JOURNAL OF MATERIALS SCIENCE 35 (2000) 4533–4537

Nitrogen-doped TiAl alloys

Part II *Plastic deformation behavior*

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Effects of the nitrogen addition on high temperature tensile properties and creep resistance of the fully lamellar and the duplex Ti-48.5Al-1.5Mo (at.%) alloy were investigated. High temperature yield strength of the nitrogen-doped alloys increased due to solute hardening and precipitation hardening of Ti_3AlN. Nitrogen addition led to remarkable improvement of creep resistance in the duplex microstructure as well as in the fully lamellar microstructure. In particular, the primary creep deformation of the 1.0 at.% nitrogen-doped alloy with the duplex microstructure decreased definitely to a similar or superior level of the fully lamellar alloy. We consider that precipitate hardening of p-phase (Ti_3AlN) and solute hardening of nitrogen atoms may be responsible for such remarkable creep resistance of the nitrogen-doped alloys.

1. Introduction

Gamma-based TiAl alloys have been developed for application to high temperature structural components [1]. For high temperature application, high temperature yield strength and creep resistance must be considered preferentially. In general, fully lamellar TiAl alloys have superior creep resistance to the duplex alloys.

The major factors determining the creep resistance of TiAl alloys are microstructural variables such as the volume fraction of lamellar grains, size of equiaxed γ-phase, lamellar spacing, reinforcements/particles, and alloying elements [2]. Recently, the effects of interstitial alloying elements such as B, C and N on mechanical properties, especially on creep, have been investigated [3–5]. Addition of 0.3 at.% C to Ti-48Al-1V enhanced the creep resistance significantly regardless of its microstructure [3]. The authors of the present study have reported that the 1.0 at.% N addition to the lamellar Ti-48.5Al-1.5Mo alloy is very effective in the improvement of creep resistance [4]. In the case of B, it is known to be detrimental to the creep resistance of lamellar Ti-46.5Al-2.5Nb-2Cr [5].

We also have reported that both the carbon- and nitrogen-doped alloys of fully lamellar structure show similar microstructural characteristics; the size and volume fraction of precipitates such as Ti_3AlC/Ti_3AlN and Ti_2AlC/Ti_2AlN, and lamellar grain size on the amount of C or N from 0 to 2 at.% [6]. In addition, room temperature tensile properties are not very different from each other [6]. Therefore, we decided to study the high temperature mechanical properties of only the nitrogen-doped TiAl alloys. The main purposes of the present study are to investigate the high temperature tensile properties and the creep resistance of the nitrogen-doped alloys, and to consider their dependence on the amount of nitrogen and microstructures.

2. Experimental

The 1.5Mo (Ti-48.5Al-1.5Mo), 0.3N (Ti-48.5Al-1.5Mo-0.3N), and 1.0N (Ti-48.5Al-1.5Mo-1.0N) alloys were prepared by the same process described in Part I. The ingots were homogenized at 1200°C for 24 hours. Then they were annealed at 1390°C for 2 hours to obtain the fully lamellar microstructure, and annealed at 1300°C for 24 hours for the duplex microstructure.

The high temperature tensile specimens were manufactured into plates with the gauge length of 5 mm and the effective cross section area of 2×1 mm^2 using a spark cutting machine. The specimens were polished first mechanically and then electrically. The tensile tests were performed at 800°C in the rate of 2×10^{-4} s^{-1}. The creep specimens of 3.2 mm in diameter and 15 mm in gauge length were machined from the ingots after heat treatment. Constant stress tensile creep tests were performed using a creep machine equipped with the constant stress Andrade-Chalmers cam. A linear variable differential transformer (LVDT) and an LVDT signal conditioner were used to measure the creep strain. The displacement measurement accuracy was 5×10^{-7} m. The creep tests were performed under 200 MPa stress at 800°C.

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3. Results and discussion

3.1. High temperature tensile properties

Fig. 1 shows the tensile stress-strain curves obtained for 1.5Mo, 0.3N, 1.0N alloys at 800°C. The marks of FLA and DA in this figure represent the fully lamellar and the duplex microstructure, respectively. Letter "A" after FL and D means the aging treatment at 900°C for 12 hours following the annealing for microstructure control. Table I shows high temperature tensile properties obtained from Fig. 1.

In the case of fully lamellar alloys marked FLA in the figure, high temperature ductility shows similar tendency to room temperature ductility. 35.5% of ductility of the 1.5Mo alloy decreased 3.0 % of the 0.3N alloy and recovered 9.3% of the 1.0N alloy. This behavior seems due to the precipitation hardening and grain size refinement. Precipitate hardening from Ti_3AlN precipitate decreased ductility markedly in the case of nitrogen-doped alloys. However the 1.0N alloy with fine lamellar grains seems to be deformed easily at high temperatures due to grain boundary sliding. The yield and ultimate tensile stresses of the nitrogen-doped alloys were higher than that of the 1.5Mo alloy at 800°C, as well as at room temperature.

In the case of duplex alloys marked DA in the figure, all alloys have better ductility than the fully lamellar alloys. Moreover the tendency of ductility in the duplex alloys is similar to that of fully lamellar alloys. The reason for better ductility of the duplex alloys than the fully lamellar alloys is finer lamellar and gamma grains. Thus grain boundary sliding from 20–50 μm of fine grains is the predominant effect on high temperature than precipitation hardening. High temperature yield strength of the duplex alloy also increased with nitrogen content. In general, the yield strength of fully lamellar alloy is higher than that of the duplex microstructure. On the contrary, the yield strength of the 1.0N alloy with duplex microstructure is higher than that of the 1.0N alloy with fully lamellar microstructure. Since the high-temperature yield stress has an important engineering implication for creep properties, it is expected that the 1.0N alloy with the duplex microstructure has good creep properties.

TABLE I Tensile properties of 1.5Mo alloy and its nitrogen-doped alloys at 800°C

Alloy	Microstructure	Elongation (%)	YS (MPa)	UTS (MPa)
1.5Mo	Fully lamellar	35.5	207	401
	Duplex	81.4	219	362
0.3N	Fully lamellar	3.0	176	264
	Duplex	18.2	265	398
1.0N	Fully lamellar	9.3	326	514
	Duplex	61.2	345	506

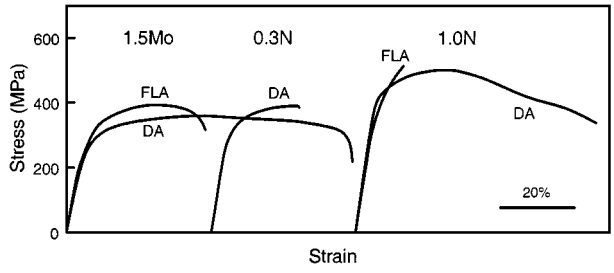

Figure 1 Stress-strain curves obtained for 1.5Mo, 0.3N and 1.0N alloys at 800°C.

3.2. Creep resistance

The effect of nitrogen addition on creep resistance of fully lamellar 1.5Mo alloy is presented in Figs 2 and 3. In Fig. 2a and b, creep curves of the whole region and the primary regimes are shown, respectively. Primary creep resistance of the TiAl alloys are one of the most important high temperature mechanical properties because their deformation tolerance limits are usually less than 1% [7]. The time required to reach 0.2, 0.5 and 1.0% creep strains, and minimum creep rates are shown in Fig. 3 and Table II where all the creep data conducted in the present investigation are summarized. It is clear that the creep resistance of 1.5Mo alloy is much enhanced by addition of nitrogen from 0.3 to 1.0%. Enhancement

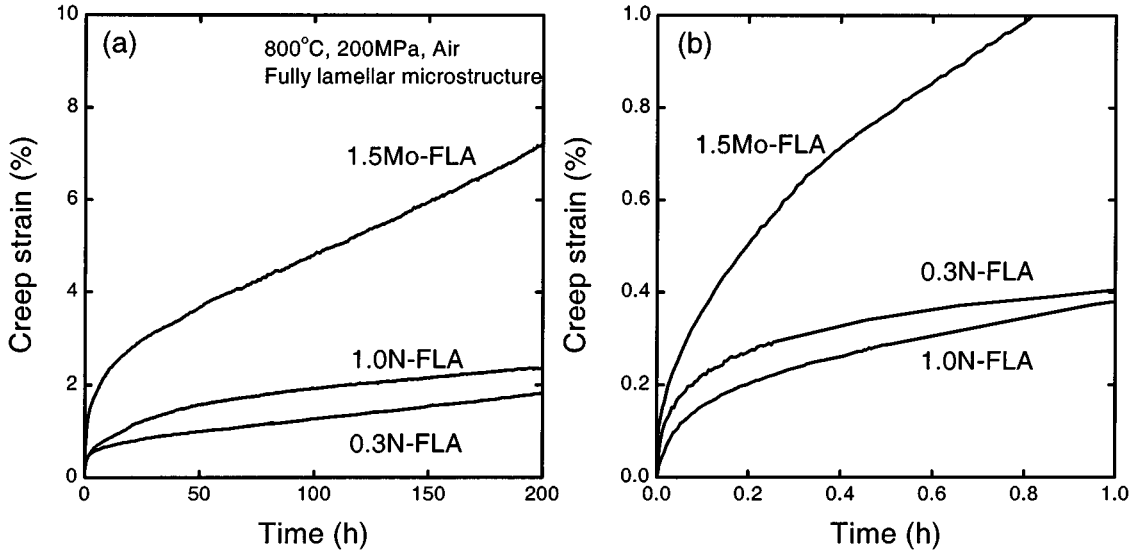

Figure 2 (a) Creep curves of nitrogen-free and nitrogen-doped alloys with fully lamellar microstructure at the condition of 800°C/200 MPa and (b) their zoomed curves in the primary regime.

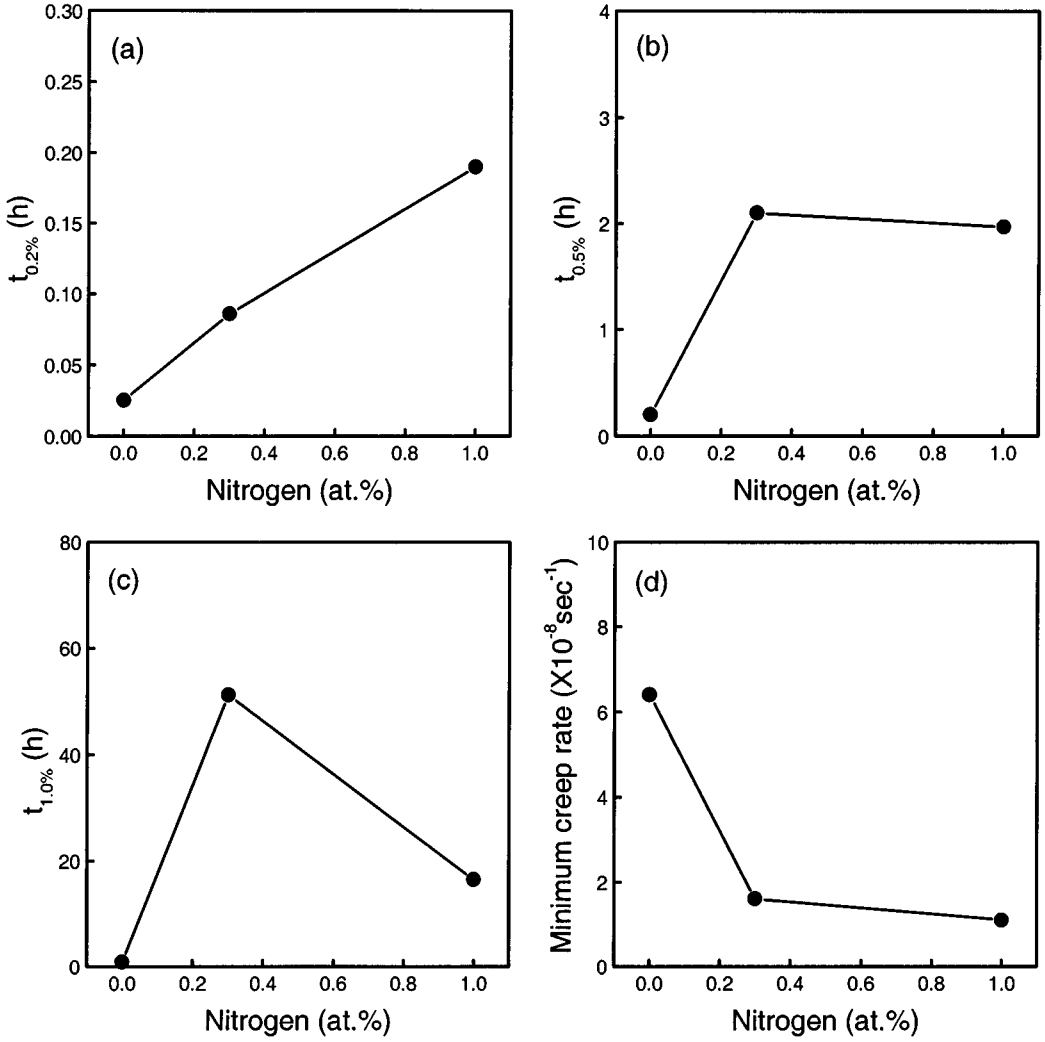

Figure 3 Effect of nitrogen contents on creep resistance of the alloys with the fully lamellar microstructure under the condition of 800°C/200 MPa : (a) $t_{0.2\%}$, (b) $t_{0.5\%}$, (c) $t_{1.0\%}$, and (d) minimum creep rate.

TABLE II Creep tests results of 1.5Mo alloy and its nitrogen-doped alloys at 800°C/200 MPa

Alloy	Microstructure	$t_{0.2\%}$ (h)	$t_{0.5\%}$ (h)	$t_{1.0\%}$ (h)	$\dot{\varepsilon}_m$ (1/sec)
1.5Mo	Fully lamellar	0.025	0.20	0.83	6.4×10^{-8}
	Duplex	0.009	0.11	1.09	4.2×10^{-7}
0.3N	Fully lamellar	0.086	2.10	51.2	1.6×10^{-8}
	Duplex	1.18	29.9	134.6	1.3×10^{-8}
1.0N	Fully lamellar	0.19	1.97	16.5	1.1×10^{-8}
	Duplex	1.43	77.6	230.0	0.89×10^{-8}

of creep resistance by 1.0 at.% N addition has been reported by the authors of this present investigation [4]. Precipitation hardening of fine p-phase (Ti_3AlN) and solute hardening of nitrogen atoms were thought to be responsible for the beneficial effect of 1.0 at.% N addition [4]. While the lamellar grain size and blocky precipitate of Ti_2AlN are different between the 0.3N and 1.0N alloys [6], these features do not affect creep resistance of fully lamellar TiAl alloys [4, 8, 9]. The 0.3N alloy also contains the soluted nitrogen atoms in γ-phase and the fine precipitates of Ti_3AlN as mentioned before in Part I. Therefore, our previous suggestion is clearly confirmed by considering the result that the addition of 0.3 at% N has also a beneficial effect on creep resistance in the same manner as 1.0N alloy (see Figs 2 and 3).

Enhancement of creep resistance of duplex 1.5Mo alloy by adding 0.3–1.0 at.% nitrogen was observed as shown in Figs 4 and 5. Fig. 4a and b show creep curves of the whole region and the primary regime, respectively. The time required to reach 0.5 and 1.0% creep strains and minimum creep rates are plotted in Fig. 5. In the duplex nitrogen-doped alloys, about 30-fold decreased minimum creep rates and more than 2 orders increased $t_{0.5\%}$ and $t_{1.0\%}$ were measured compared with duplex 1.5Mo alloy. In a similar manner as the fully lamellar structure, precipitation hardening of fine p-phase (Ti_3AlN) and solute hardening of nitrogen atoms are certainly responsible for this enhancement of creep resistance in the duplex microstructure.

The effect of microstructure on creep resistance of the 1.5Mo, the 0.3N and the 1.0N alloys are shown in Fig. 6. Here, $t_{0.2\%}$, $t_{0.5\%}$, $t_{1.0\%}$ and minimum creep rates of duplex microstructure are normalized to those of a fully lamellar one. While duplex 1.5Mo alloy shows poorer creep resistance than the fully lamellar one in the same manner as other TiAl alloys [3, 10], we obtained very interesting results for nitrogen-doped alloys (0.3N and 1.0N alloys). That is, the duplex nitrogen-doped alloys have almost the same minimum creep rates

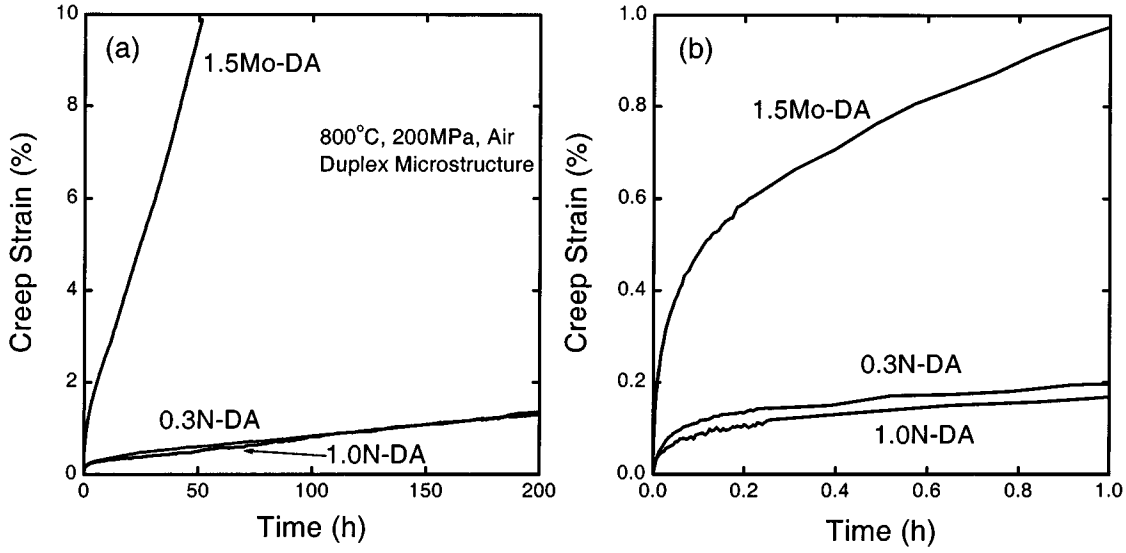

Figure 4 (a) Creep curves of nitrogen-free and nitrogen-doped alloys with duplex microstructure at the condition of 800°C/200 MPa and (b) their zoomed curves in the primary regime.

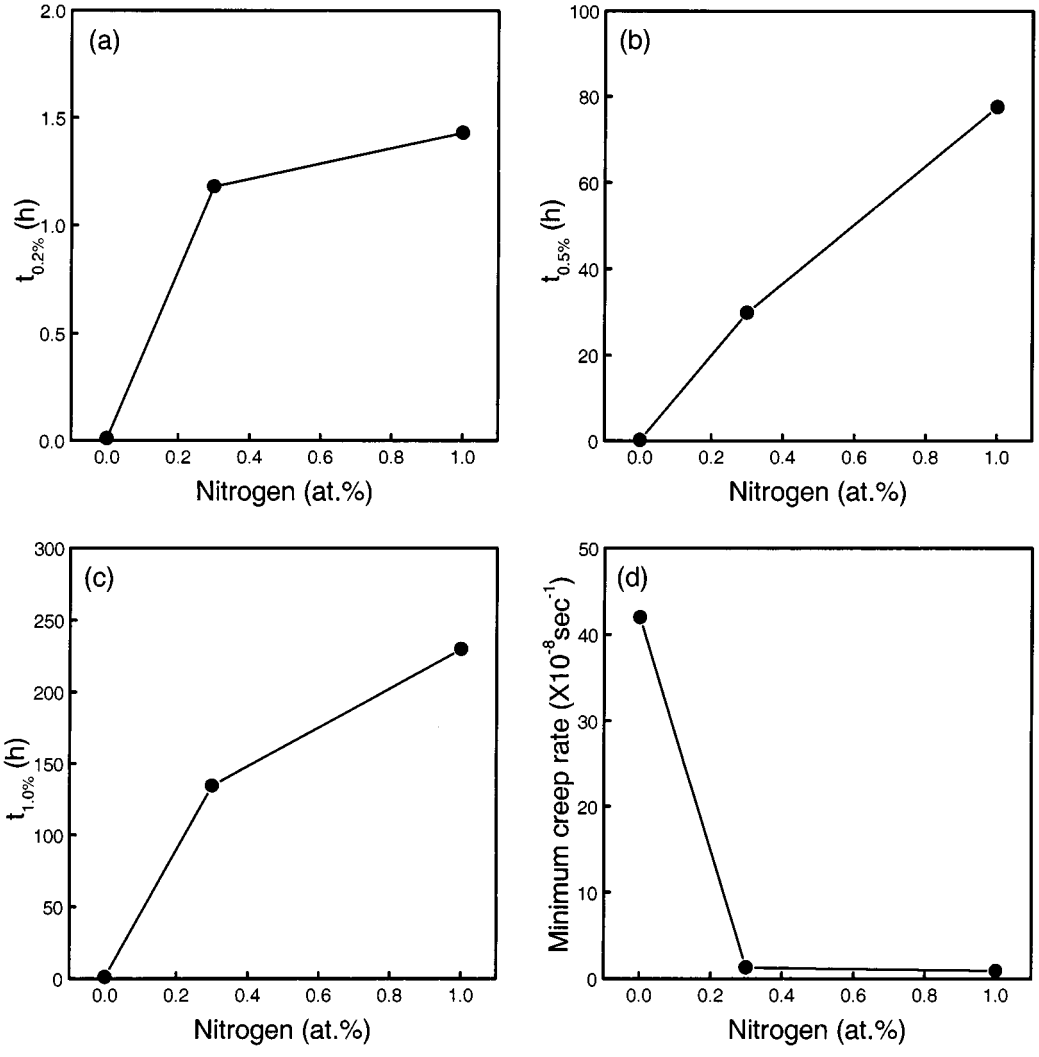

Figure 5 Effect of nitrogen contents on creep resistance of the alloys with the duplex microstructure under the condition of 800°C/200 MPa : (a) $t_{0.2\%}$, (b) $t_{0.5\%}$, (c) $t_{1.0\%}$, and (d) minimum creep rate.

as those of fully lamellar nitrogen-doped alloys, and show 2–25 times longer $t_{0.5\%}$ and $t_{1.0\%}$ than fully lamellar ones. It is also important to note that the amount of primary creep strain (ε_p, at which primary regime ends) of the duplex nitrogen-doped alloys, 0.2–0.3%, are much smaller than those of fully lamellar structures, 0.8–1.1%. In the case of 1.5Mo alloy, this trend is reversed; 2.3% and 1.0% for fully lamellar and duplex,

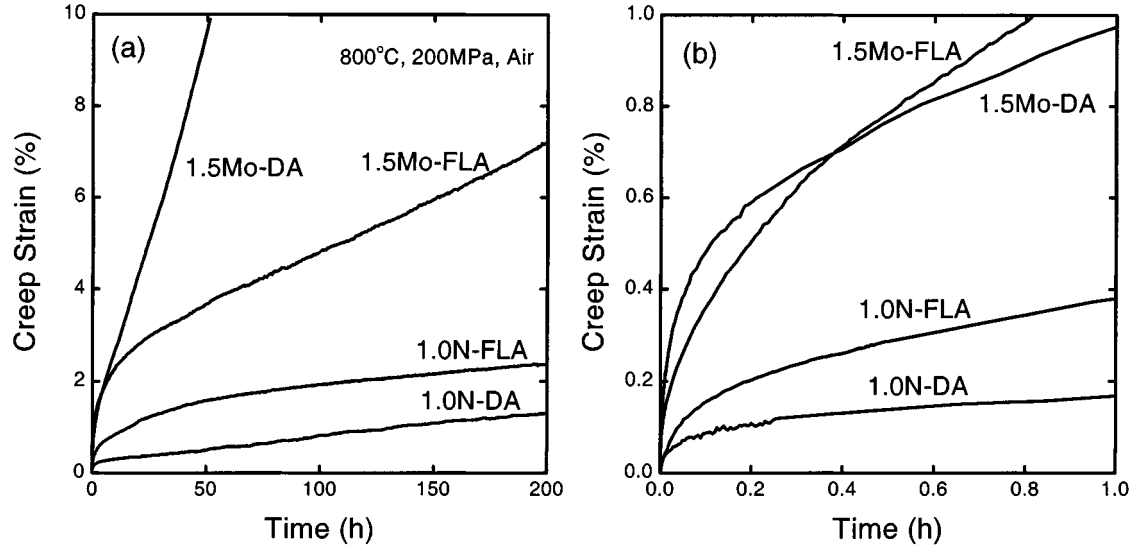

Figure 6 (a) Creep curves of the 1.5Mo and 1.0N alloy with the fully lamellar and the duplex microstructure, respectively and (b) their zoomed curves in the primary regime.

respectively. While Worth *et al*. [3] have reported that the beneficial effect of 0.3%C addition on creep resistance in the duplex structure is more significant than in the fully lamellar, the peculiar results of Fig. 6 have never been reported before. The reason of these results can not be understood at present. In order to understand why the duplex nitrogen-doped alloys have superior creep resistance to fully lamellar, it needs further investigation; such as measurement of activation energies and observation of the sub-structure evolution during creep deformation. By measuring the activation energy of duplex nitrogen-doped alloys, we may conjecture the rate controlling process. Activation energies for fully lamellar microstructure of 1.5Mo and nitrogen-doped alloys were measured and found to have the same values, 430 kJ/mol, so that we suggest that the rate controlling process of the fully lamellar 1.5Mo alloy is not changed by 1 at.% N addition [4].

4. Conclusions

High temperature yield strength increased with increasing nitrogen content in both fully lamellar and duplex microstructure due to solute hardening and precipitation hardening of Ti_3AlN. Moreover, these hardening mechanisms had an effect on the reduction of primary creep strain and steady state creep rate. Therefore nitrogen addition led to remarkable improvement of creep resistance in the duplex microstructure as well as in the fully lamellar microstructure. In particular, primary creep deformation of the 1.0 at.% nitrogen-doped alloy with duplex microstructure decreased definitely to a similar or superior level of the fully lamellar nitrogen-doped alloys.

Acknowledgements

This work was supported by the Korea Science and Engineering Foundation through the Center for Advanced Aerospace Materials, POSTECH, and in part by STEPI.

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Received 14 September 1999
and accepted 22 February 2000