Formation of submicrocrystalline structure in TiAl intermetallic compound

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The TiAl intermetallic compound was used to illustrate an approach which enables the creation of a submicrocrystalline structure ($d \simeq 0.1$ μm) in massive semifinished products made of hard-to-deform materials by means of their deformation at elevated temperatures. Tensile mechanical properties of the TiAl intermetallic compound with a mean grain size of 0.4 μm were tested. In this state, the lower temperature limit of superplasticity in TiAl was found to be 800 °C. At this temperature and at an initial strain rate of 8.3×10^{-4} s^{-1}, the relative elongation to rupture attains 225%.

1. Introduction

Interest in metallic materials with submicrocrystalline structures ($d < 1$ μm) has recently considerably increased. This is due to the fact that such materials possess specific physical and mechanical properties [1–8]. One of the ways of creating the submicrocrystalline structure in massive semifinished metallic products is their deformation at elevated temperatures. The formation of such a structure is induced by dynamic recrystallization developing in the material [9–11]. The application of this technique is substantially complicated with reference to hard-to-deform materials, such as intermetallics and ceramics. The fact that the ductility of these materials is limited even at high temperatures often prevents the attainment of a strain level required to initiate dynamic recrystallization. However, selecting the optimal scheme of loading, temperature–rate conditions of deformation, and original microstructure, the deformability of intermetallics and ceramics can probably be substantially increased and dynamic recrystallization can be induced in them. So far, practically no systematic studies in this field have been carried out.

The present paper is concerned with elaborating a universal method of obtaining submicrocrystalline structures in massive semifinished products prepared out of hard-to-deform materials.

The TiAl intermetallic compound (superlattice $L1_0$) was used for the investigation. This compound possesses properties that are common for both the majority of intermetallics and ceramics. It is brittle and hard-to-deform [12, 13], and was selected because dynamic recrystallization in it is facilitated due to intensive high-temperature twinning [14, 15]. Thus, TiAl is rather convenient for solving the problem in question.

2. Experimental procedure

The Ti–35.8 wt % Al (TiAl) alloy was used for the experiment. The material was prepared in the following two ways: (1) compaction of granules, and (2) casting. The intermetallic microstructure was refined by means of hot straining in the modes presented in Table I.

Flat samples with the working area of 10 mm × 5 mm × 2 mm and 10 mm × 3 mm × 1 mm were tensile strained in an Instron machine in the temperature ranges 900–1050 and 600–900 °C, respectively, at a strain rate of 8.3×10^{-4} s^{-1}. At 1025 °C, the samples were strained in the strain rate interval (1.6–83) $\times 10^{-4}$ s^{-1}.

Samples of diameter 10 and 15 mm high were compression strained in a testing machine 1231U-10 at temperatures ranging from 900–1100 °C at initial strain rates of 1.6×10^{-4}, 8.3×10^{-4}, and 8.3×10^{-3} s^{-1}. Samples with dimensions of 16 mm × 16 mm × 35 mm were compressed in a hydraulic press EU-100 in the temperature interval 700–950 °C at an initial strain rate of 8.3×10^{-4} s^{-1}.

In all cases, the time of removal of the sample from the hot zone never exceeded 1 min.

The tensile diagrams helped to define the following parameters: δ, relative elongation to rupture; σ_{50}, true flow stress at 50% strain; σ_u, conventional ultimate strength. The strain rate sensitivity coefficient m, was evaluated by the inclination of the $\lg\sigma_{50}$–$\lg\dot{\varepsilon}$ (for State 1) and by switching the rates (for State 3).

TABLE I TiAl processing methods

State	Processing method
1	Hot compression straining of compacted TiAl at 1000–1025 °C at an initial strain rate of 10^{-3} s^{-1} and 80% strain
2	As (1), plus 70% restraining under the same conditions changing the compression axis by 90°, but for cast TiAl
3	As (2), plus additional 80% straining at 800 °C at the same rate with another change of compression axis by 90°

TABLE II Dependence of the average size of recrystallized grains, d_{rec}, and specific recrystallized volume, V_{rec}, on the temperature of deformation, t (at $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$) and on the strain rate, $\dot{\varepsilon}$ (at $t = 1025\,°C$), in the TiAl intermetallic compound

Microstructure parameters	t (°C)				$\dot{\varepsilon}$ (s^{-1})		
	950	1000	1050	1100	1.6×10^{-4}	8.3×10^{-4}	8.3×10^{-3}
d_{rec} (μm)	1–2	4.5	10.5	12.5	6	4.5	4
V_{rec} (%)	80	87	90	93	90	87	80

TABLE III Temperature dependence of the TiAl mechanical properties ($\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$)

	t_{def} (°C)							
	750	800	900	950	975	1000	1025	1050
σ_u (MPa)	485	325	195	150	135	120	110	100
δ (%)	45	95	155	175	195	215	250	175

Electron microscopy was performed on a JEM-2000 EX microscope with the accelerating voltage of 200 kV.

The mean grain size of the tensile-strained samples was defined near the rupture site by optical photographs; that of the compression-strained samples was measured in the sample centre from the electron micrographs. The grain size, d, the recrystallized volume, V_{rec}, and the fraction of second phase were measured using the linear intercept method.

3. Results and discussion

The effect of hot compression straining on the microstructure of the coarse-grained TiAl intermetallic compound was studied. The compacted state was used as coarse grained. Its microstructure is characterized by a considerable diversity of grain sizes which is due to the non-uniform distribution of the α_2-phase grains (Ti_3Al-superlattice DO_{19}) size 0.1–3 μm, their volume fraction being 3% or less. "Coarse"-grained and "fine"-grained fractions constitute 80% and 20%, respectively. The grain size of the "fine"-grained fraction is 3–8 μm, while the mean grain size of the "coarse"-grained one is 35 μm. It was established that the development of dynamic recrystallization during the deformation of TiAl, the original microstructure is refined. Table II presents the dependencies of the mean grain size and specific volume of recrystallized grains on the temperature–rate conditions of deformation. It can be seen that as the temperature decreases and the strain rate grows, V_{rec} and d_{rec} remarkably diminish. The latter is in an agreement with a well-known dependence of d_{rec} on the Zener–Hollomon parameter [16]. The reduction of the recrystallized volume correlates with a drop in the TiAl ductility. Thus, during compression at 900 °C and $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$, the maximum strain to cracking is only 30%–40%. Hence, to stimulate recrystallization in the intermetallic compound, its deformability must be substantially increased. This is possible if the original microstructure is refined, for it is known [17] that the ductility of TiAl considerably increases when fine grains are available.

Table III demonstrates the dependence of the fine-grained TiAl tensile properties (State 1) on the temperature of deformation. In spite of the transition to a more rigid scheme of loading, the intermetallic compound exhibits high ductility. The relative elongation grows with the rising temperature of deformation, but upon reaching its maximum value at 1025 °C, it abruptly drops.

The rate dependence of the mechanical properties was studied at temperatures matching the highest values of the relative elongation (Fig. 1a). It can be seen that with the growing strain rate, the relative elongation increases, reaching its maximum ($\delta = 250\%$) at $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$, but decreases with a further growth of the strain rate. The flow stress, σ_{50}, also depends on the strain rate to a great extent and in a certain rate interval its strain-rate sensitivity is rather high. The latter fact is confirmed by a non-monotonic dependence of the coefficient m on strain-rate characteristic for the structural superplasticity of metals [18]. The maximum value of m is 0.43 (Fig. 1b). The interval of the highest values of m is matched with the largest values of δ. Under these conditions, the samples are uniformly elongated to rupture. Fig. 2 shows stress–strain curves corresponding to different strain rates at 1025 °C. On the whole, they are quite similar to those conventionally observed in different regions of superplasticity [18]. Hence, in State 1, the TiAl intermetallic compound exhibits superplastic features.

The metallographic analysis of the deformed samples revealed the peculiarities of the influence of the temperature–rate conditions of deformation on the microstructural changes in the intermetallic compound. Table IV presents the strain-rate dependence of the mean grain size in the sample working area and head at 1025 °C. It can be seen that as the strain rate increases compared with a certain optimal one, the microstructure is rapidly refined and, vice versa, decreasing strain rate entails the growth of grains, the

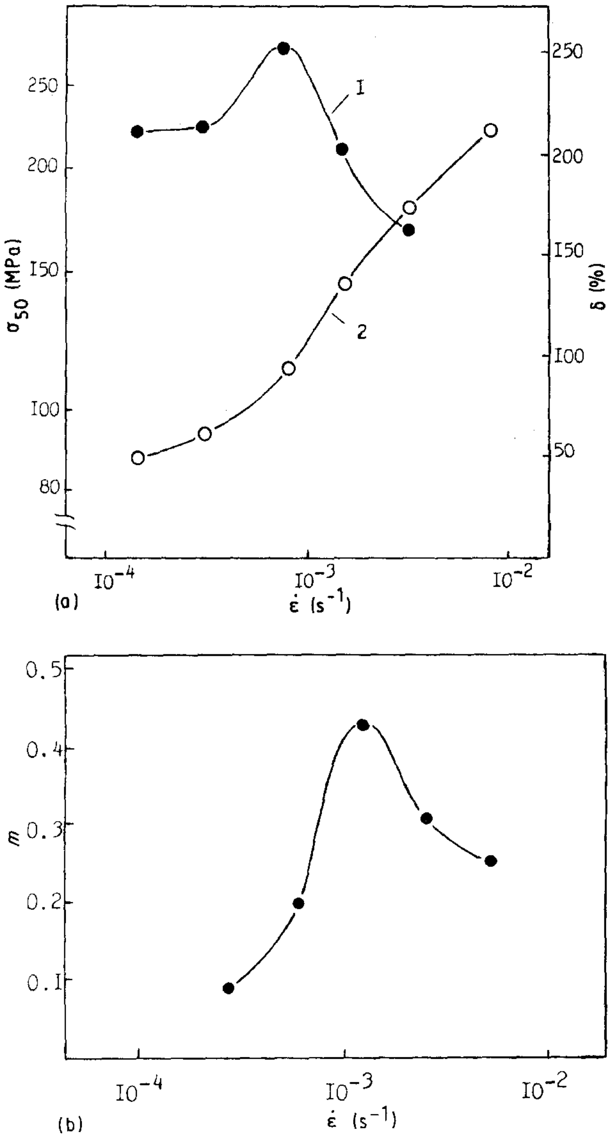


Figure 1 Strain-rate dependence rate of (a) the relative elongation to rupture, δ (●), and true flow stress, σ_{50} (○), and (b) strain-rate sensitivity of flow stress m, in State 1 TiAl at 1025 °C.

mean grain size of the sample head being independent of the testing time.

Deformation at an optimal superplasticity strain rate remarkably transforms the original microstructure of the intermetallic compound (Fig. 3a, b). It becomes much more homogeneous. A comparison of the histograms of the distribution of grain sizes in the working area in the sample strained 160% and in that of the original sample (heated and exposed at the test temperature for 30 min), indicates that the microstructure homogeneity increases due to the simultaneous growth of "fine" grains and refinement of

TABLE IV Strain-rate dependence of the average grain size, d, of the TiAl intermetallic compound in the sample head and working area

Microstructure parameters	$\dot{\varepsilon}$ (s^{-1})		
	1.6×10^{-4}	8.3×10^{-4}	8.3×10^{-3}
d (μm): head	8	8	8
working area	11	7.5	4.5

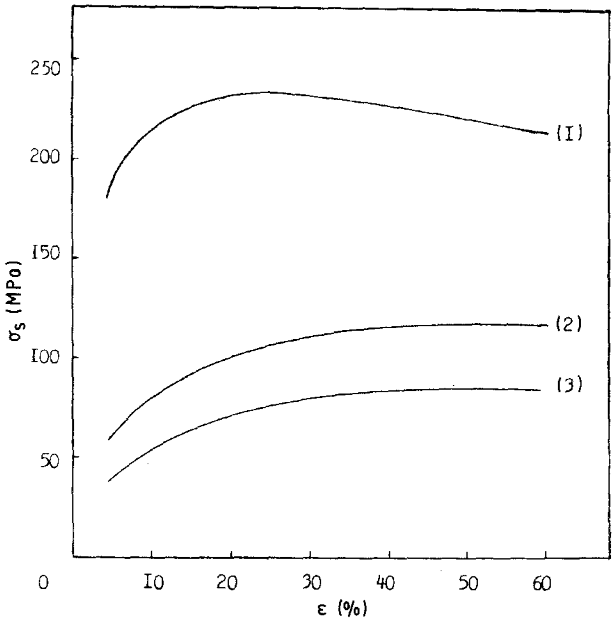


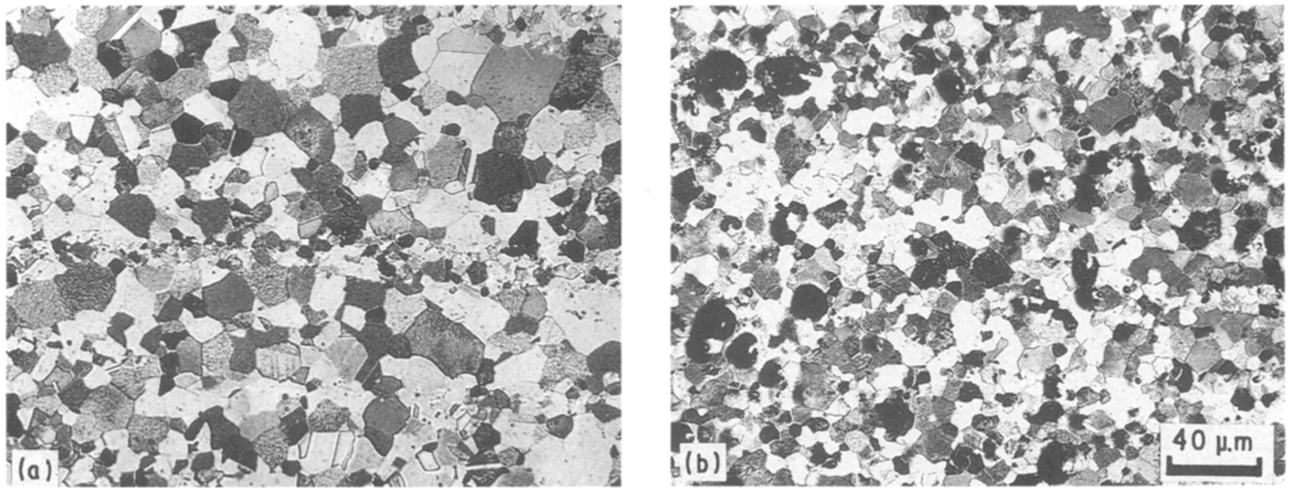
Figure 2 Stress–strain curves of TiAl in State 1 at 1025 °C and different strain rates: (1) $\dot{\varepsilon} = 8.34\times10^{-3}\ s^{-1}$, (2) $\dot{\varepsilon} = 8.3\times10^{-4}\ s^{-1}$, (3) $\dot{\varepsilon} = 1.6\times10^{-4}\ s^{-1}$.

"coarse" ones (Fig. 3c). The distribution of the second, i.e. α_2-phase (superlattice DO_{19}), becomes more homogeneous, its fraction being no larger than 3 vol %. The α_2-phase particles sized 0.1–3 μm are mainly of a spherical shape and can be found both in the boundaries and within the grains of TiAl.

Thus, at 1025 °C and $\dot{\varepsilon} = 8.3\times10^{-4}\ s^{-1}$, the following two processes are concurrently in progress in TiAl during its deformation: the growth of "fine" grains and simultaneous refinement of "coarse" ones, both the processes being in relative equilibrium. As the temperature–rate conditions change, the equilibrium probably shifts to one side or the other. This condition is indispensable for a further refinement of structure. From the above observations, let us proceed to consider microstructural changes occurring in TiAl during the deformation at temperatures below those at which the superplastic features are displayed. The tests were performed on samples in State 2 prepared by superplastic treatment which yielded a homogeneous fine-grained microstructure (d = 5 μm). Samples with such microstructure were compression strained in the temperature range 700–950 °C. In this case, too, the intermetallic compound contains about 3 vol % α_2-phase shaped as spherical particles of size 0.1–3 μm.

Fig. 4 shows stress–strain curves obtained at different test temperatures. The flow stress decreases with the rising temperature. In the studied temperature range, a peak of flow stress is observed at the initial stage. As the temperature decreases, this peak shifts towards higher strain levels. With further straining, the flow stress monotonically decreases and the steady flow stage is then observed.

Analysis of the 80% strained samples at 700–950 °C indicates that due to dynamic recrystallization, "new" grains are formed in the intermetallic compound, their size being substantially smaller than in the original state (Fig. 5a, b). As the temperature rises, the mean



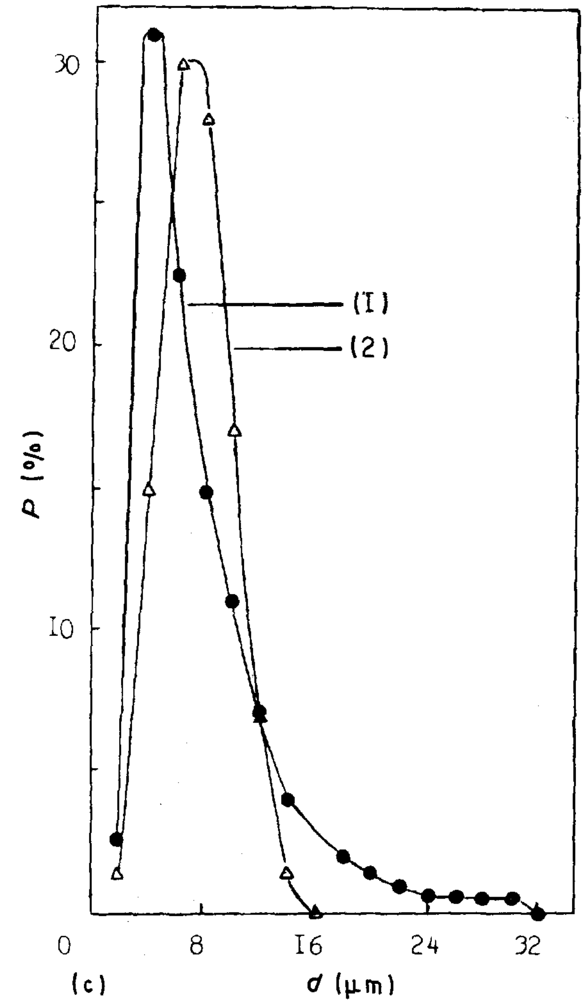


Figure 3 Microstructure and histograms of grain-size distribution in State 1 TiAl, 160% strained at 1025 °C and $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$: (a) head; (b) working area; (c) histograms, (1) head, (2) working area.

grain size and specific volume of the recrystallized grains increases (Table V). Comparing Tables II and V, it can be seen that at a deformation temperature of 950 °C, the specific volume of the recrystallized grains in the case of preliminary refined microstructure is remarkably larger than one in the coarse-grained samples.

To study the peculiarities of the low-temperature dynamic recrystallization developing in TiAl, an evolution of microstructure in the course of deformation at 800 °C was investigated. The initial stage of plastic flow ($\varepsilon = 10\%$) is characterized by the rapid accumulation of dislocations, development of mechanical twinning in many grains, and local migration of original grain boundaries (Fig. 6a, b). At this stage of flow, subgrains have already been formed near the original boundaries (Fig. 6a) and subboundaries appear in the intertwin space (Fig. 6b). After 40% strain, recrystallized grains are found in the microstructure (Fig. 6c); the twin boundaries bulge and the twins themselves become lens-shaped (Fig. 6d). New grains are probably formed due to (a) the increasing misorientation of subgrain boundaries up to high-angle, (b) fragmentation of intertwin space and rearrangement of the fragments' twin boundaries into random high-angle grain boundaries (Fig. 6d), (c) appearance of "necking" in the "bulging" regions of the boundary. On the whole, the mechanisms of the recrystallized grain formation are similar to those described previously [15] where the TiAl dynamic recrystallization in a coarse-grained state was reported.

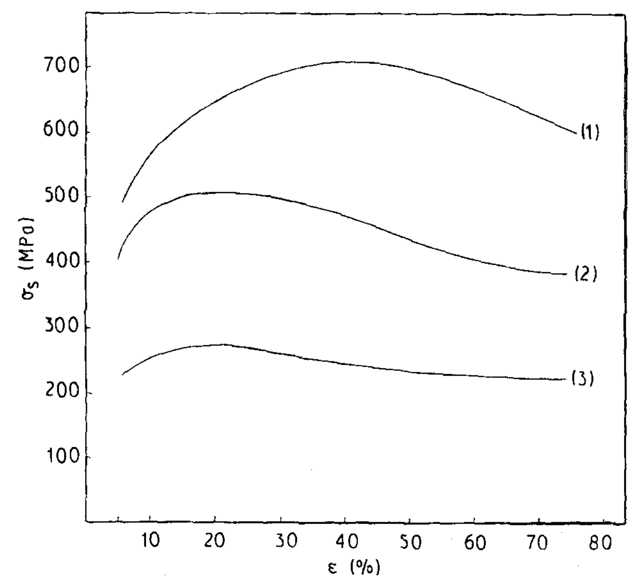


Figure 4 Stress–strain curves of State 2 TiAl at 700–900 °C and $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$.

TABLE V Temperature dependence of the average size of recrystallized grains d_{rec}, and specific recrystallized volume, V_{rec}, at $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$ in the TiAl intermetallic compound

Microstructure parameters	t (°C)					
	700	750	800	850	900	950
d_{rec} (μm)	0.13	0.25	0.45	0.75	1.2	1.9
V_{rec} (%)	56	70	78	84	90	96

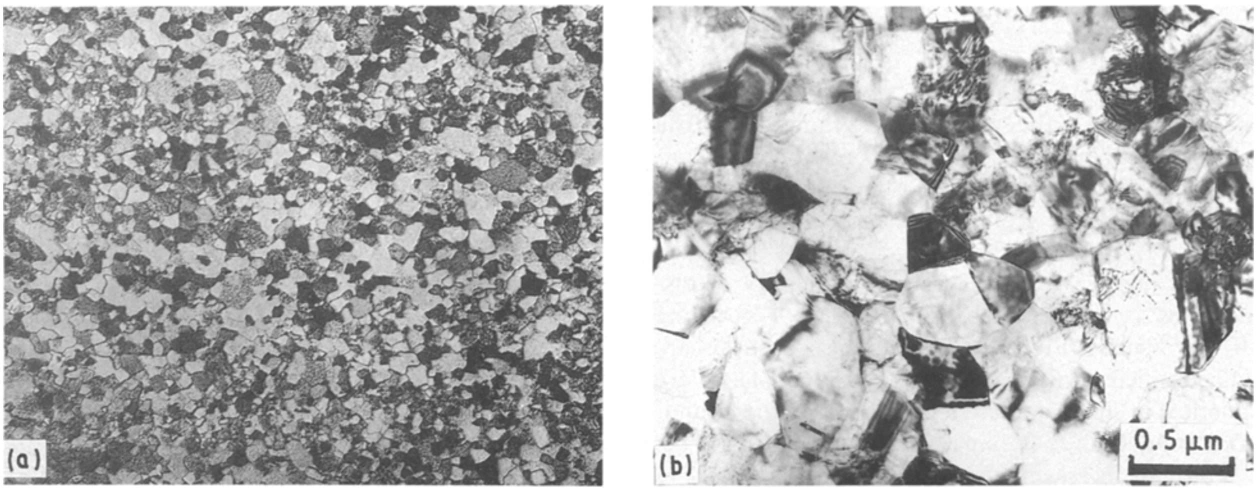


Figure 5 Microstructure of State 2 TiAl (a) before deformation and (b) after 80% strain at 800 °C and $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$.

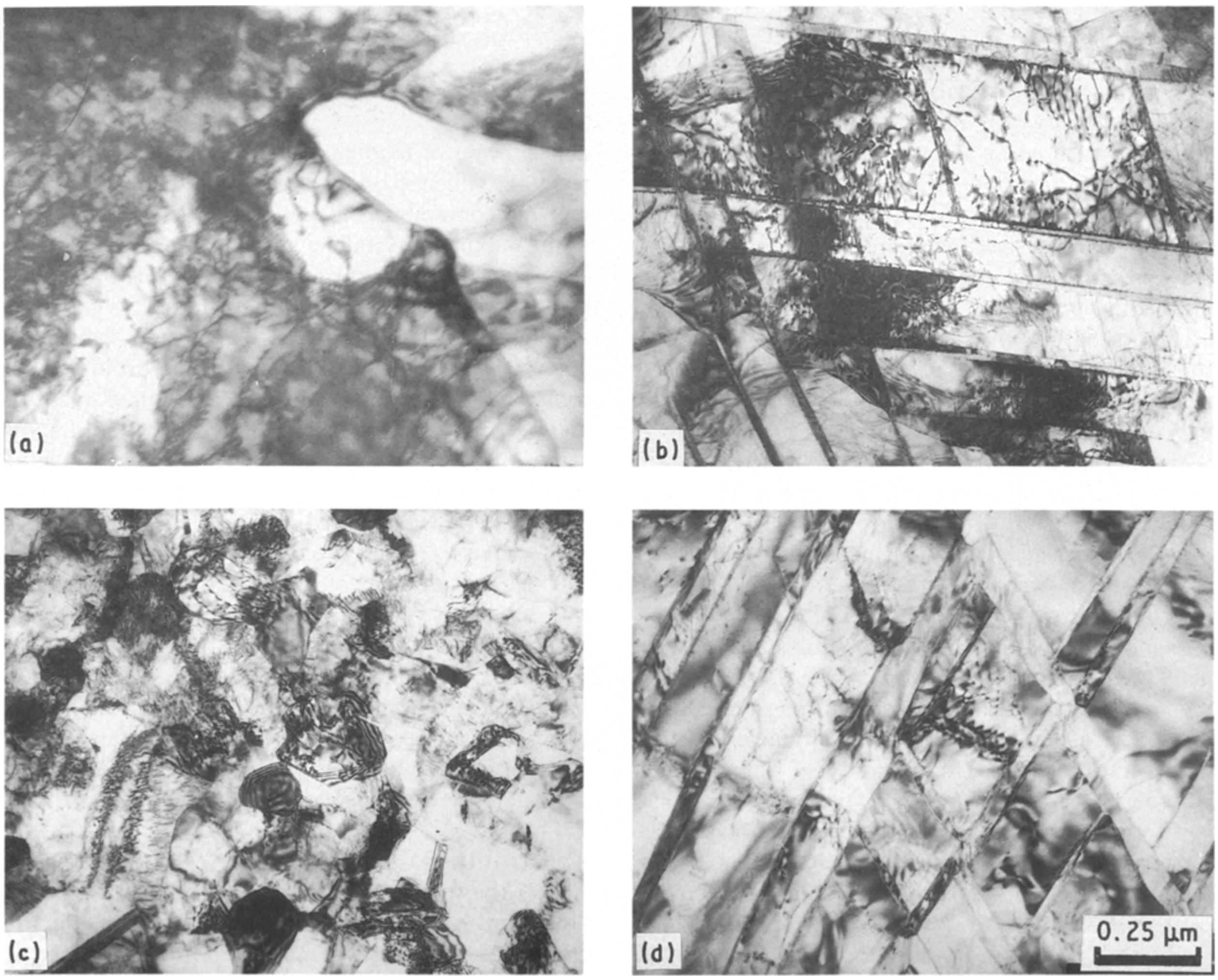


Figure 6 Evolution of State 2 TiAl microstructure during deformation at 800 °C and $\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$: (a, b) $\varepsilon = 10\%$; (c, d) $\varepsilon = 40\%$.

TABLE VI Temperature dependence of the TiAl mechanical properties ($\dot{\varepsilon} = 8.3 \times 10^{-4}\ s^{-1}$)

	t_{def} (°C)					
	600	650	700	800	850	900
δ (%)	13.3	75	130	225	260	220
σ_u (MPa)	960	860	730	225	120	120
$\sigma_{0.2}$ (MPa)	840	730	570	140	100	95

As the strain level increases ($\varepsilon = 40\%$–80%), the specific volume of the recrystallized grains grows. The grains are of equiaxial shape and contain no mechanical twins. In the majority of "fine" and "average"-sized recrystallized grains, the density of dislocations is rather low ($\rho = 10^8$–$5 \times 10^8\ cm^{-2}$). At the same time, in relatively coarse grains, the density of dislocations is remarkably higher, reaching 10^9–$10^{10}\ cm^{-2}$ (Fig. 5b). The α_2-phase particles are refined, their size becoming 0.1–0.3 μm. They are spherical in shape and are located both in the boundaries and within the grains.

Thus, the increasing flow stress at the initial stage of deformation at 700–950 °C is probably associated with defects accumulated in the intermetallic compound, i.e. dislocations and mechanical twins. The decrease of the flow stress during further deformation is due to the development of dynamic recrystallization. The attainment of the steady flow stage probably indicates a transition from dynamic recrystallization to superplastic flow. This is also confirmed by the reduced density of dislocations in "fine"- and "average"-sized grains after deformation. It should be noted that at a certain stage of hot deformation, this transition occurs in many materials [19–21].

Thus, in a massive TiAl semifinished product, the submicrocrystalline state can be realized in the following three stages:

1. deformation under the temperature–rate conditions of superplasticity resulting in the refinement of the original cast structure due to dynamic recrystallization;
2. superplastic treatment yielding a homogeneous microcrystalline structure with a mean grain size of about 5 μm;
3. the increasing ductility enables deformation at temperatures lower than in Stages 1 and 2, providing for dynamic recrystallization.

As indicated by the experiment, each stage of treatment requires a strict observation of certain temperature–rate conditions of deformation. This is due to the fact that during deformation at elevated temperatures in TiAl, the growth of "fine" grains can coexist with refinement of "coarse" grains. The coexistence of these processes under superplastic deformation, provides for the homogenization of the microstructure. This is why at the final stage of treatment, at lower temperatures, such temperature–rate conditions of deformation must be selected that could induce superplasticity in the intermetallic compound at the final stage of flow. The utilization of a favourable scheme of loading is another factor exerting a positive influence on the deformability of the intermetallic compound and the homogeneity of the resulting submicrocrystalline structure.

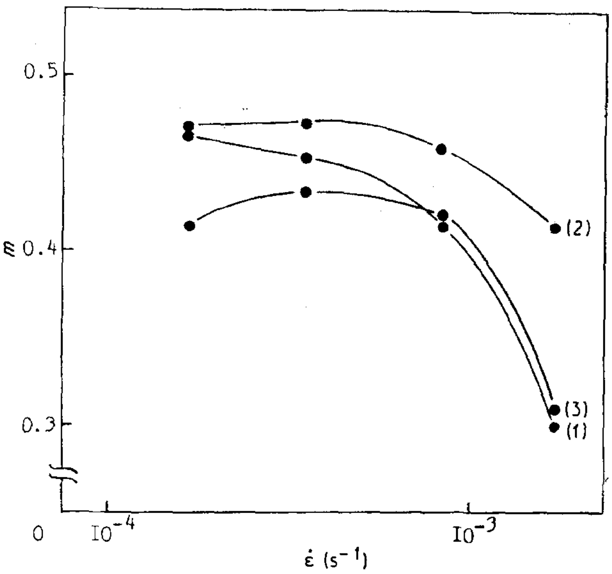


Figure 7 Strain-rate dependence of the strain-rate sensitivity coefficient, *m*, of State 3 TiAl at (1) 800 °C, (2) 850 °C, and (3) 900 °C.

Table VI presents the mechanical tensile properties of submicrocrystalline TiAl (State 3). In this case, the average grain size is 0.4 μm. Comparing Tables III and VI, it can be seen that the transition from State 1 to State 3 results in a considerable growth of ductility and at 800 °C, the strain rate sensitivity is already rather high ($m > 0.3$) (Fig. 7). The maximum value of m is attained at a test temperature of 850 °C ($m = 0.47$). The increased values of m are matched with the largest relative elongations.

Thus, the creation of a submicrocrystalline structure in massive TiAl semifinished products substantially decreases the lower temperature limit of superplasticity. This fact supports the use of the above method for processing the intermetallic compound under study. The method is probably also applicable to other hard-to-deform materials.

4. Conclusion

The TiAl intermetallic compound was used to elaborate a universal method of obtaining a submicrocrystalline structure in semifinished products made of hard-to-deform materials. It comprises the following stages:

1. thermomechanical treatment yielding a fine-grained microstructure;

2. superplastic treatment improving the microstructural homogeneity;

3. final thermomechanical treatment at temperatures lower than in Stages 1 and 2 providing for the formation of a submicrocrystalline structure. In this case, the temperature–rate conditions must ensure the development of superplasticity in the final stage of plastic flow.

The creation of the submicrocrystalline structure results in a considerable broadening of the temperature range where superplasticity can be displayed in the TiAl intermetallic compound.

References

1. J. RUPP, and R. BIRRINGER, *Phys. Rev.* **B.36** (1987) 7888.
2. H. GLEITER, *J. Phys.* **C4** (1985) 393.
3. H. SUGIMOTO, K. AMEYAMA, T. INABE, M. TOKIZANE, *J. Jpn Inst. Metals* **53** (1989) 628.
4. R. S. VALIEV, O. A. KAIBYSHEV, R. I. KUZNETSOV, R. SH. MUSALIMOV and N. K. TSENEV, *Dokl. Akad. Nauk SSSR (DAN)* **301** (1988) 864.
5. YU. V. EFIMOV, R. M. GALEYEV and G. A. SALISHCHEV, in "Metastable and Nonequilibrium Alloys", edited by Yu. V. Efimov (Metallurgia, Moscow, 1988) p. 383.
6. O. R. VALIAKHMETOV, R. M. GALEYEV and G. A. SALISHCHEV, *Phiz. Met. Metalloved.* **10** (1990) 204.
7. V. I. NOVIKOV, L. I. TRUSOV, V. N. LAPOVOK, A. I. KAPUSTIN, S. V. SVIRIDA and E. N. YAKOVLEV, *Poroshk. Metally. (Powder Metallurgu)* **9** (1985) 84.
8. A. H. CHOKSHI, A. ROSEN, J. KARCH and H. GLEITER, *Scripta Metall.* **23** (1989) 1679.
9. F. HESSNER (ed.), "Recrystallization of Metallic Materials" (Metallurgia, Moscow, 1982) p. 352.
10. R. M. GALEYEV, O. R. VALIAKHMETOV, M. I. MAZURSKII and G. A. SALISHCHEV, in "Proceedings of the 12th All-Union Conference on Physical Strength and Ductility of Metals and Alloys", Kuibyshev 1989, edited by A. N. Bekrenev (Polytechnic Institute, Kuibyshev, 1989) p. 252.
11. M. UEKI, M. HATTORI, S. HORIE and T. NAKAMURA, *Trans. JIM* **26** (1986) 906.
12. J. B. McANDREW and H. D. KESSLER, *Trans. AIME* **206** (1956) 1348.
13. T. KAWABATA, M. TADANO and O. IZUMI, *Scripta Metall.* **22** (1988) 1725.
14. O. A. KAIBYSHEV, S. G. GLAZUNOV, G. A. SALISHCHEV, R. M. IMAYEV and V. I. IVANOV, *Phiz. Met. Metalloved.* **64** (1987) 1005.
15. G. A. SALISHCHEV, R. M. IMAYEV, A. B. NOTKIN and D. V. ELAGIN, *Tzvetnye Metally (Non-ferrous Metals)* **7** (1988) 95.
16. J. P. POIRIER, "High-temperature Ductility of Crystalline Solids" (Metallurgia, Moscow, 1982) p. 272.
17. O. A. KAIBYSHEV, G. A. SALISHCHEV, R. M. IMAYEV and A. B. NOTKIN, *Phiz. Met. Metalloved.* **65** (1988) 998.
18. O. A. KAIBYSHEV, in "Superplasticity of Commercial Alloys" (Metallurgia, Moscow, 1984) p. 263.
19. N. G. ZARIPOV, A. R. VAGAPOV and R. O. KAIBYSHEV, *Phiz. Met. Metalloved.* **63** (1987) 774.
20. O. A. KAIBYSHEV, R. M. IMAYEV and M. F. IMAYEV, *Dokl. Akad. Nauk SSSR (DAN)* **305** (1989) 1120.
21. O. A. KAIBYSHEV, in "Proceedings of an International Conference on Superplasticity and Superplastic Forming", Washington, 1988, edited by C. H. Hamilton and N. E. Paton (Minerals, Metals and Materials Soc. Warrendale, PA, 1988) p. 3.

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