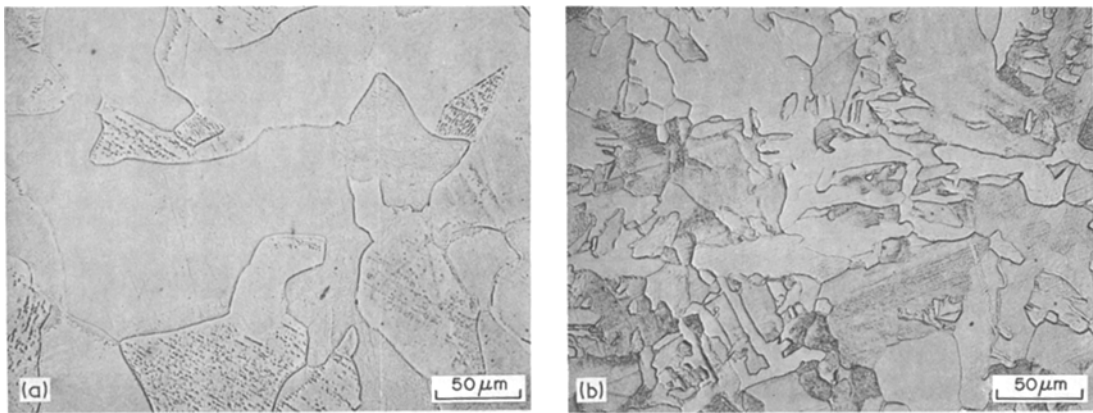
## 1. Introduction

Considerable research efforts are presently being directed toward development of  $\alpha_2$ -phase titanium aluminides as structural materials for aircraft turbine engines. The  $\alpha_2$ -phase is based upon the composition Ti<sub>3</sub>Al and has an ordered DO<sub>19</sub>-type lattice structure. Recent work [1] has shown that the Ti<sub>3</sub>Al-base alloys, although brittle at low temperatures, retain reasonable strength levels up to 800° C. At about 550° C the alloys begin to exhibit measurable ductility which increases gradually with increasing temperature. The tensile deformation modes in Ti<sub>3</sub>Al have been studied by Williams and Blackburn at room temperature [2] and by Sastry and Lipsitt at 700° C [3]. The main findings of these investigations are that slip in Ti<sub>3</sub>Al is highly planar and is comprised mostly of  $\frac{a}{2}\langle 11\bar{2}0 \rangle$ -type (“a”) dislocations. A few  $\frac{a}{2}\langle 11\bar{2}6 \rangle$ -type (“c + a”) dislocations have also been observed [2]. The ordered lattice structure and restriction of slip systems observed in Ti<sub>3</sub>Al appear to be responsible for both the high-temperature strength and the low-temperature brittleness. Ordering-transformations [3] and strain-amplitude controlled cyclic deformation [4] studies have also been carried out recently on Ti<sub>3</sub>Al-base intermetallics.

The present investigation is directed toward an understanding of the steady-state creep behaviour in Ti<sub>3</sub>Al-base materials in the stress and temperature ranges which correspond to practical applications. This goal was pursued by establishing the stress and temperature dependences of the steady-state creep rates.

## 2. Experimental procedure

Two intermetallics were used in this investigation: the near-stoichiometric Ti<sub>3</sub>Al (of nominal composition Ti–16 wt% Al) and Ti<sub>3</sub>Al + 10 wt% Nb (of nominal composition Ti–16 wt% Al–10 wt% Nb). Nb is added in the Ti<sub>3</sub>Al-base alloy primarily to improve the oxidation resistance; significant improvement has been shown to occur [5] with the addition of 5 to 10 wt% Nb. The alloys used in this study were cast as rod ingots and converted by Nuclear Metals Inc., to a powder form of –35 mesh using a rotating electrode process. The powders were packed in Ti–6Al–4V cans, evacuated, sealed, and hot-extruded at 1200° C with an extrusion ratio of 26:1. The details of the extrusion process, the method of specimen machining, and the geometry of the specimen are described in a recent paper [6]. The machined specimens were given an annealing treatment of



**Figure 1** (a) Microstructure of extruded and heat-treated Ti-16 wt % Al. Extrusion is followed by heat treatment at 1000° C for 4 h and then furnace cooled. (b) Light micrograph showing microstructure of extruded and heat-treated  $Ti_3Al + 10 \text{ wt \% Nb}$ . Extrusion is followed by heat treatment at 1000° C for 4 h and then air cooled.

1000° C for 4 h in vacuum, in order to stabilize the microstructure, and were then furnace cooled.

A lever-type constant-load creep machine was utilized for the creep experiments. The tests were performed in air at stresses ranging from 69 to 312  $MN m^{-2}$  and at temperatures ranging from 550 to 825° C. During the creep tests the temperature was controlled to within  $\pm 2^\circ$  C. Most of the tests were carried out at constant (nominal) stresses; however, in some tests, stress levels were increased on the same specimen after the onset of steady-state creep. In all experiments the steady-state creep regimes were observed to be well established below 2% total strain. Thus, the correction necessary to convert the nominal stress to true stress was very small and, therefore, neglected. In the creep experiments the specimen elongation was continuously recorded by means of a linear variable differential transformer (LVDT) and a mechanical extensometer clamped onto the flanges of the specimens. After the creep had proceeded well into the steady-state regimes, creep tests were discontinued and specimens cooled under load to minimize recovery of the deformation structures.

### 3. Results and discussion

#### 3.1. Microstructures of the heat-treated intermetallics

The microstructures of the extruded and subsequently heat-treated  $Ti_3Al$  and  $Ti_3Al + 10 \text{ wt \% Nb}$  are shown in the light micrographs of Fig. 1a and b, respectively. For  $Ti_3Al$  the heat treatment produced a nearly equiaxed grain structure (with average grain size approximately

100  $\mu m$ ) with some jagged grain boundaries. For  $Ti_3Al + 10 \text{ wt \% Nb}$  the microstructure consisted of highly irregular (non-equiaxed) grains having very jagged boundaries. Also in  $Ti_3Al + 10 \text{ wt \% Nb}$  the average grain size was smaller (about 60  $\mu m$ ) than that in  $Ti_3Al$ . It appears that Nb additions in  $Ti_3Al$  retard the diffusive processes required for recrystallization and grain growth. Polished specimens of both intermetallics were scanned using the electron microprobe over distances up to 1 mm; the characteristic X-ray intensities corresponding to Ti, Al, and Nb did not exhibit compositional heterogeneities. TEM bright-field and selected-area diffraction examination of thin foils revealed the presence of only the ordered  $\alpha_2$ -phase in both the  $Ti_3Al$  and  $Ti_3Al + 10 \text{ wt \% Nb}$ . In  $Ti_3Al$  the grains contained a very low density of dislocations and were uniformly recovered. In  $Ti_3Al + 10 \text{ wt \% Nb}$ , however, while some of the grains were observed to be nearly fully recovered, most of them consisted of aligned, rather thin, long sub-grains. Most of the sub-boundaries were simple tilt boundaries containing  $a$ -type dislocations; however, some consisted of  $a$ -type dislocation networks. The long sub-grain morphology is thought to be the result of partial annealing of the martensitic substructure that formed during cooling in  $Ti_3Al + 10 \text{ wt \% Nb}$  after the extrusion process [3]. The martensitic transformation is related to the stabilization of a high-temperature bcc  $\beta$ -phase due to the addition of Nb in  $Ti_3Al$ ; in  $Ti_3Al$  without Nb the furnace cooling is not sufficiently fast to produce a martensitic transformation [3].

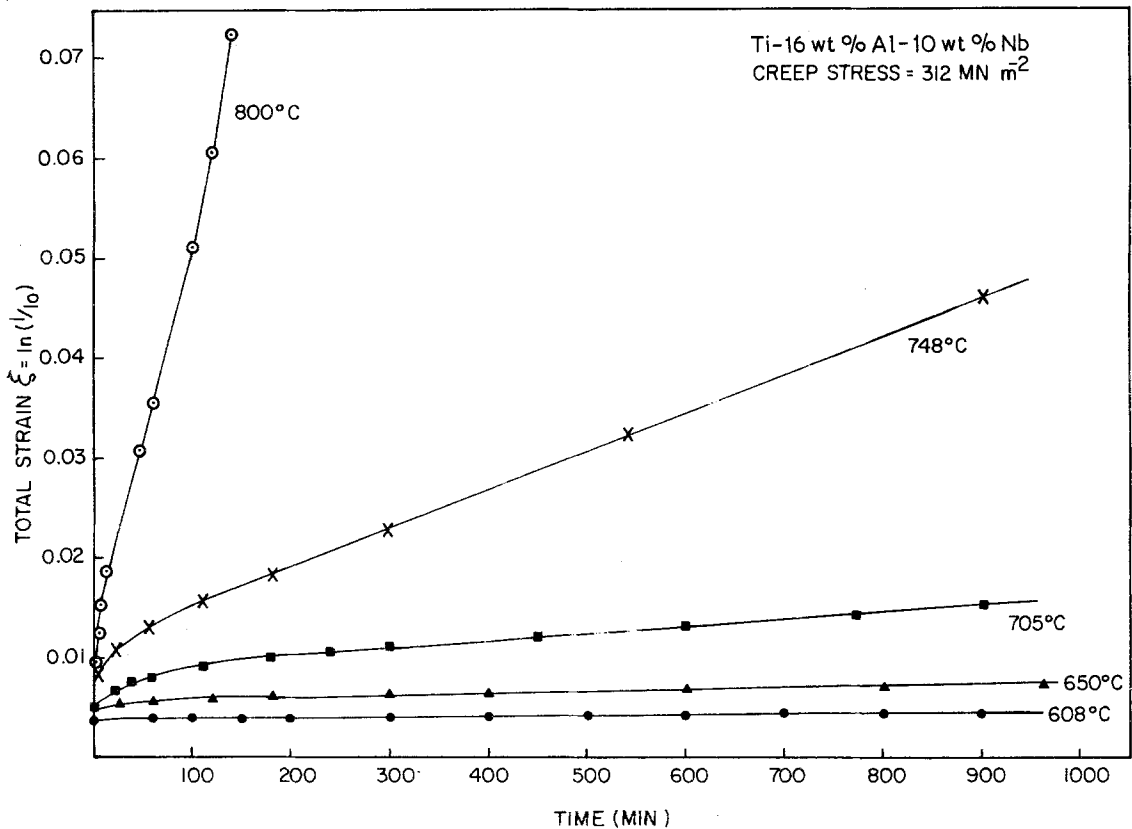


Figure 2 Creep curves for  $Ti_3Al + 10 \text{ wt \% Nb}$ .

### 3.2. Creep curves

Fig. 2 is an example of the type of creep exhibited by the intermetallics. At all stresses and temperatures utilized, the creep curves exhibited primary regimes followed by steady-state creep. In the stress-change experiments in which the stress was increased after establishment of the steady-state creep regime, further primary creep was exhibited. These observations indicated that, in the intermetallics used in this investigation, the creep deformation proceeds by the continued formation of a more creep-resistant sub-structure during the primary stage and by stabilization of this structure by some recovery process or processes during the steady-state stage. For the same stress values, the measured steady-state creep rates were found to be about the same in both the single-stress and the stress-change experiments. This indicated that the steady-state creep was not sensitive to the differences in the prior deformation structure.

### 3.3. Temperature dependence of steady-state creep rate

#### 3.3.1. $Ti_3Al$

Steady-state creep rates,  $\dot{\epsilon}_s$ , were obtained by

calculating the slopes of the secondary regimes of the types of creep curves shown in Fig. 2. For  $Ti_3Al$ ,  $\dot{\epsilon}_s$  is plotted in Fig. 3 as a function of the reciprocal of the absolute temperature,  $1/T$ , for four stress values, 138, 172.4, 207, and  $241 \text{ MN m}^{-2}$ . The data were represented by parallel straight lines indicating that the rate-controlling thermal-activation process is independent of stress level. From the least-squares slopes of these lines, an average value of  $2.23 \times 10^5 \text{ J mol}^{-1}$  was obtained for the apparent activation energy of creep,  $Q_a$ .

#### 3.3.2. $Ti_3Al + 10 \text{ wt \% Nb}$

Fig. 4 is an Arrhenius plot showing the temperature dependence of  $\dot{\epsilon}_s$  for  $Ti_3Al + 10 \text{ wt \% Nb}$  for two stress values, 312 and  $138 \text{ MN m}^{-2}$ . The data corresponding to the  $312 \text{ MN m}^{-2}$  stress could be represented by two straight lines having different slopes. Above about  $650^\circ \text{C}$ , the straight line had a larger slope and, from a least-square calculation, an apparent activation energy,  $Q_a$ , of approximately  $3.1 \times 10^5 \text{ J mol}^{-1}$  was obtained. Below about  $650^\circ \text{C}$ , the straight lines had a smaller slope yielding a value of  $Q_a$  equal to

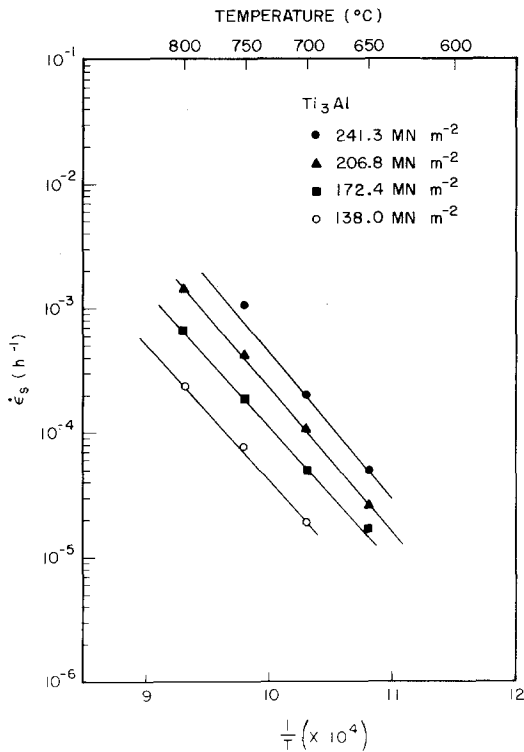


Figure 3 Temperature dependence of steady-state creep rate for  $Ti_3Al$ .

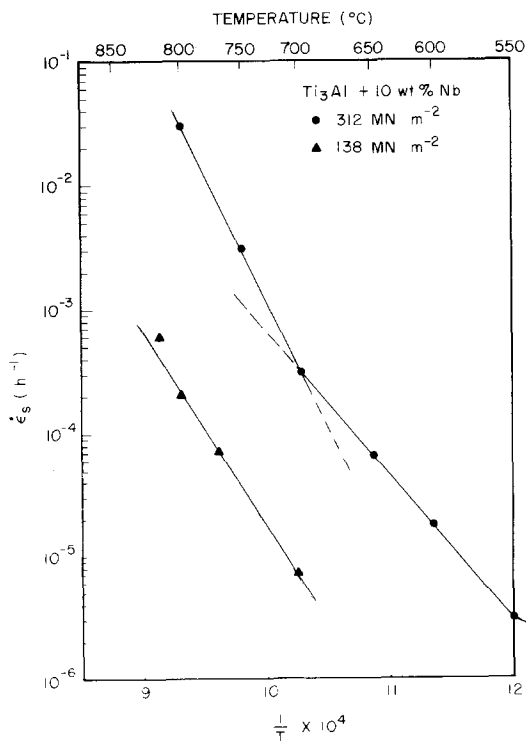


Figure 4 Temperature dependence of steady-state creep rate for  $Ti_3Al + 10 \text{ wt } \% Nb$ .

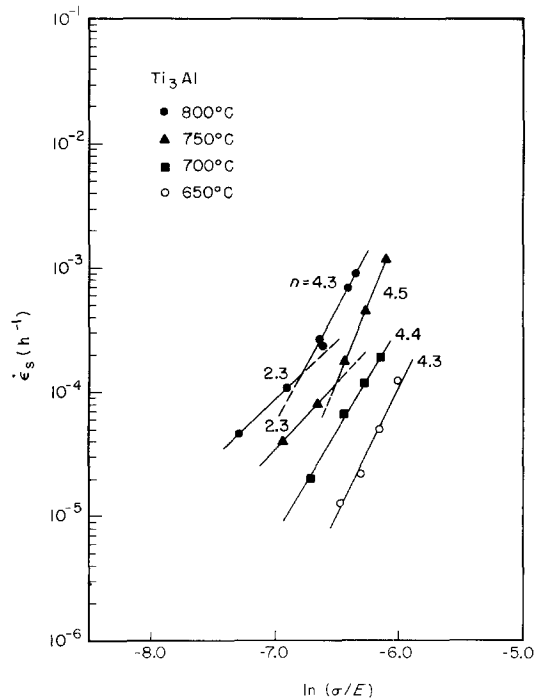


Figure 5 Normalized stress dependence of steady-state creep rate for  $Ti_3Al$ .

approximately  $1.9 \times 10^5 \text{ J mol}^{-1}$ . For a stress value of  $138 \text{ MN m}^{-2}$ , the creep runs were made only in the  $700$  to  $825^\circ \text{C}$  range, the creep strains being extremely small below  $700^\circ \text{C}$  because of the low stress utilized. The data could be represented by a straight line which was very nearly parallel to the straight line for  $312 \text{ MN m}^{-2}$  creep runs above about  $650^\circ \text{C}$ . Thus, above about  $650^\circ \text{C}$ ,  $Q_a$  appears to be independent of stress. Below  $650^\circ \text{C}$ , a lower-activation-energy creep mechanism appears to be rate controlling.

### 3.4. Stress dependence of steady-state creep rates

#### 3.4.1. $Ti_3Al$

In Fig. 5, values of  $\dot{\epsilon}_s$  for  $Ti_3Al$  are plotted as a function of  $\ln(\sigma/E)$  for temperatures varying from  $650$  to  $800^\circ \text{C}$ . The temperature dependence of Young's modulus,  $E$ , has been reported for both  $Ti_3Al$  and  $Ti_3Al + 10 \text{ wt } \% Nb$  over the temperature range between  $25$  and  $940^\circ \text{C}$  [8]. The fact that the creep data could be represented by straight lines indicates a power-law stress dependence of  $\dot{\epsilon}_s$ . The data corresponding to  $750$  and  $800^\circ \text{C}$  could not be represented by single straight lines. At stresses below  $138 \text{ MN m}^{-2}$ , the data fit straight lines having smaller slopes than

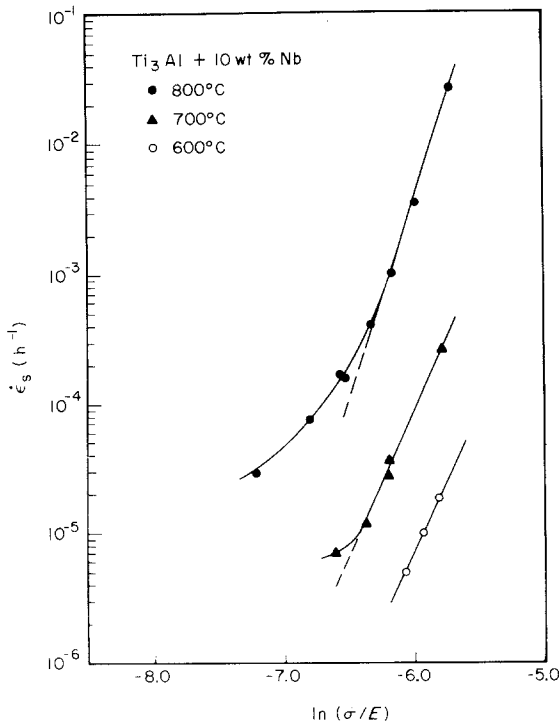


Figure 6 Normalized stress dependence of steady-state creep rate for  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ .

those for the higher stresses. This is taken as an indication of a change in the creep mechanism at these low stresses.

### 3.4.2. $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$

In Fig. 6, values of  $\dot{\epsilon}_s$  for  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$  are plotted as a function of  $\ln(\sigma/E)$  obtained at 600, 700, and 800°C. From Fig. 6 it can be seen that above a stress level of about  $172.5 \text{ MN m}^{-2}$ , the data for all temperatures could be represented by nearly parallel straight lines, indicating the power-law stress dependence of  $\dot{\epsilon}_s$ . Below a stress level of  $172.5 \text{ MN m}^{-2}$ , the straight lines had a smaller slope for both 800 and 700°C, indicating a change in mechanism. This behaviour is similar to that observed in  $\text{Ti}_3\text{Al}$  except for the fact that in  $\text{Ti}_3\text{Al}$ , the change occurs at a stress level of about  $138 \text{ MN m}^{-2}$ .

The change in mechanism indicated by the data in Figs. 5 and 6 can be better represented by plotting  $\dot{\epsilon}_s$  against  $(\sigma/E)$  on a temperature-compensated basis. A correction in the value of  $Q_a$  is required before temperature compensation can be attempted. The physical basis for this correction lies in the fact that, in the Arrhenius plots of Figs 3 and 4, the temperature dependence of  $E$  is

not taken into account, even though  $\dot{\epsilon}_s$  is correlated with  $\sigma/E(T)$  in Figs 5 and 6. Lund and Nix [9] have shown that in such a case, the activation energy for creep,  $Q_c$ , is given by

$$Q_c = Q_a + \left( nR \frac{T^2}{E} \right) \left( \frac{dE}{dT} \right), \quad (1)$$

where  $Q_a$  is defined as the apparent activation energy obtained from an Arrhenius plot,  $n$  is the normalized stress exponent,  $R$  is the gas constant,  $T$  is the absolute temperature, and  $dE/dT$  is the temperature dependence of Young's modulus. The value of  $dE/dT$  for most materials is negative and so  $Q_c < Q_a$ . The values of  $dE/dT$  were obtained from the  $E$  against  $T$  data given in [8] for both  $\text{Ti}_3\text{Al}$  and  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ . Taking average stress exponent values of 4.5 and 6 for  $\text{Ti}_3\text{Al}$  and  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ , respectively (from Figs. 5 and 6), the average correction values (second term in Equation 1) for the range of creep temperatures were calculated to be about  $-1.65 \times 10^{-4} \text{ J mol}^{-1}$  for  $\text{Ti}_3\text{Al}$  and about  $-2.48 \times 10^{-4} \text{ J mol}^{-1}$  for  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ . These values are small and therefore, not significant.

The temperature compensation can now be achieved by plotting  $\ln \dot{\epsilon}_s + Q_c/RT$  as a function of  $\ln(\sigma/E)$  for all  $\dot{\epsilon}_s$  data for  $\text{Ti}_3\text{Al}$  from 650 to 800°C and for  $\dot{\epsilon}_s$  data corresponding to 700 and 800°C for  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ . The 600°C data for  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$  cannot be included because of the lower activation energy shown in Fig. 3. The temperature-compensated plots of Fig. 7 clearly show the change in creep mechanism at lower stresses.

### 3.5. Phenomenology of steady-state creep

The temperature and stress dependences of  $\dot{\epsilon}_s$  for both  $\text{Ti}_3\text{Al}$  and  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$  indicate that, for the stress and temperature range investigated,  $\dot{\epsilon}_s$  can be described by

$$\dot{\epsilon}_s \propto (\sigma/E)^n \exp(-Q_c/RT) \quad (2)$$

Creep in  $\text{Ti}_3\text{Al}$  is characterized by an activation energy of  $2.06 \times 10^5 \text{ J mol}^{-1}$  at all stresses and temperatures. The stress exponent,  $n$ , has a value of between 4.3 and 5 at stresses above about  $138 \text{ MN m}^{-2}$ . At temperatures at and above 750°C and at stresses below about  $138 \text{ MN m}^{-2}$ , the stress exponent has a value of between 2.3 and 2.5.

For  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$  above 650°C, creep is characterized by an activation energy of  $2.85 \times 10^5 \text{ J mol}^{-1}$ . Two distinct rate-controlling creep mech-

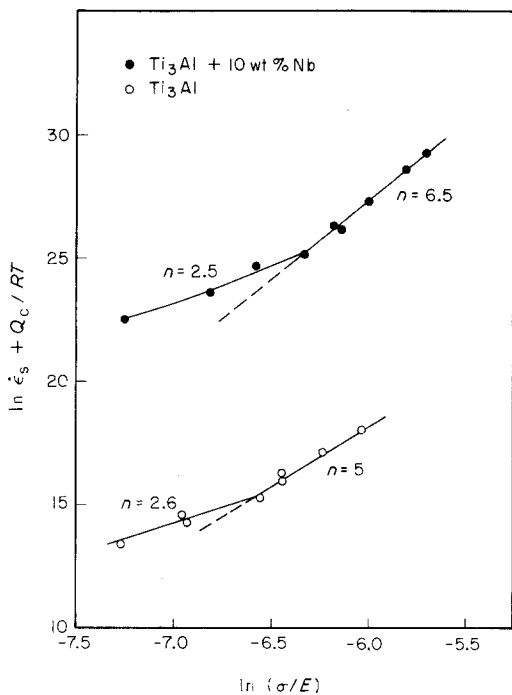


Figure 7 Normalized dependence of temperature-compensated steady-state creep for both  $\text{Ti}_3\text{Al}$  and  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ .

anisms are indicated above  $650^\circ \text{C}$ , one mechanism having an  $n$  value of 6.5 above  $172.5 \text{ MN m}^{-2}$  stress and the other having an  $n$  value of 2.5 below a stress value of  $172.5 \text{ MN m}^{-2}$ . Below  $650^\circ \text{C}$  the creep is controlled by yet another activated process having an activation energy of about  $1.9 \times 10^5 \text{ J mol}^{-1}$  and a stress exponent of 5.5.

At present, it is difficult to identify which particular creep mechanism is rate-controlling in different temperature and stress regimes; this difficulty stems primarily from the non-availability of diffusion data for  $\text{Ti}_3\text{Al}$ -base intermetallics. The measured creep-activation energies cannot be compared to those for diffusion. At high temperatures and stresses, activation energies of  $2.06 \times 10^5 \text{ J mol}^{-1}$  and  $2.85 \times 10^5 \text{ J mol}^{-1}$  for  $\text{Ti}_3\text{Al}$  and  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ , respectively, and the stress exponent values of between 4 and 6, indicate that dislocation climb is the most probable rate-controlling process. The change in mechanism indicated at low stresses may be, in fact, a transition regime and, at very low stresses and high temperatures, grain-boundary sliding may be that rate-controlling mechanism.

#### 4. Conclusions

The creep activation energy for  $\text{Ti}_3\text{Al}$  was found to be  $2.06 \times 10^5 \text{ J mol}^{-1}$  for the stress and temperature range investigated. Addition of Nb increased the activation energy to a value of  $2.85 \times 10^5 \text{ J mol}^{-1}$  for creep above  $650^\circ \text{C}$  and decreased it to a value of  $1.9 \times 10^5 \text{ J mol}^{-1}$  for creep below  $650^\circ \text{C}$ .

The stress exponent for both intermetallics was found to be stress dependent at high temperatures. For  $\text{Ti}_3\text{Al}$ , the stress exponent had a value of between 4.3 and 5 above a stress value of  $138 \text{ MN m}^{-2}$  and between 2.3 and 2.5 below a stress value of  $138 \text{ MN m}^{-2}$ . For  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$  the stress exponent had a value of 6.5 above a stress of  $172.5 \text{ MN m}^{-2}$  and 2.5 below a stress of  $172.5 \text{ MN m}^{-2}$ . At low temperatures the stress exponent was found to be independent of stress for both materials, having values of between 4.3 and 5 and of 6.5 for  $\text{Ti}_3\text{Al}$  and  $\text{Ti}_3\text{Al} + 10 \text{ wt } \% \text{ Nb}$ , respectively.

Due to non-availability of diffusion data at present, it was not possible to identify the rate-controlling creep mechanisms in these intermetallics.

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