

# Mechanical Properties of Ti–6Al–4V Titanium Alloy with Submicrocrystalline Structure Produced by Severe Plastic Deformation

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A comparative investigation of mechanical properties of Ti–6Al–4V titanium alloy with microcrystalline and submicrocrystalline structures in the temperature range of 20–600°C has been carried out. The grain sizes under the submicrocrystalline and microcrystalline conditions are 0.4 and 10 µm, respectively. The alloy with the microcrystalline structure has been additionally subjected to a heat-strengthened treatment. The structure refinement of the alloy results in increase in both strength and fatigue limit at room temperature by about 20%. With increasing deformation temperature, the strength of the submicrocrystalline alloy is higher than that of the microcrystalline alloy up to 400°C. However, the creep strength of the submicrocrystalline alloy is slightly lower than that of the heat-strengthened microcrystalline alloy already at 250°C.

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## 1. Introduction

Submicrocrystalline (SMC) materials having a grain size of 0.1–0.5 µm range, that offer improved mechanical properties, have recently gained a great interest among researches in the materials science.<sup>1)</sup> Microstructure refinement of metals and alloys into the SMC regime considerably increases strength and decreases the temperature at which superplasticity occurs by hundreds degrees, compared to their larger-grained counterparts.<sup>1–4)</sup>

Industrial applications of the SMC materials have not been found yet, because the production of SMC billets with quite large dimensions involves difficult scientific and engineering problems. However, some severe plastic deformation (SPD) techniques have been developed recently for the production of SMC structure in samples with a size of tens to hundreds of millimeters.<sup>1,5,6)</sup> The equal-channel angular extrusion (ECAE)<sup>1)</sup> is a technique for producing SMC structure in ductile and soft enough metals and alloys; for instance, aluminum, aluminum alloys and pure titanium. Massive samples of hard-to-deform alloys, including titanium alloys and steel, with SMC structure can be produced by means of subsequent work consisted of sequential deformation along three orthogonal directions, which is defined as the “abc” deformation method.<sup>5–7)</sup>

Another reason why SMC metals and alloys are not used as engineering materials arises from the issue described above. The limited dimension of specimens is an impediment to extracting “valid” mechanical properties of SMC materials produced by some other processing methods. Owing to a very small size of samples, most mechanical properties of SMC materials have been primarily derived from uniaxial tension/compression tests and micro- or nano-indentation.<sup>2)</sup> This is the reason why data for the mechanical properties of SMC materials are rather incomplete. There are plenty of data concerning microhardness, strength and superplasticity of SMC materials, which shows that very fine-grained materials possess outstanding properties. However, not enough infor-

mation is presently available in literature on the fatigue, ductile, high-temperature strength and creep resistance of SMC materials.<sup>2)</sup>

The development of the method for producing massive samples with SMC structures allows us to study their mechanical properties more systematically by using standard samples along with standard procedures. The aim of the present paper is to evaluate the mechanical properties of the SMC Ti–6Al–4V alloy both at room and high temperatures in comparison with those of the microcrystalline (MC, grain dimension of some micrometers) heat-strengthened counterpart.

## 2. Experimental

The material used in this investigation is  $\alpha/\beta$  titanium alloy Ti–6Al–4V with the nominal chemical composition of 6.3 Al, 4.1 V, 0.18 Fe, 0.182 O, 0.03 Si in mass percent and balance titanium. The alloy has a  $\beta$  transition temperature (at which  $\alpha + \beta \rightarrow \beta$ ) of 995°C. The material was received in the form of a hot rolled cylindrical rod with diameter of 30 mm. Samples measuring 30 mm in diameter and 50 mm in length were cut from the as-received bar for the next processing.

Samples with SMC structure have been prepared by means of subsequent work consisted of sequential deformation of a sample along three orthogonal directions, defined as the “abc” deformation method (Fig. 1).<sup>5,6)</sup> The grain size produced by the method depends on temperature during the deformation.<sup>3)</sup> Therefore, the deformation temperature should be reduced as much as possible to have the smallest grain size. On the other hand, ductility of the alloy at low temperature is limited whereas a large deformation is required to produce samples with the SMC structure.<sup>6)</sup> Accordingly, the process should be started from a relatively high temperature of about 700°C to avoid fracture of the sample. As ductility of the sample is improved by the first deformation, the deformation temperature in the next stage

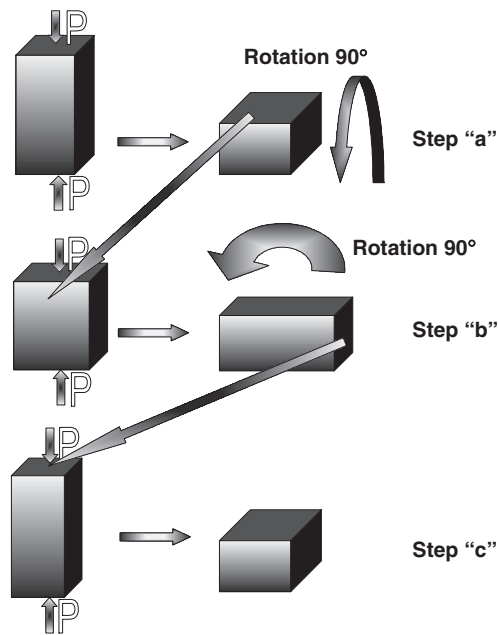


Fig. 1 Scheme of “abc” deformation.

can be decreased by 50–100°C. The final temperature of the “abc” deformation was 550°C. To attain a homogeneous fine-grained microstructure in a sample at least six or seven sequential compressions (conducting steps “a” through “c” in Fig. 1 two times, as “a-b-c-a-b-c”) at each temperature are required.<sup>6)</sup>

The deformation at each temperature was carried out under an isothermal condition at a strain rate of about  $10^{-3} \text{ s}^{-1}$ . After the “abc” deformation, the Ti-6Al-4V samples with SMC structure were annealed at 650°C for an hour to reduce internal stresses. Optimum annealing conditions to attain the maximum structure improvement without grain growth were determined from pilot experiments.

In this study mechanical properties of the SMC alloy are compared with those of the MC alloy. A homogeneous MC structure of the Ti-6Al-4V alloy was produced by means of the “abc” deformation process conducted at a higher temperature of about 930°C.<sup>8)</sup> In this case the temperature of deformation was not changed during the processing. After that, the MC alloy was subjected to a heat-strengthening treatment through water quenching from 945°C and then aging at 500°C for 3 h. According to<sup>8)</sup> the alloy has almost maximum strength and fatigue limit after such treatment.

The samples for mechanical tests were cut out from billets of  $10 \times 40 \times 65 \text{ mm}^3$  with SMC and MC structures. The tensile tests using cylindrical samples with of 3 mm diameter and 18 mm length were conducted at temperatures of 20–600°C at a speed of 1 mm/min. Tensile tests at room temperature were carried out on a “Schenk” machine using an extensometer for a precise strain measurement. Tests at high temperatures were conducted on an “Instron” machine in air. Mechanical properties including yield stress (YS), ultimate tensile stress (UTS), area reduction (AR), total elongation (TE), uniform elongation (UE) were determined according to the Russian government standard GOST 1497-84. Specific work of deformation was calculated as an area

under the corresponding true stress–true area reduction curve. Since plastic flow localizes very fast during the tensile test (see section 3.2), use  $\sigma$ – $\phi$  curve, rather than  $\sigma$ – $\varepsilon$ , seems to be more suitable for determination specific work of deformation. Because, in this case, the  $\sigma$ – $\phi$  curves show real ability of the alloys to plastic deformation. However specific work of deformation has been estimated not for the whole sample, but for a small sample portion, which was really deformed during the tensile test.

A stress-controlled bending fatigue test was carried out to evaluate a fatigue limit (FL) of the SMC and MC alloys. Specimens with 8 mm gage width and 2 mm thick were tested for  $2 \times 10^7$  loading cycles. Ten specimens of each condition were utilized for the fatigue test. The fatigue test was conducted at room temperature in air. The stress was prescribed as a sinusoidal pulse at the frequency of 500 Hz. The load ratio, which is defined as the ratio of the minimum load to the maximum load, was equal to 0.

A creep test of the alloy in SMC and MC conditions was carried out using a creep machine of type 2147P-30/1000 at temperatures of 250 and 350°C in air. Cylindrical tensile specimens with gage dimensions of 5 mm diameter and 18 mm length were used in accordance with the Russian government standard GOST 3248-81. Creep strength was evaluated as the stress required to attain 0.2% strain for 100 h at those temperatures.

The microstructure was investigated using a JEM-840 SEM and a JEM-2000 EX TEM. X-ray investigations were conducted on a DRON-3 diffractometer with Cu- $K_\alpha$  irradiation.

### 3. Results

#### 3.1 Microstructure

The structure of the Ti-6Al-4V alloy before the “abs” deformation consists of primary  $\beta$ -grains with Widmanstätten colonies of  $\alpha$ -lamellae and thin  $\beta$ -laths oriented in different directions [Fig. 2(a)]. As a result of the “abc” deformation, the lamellar structure transforms into globular one, consisting of grains of  $\alpha$ - and  $\beta$ -phases<sup>6)</sup> with a mean grain size of about  $0.4 \mu\text{m}$  [Fig. 2(b)]. Non-homogeneous diffraction contrast and high dislocation density inside some grains in the microstructure of alloys indicate a high level of internal stresses and elastic distortions of the crystal lattice. In the MC condition the Ti-6Al-4V alloy has a bi-modal structure composed of primary  $\alpha$ -grains with a mean size of  $5 \mu\text{m}$  and colonies of very disperse  $\alpha$ - and  $\beta$ -lamellae within transformed  $\beta$ -grains [Fig. 2(c)]. X-ray analysis have shown that the volume fractions of  $\beta$ -phase for both SMC and MC conditions were 8–10%.

The analysis of  $\alpha$ -phase inverse pole figures has revealed a quite weak texture intensity and similar texture distribution for both SMC and MC conditions of the alloy (Fig. 3). When the small percentage of  $\beta$ -phase is taken into account, the effect of texture on the mechanical properties can be neglected in both conditions.

#### 3.2 Mechanical behavior at room temperature

Mechanical properties of the SMC and MC alloys at room temperature are presented in the Fig. 4 and Table 1. Grain

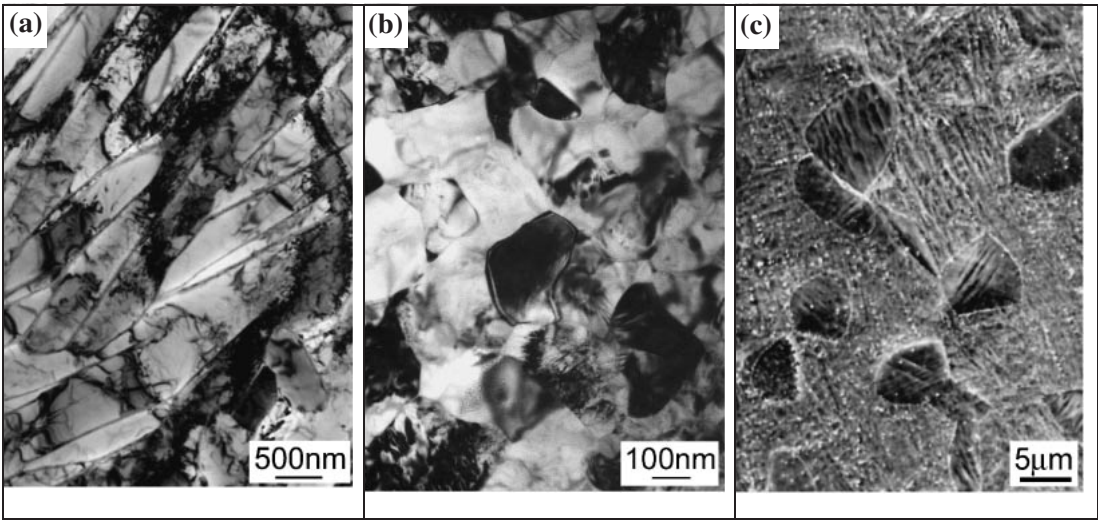


Fig. 2 Microstructure of Ti-6Al-4V alloy: (a) initial lamellar, (b) SMC and (c) MC conditions.

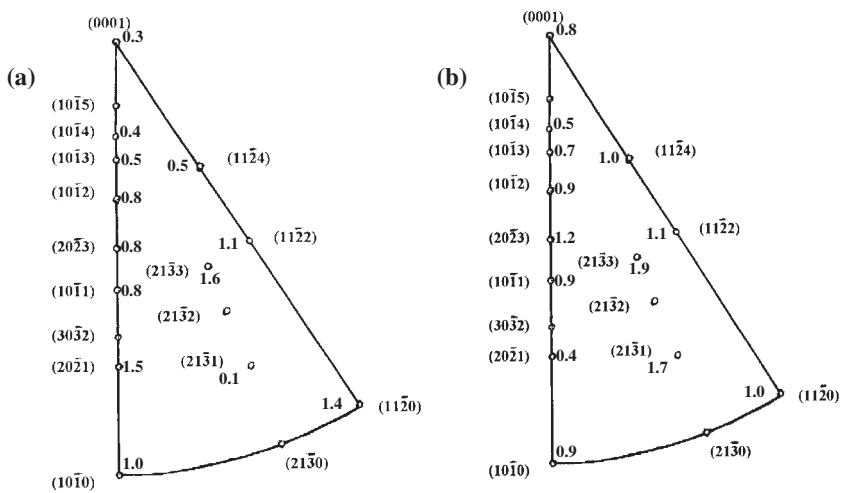


Fig. 3 Inverse pole figures of (a) SMC and (b) MC conditions of Ti-6Al-4V alloy.

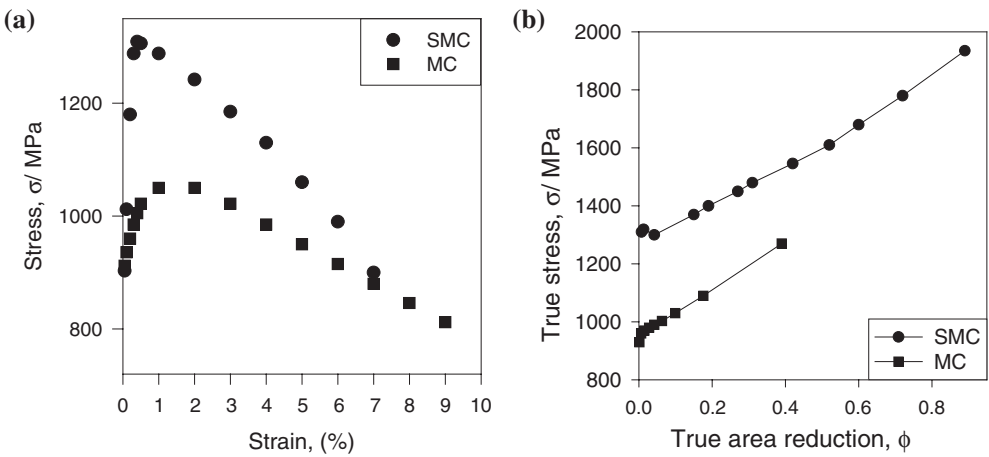


Fig. 4 Tensile test of SMC and MC conditions of Ti-6Al-4V alloy at room temperature; (a) stress–strain curves and (b) true stress–true area reduction curves.

Table 1 Mechanical properties of Ti-6Al-4V alloy at room temperature.

Condition	YS (MPa)	UTS (MPa)	AR (%)	TE (%)	UE (%)	FL (MPa)
SMC	1180	1300	60	7	0.5	693
MC	960	1050	32	9	0.9	580

YS: yield stress; UTS: ultimate tensile strength; AR: area reduction; TE: total elongation; UE: uniform elongation; FL: fatigue limit

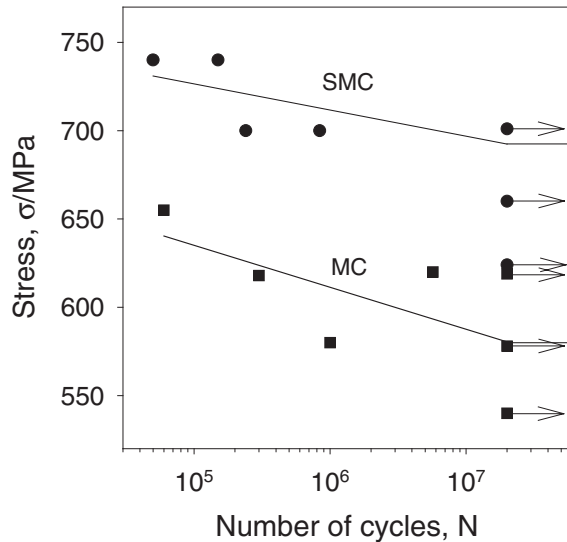


Fig. 5 Fatigue curves of SMC and MC conditions of Ti-6Al-4V alloy.

refinement in the SMC regime results in a noticeable enhancement of the tensile strength, compared with that of the MC heat-strengthened alloy. Both yield stress and ultimate tensile strength of the SMC alloy are around 22% higher than those of the MC alloy. Total elongation is rather similar between the SMC and MC alloys, whereas true area reduction of the SMC alloy is a factor of two higher than that of the MC alloy. As a result, the SMC alloy has a larger specific work of deformation to failure, 1395 kJ/m<sup>3</sup>, compared to 435 kJ/m<sup>3</sup> of the MC alloy.

Early necking took place in both alloys; the values of the uniform elongations are lower than 1% [Table 1, Fig. 4(a)]. At the same time uniform elongation of the MC alloy is almost a factor of two higher than that of the SMC alloy (Table 1). Therefore deformation in the neck portion prior to failure was severer in the sample with SMC structure than that of the MC alloy [Fig. 4(b)].

Fatigue strength of the SMC alloy is also higher than that of the MC alloy (Fig. 5 and Table 1). The SMC alloy have fatigue limit about 20% higher than that of the MC alloy (690 and 580 MPa respectively).

However it should be noted in this section, that fatigue limit and total elongation of the heat-strengthened MC alloy used in the present work are lower than they could be expected.<sup>9,10</sup> It is generally known that mechanical properties of alloys depend not only on their microstructure (or applied processing) but also on chemical compositions. Hence, the discrepancy of mechanical properties between the present results and some reported data<sup>9,10</sup> is most likely associated with admissible variations in chemical composi-

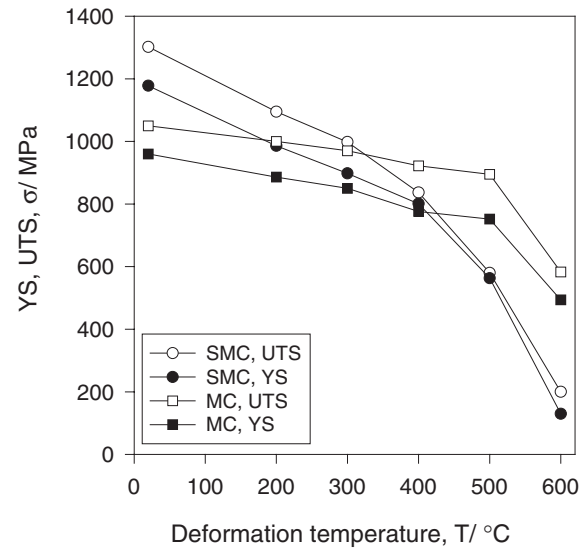


Fig. 6 Effect of temperature on yield stress (YS) and ultimate tensile stress (UTS) of SMC and MC conditions of Ti-6Al-4V alloy.

Table 2 Effect of temperature on area reduction (AR), total elongation (TE) and uniform elongation (UE) under different conditions of Ti-6Al-4V alloy.

Temp. (°C)	SMC			MC		
	AR (%)	TE (%)	UE (%)	AR (%)	TE (%)	UE (%)
20	60	7	0.6	32	9	0.8
200	69	9	0.7	67	13	1.6
300	70	10	1.2	71	16	2
400	83	14	3.5	77	17	3.2
500	97	42	10.0	87	22	4.2
600	—	200	—	—	46	—

tions. However, as both SMC and MC alloys have the same chemical composition, the data obtained allow us to evaluate a relative effect of grain refinement on mechanical properties of the Ti-6Al-4V alloy.

### 3.3 Mechanical behaviors at high temperatures

The effect of temperature on tensile strength of the SMC and MC alloys is shown in Fig. 6. With increasing temperature, both YS and UTS of the SMC alloy decrease faster than those of the MC alloy. Yield stresses of the SMC and MC alloys become nearly equal at 400°C, while the equalization of ultimate tensile stress occurs at about 300°C (Fig. 6). The most drastic softening of the alloys takes place above 400°C for SMC structure and above 500°C for MC structure.

Table 2 demonstrates the effect of temperature on ductility of the SMC and MC alloys. Total elongation at room temperature is similar and linearly increases with temperature up to 400°C for both alloy conditions. The increase of temperature to 600°C leads to a drastic increase by 200% in total elongation of the SMC alloy, while the total elongation of the MC alloy at this temperature is less than 50%.

Area reduction of the SMC and MC alloys is not the same at room temperature but similar at 200°C and above.



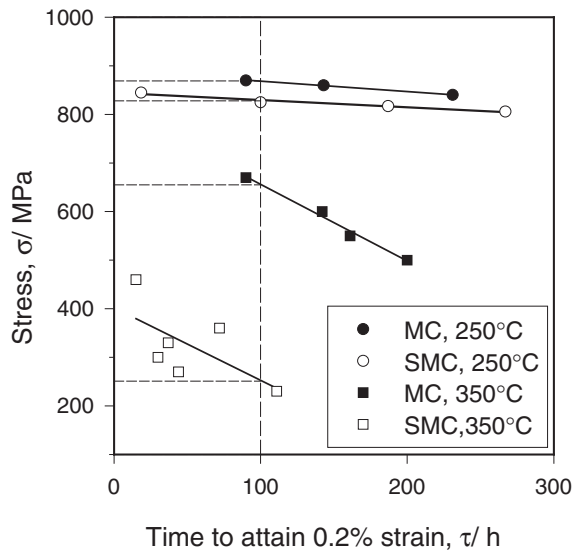


Fig. 7 Effect of temperature on stress and time requirements for attaining of 0.2% strain in SMC and MC conditions of Ti-6Al-4V alloy.

As a whole, uniform elongation of both alloys slowly increases with increasing temperature, but it increases faster at temperatures above 300°C. As a result, a uniform elongation of the SMC alloy is a factor of two higher than that of the MC alloy at 500°C.

Figure 7 shows creep resistance behaviors of the SMC and MC alloys in terms of stress-time to attain a strain of 0.2% at temperatures of 250 and 350°C. The creep resistance in the MC condition is slightly higher than that in the SMC condition at 250°C, but at 350°C the difference between the alloys increases significantly: 830 and 870 MPa at 250°C in the SMC and MC conditions, respectively (the difference is 5%), whereas 250 and 655 MPa, respectively at 350°C (the difference is 60%). It should also be noted that a broad scatter can be seen in the experimental data of the SMC alloy at 350°C while the other points are well approximated by straight lines.

#### 4. Discussion

The results of mechanical tests at room temperature indicate that strength, area reduction, specific work of deformation to failure and fatigue limit are increased by the structure refinement. Strengthening with grain refinement has been traditionally rationalized on the basis of the so-called Hall-Petch mechanism.<sup>11)</sup> In the present case, the SMC and MC conditions of the alloy differ from each other in terms of size and morphology of phase constituents as well as dislocation density within the phases. In fact, the thickness of  $\alpha$ - and  $\beta$ -laths in the MC alloy is comparable to the grain size in the SMC alloy. However, due to the Burgers orientation relationship between  $\alpha$ - and  $\beta$ -phases in a lath colony in the MC alloy behaves as a single grain even at large strains.<sup>12)</sup> Hence, the SMC alloy having both smaller grain size and higher dislocation density within the grain volume indicates higher strength compared to the MC alloy (Fig. 4).

Relationship between grain size and fatigue limit for titanium alloys can also be described by an equation similar

to the Hall-Patch law.<sup>13)</sup> Therefore, the structure refinement leads to the improvement of fatigue property, as well. This is confirmed by the results obtained in the present work as well as in other papers.<sup>1,2,14)</sup> Commonly, fracture energy is a sum of crack initiation and crack propagation. Structure refinement usually leads to the decrease in crack propagation energy because of generating smoother crack paths<sup>2)</sup> Accordingly, increases in fatigue strength of SMC materials take place owing to a considerable increase in the energy for crack initiation by itself.<sup>13)</sup> This means that improvements in the total fatigue life may simultaneously have a detrimental effect, in the sense that they can deteriorate the resistance to subcritical crack growth under constant-amplitude fatigue conditions. However, further experimental data are needed to verify this hypothesis.<sup>2)</sup>

Meanwhile, increments in strength and fatigue limit of the alloy due to the structure refinement are appreciably lower than those reported earlier for pure metals.<sup>1,2)</sup> These phenomena are attributed to much higher initial (in the well-annealed condition) strength of the alloy as compared with the pure titanium, owing to solid solution and hardening by lamellar precipitations of  $\alpha$ -phase in  $\beta$ -matrix. In the alloy the effect of grain boundary and dislocation strengthening resulted from the “abc” deformation is stronger than that of hardening by thin  $\alpha + \beta$  lamellae resulted from the heat treatment. However, difference in strength and fatigue limit between the SMC and the MC heat-strengthened conditions of the alloy is 20–25%. While the strength and fatigue limit of the SMC pure titanium are more than a factor of two higher than those of the MC condition.<sup>4,15)</sup> These results are closely related to the discussion initiated elsewhere<sup>16)</sup> concerning the role of grain refinement by severe plastic deformation in the strengthening of aluminium alloys. It has been concluded that severe plastic deformation can be more helpful to obtain a unique combination of strength and ductility in non-heat treatable alloys.<sup>16)</sup>

The results of tensile tests reveal several unusual features in ductility of the SMC alloy. For instance, the tensile elongation at fracture of the SMC alloy is lower than that of the heat-strengthened MC counterpart. According to the published literature,<sup>2,14)</sup> this behavior is usually attributed to plastic instability originating from the lack of an effective hardening mechanism; this instability appears as either shear bands or through “early” necking. At the same time, the area reduction and the specific work of deformation to failure of the SMC alloy are higher than those of the MC alloy by a factor of 2 and 3.4, respectively. Consequently, formation of the SMC structure increases both strength and ductility. Higher ductility of the SMC alloy during deformation treatment can be achieved at other strain paths where tensile stress is not the main component; *e.g.* rolling or compression.

The tensile test at elevated temperatures indicates that the grain size refinement noticeably degrades high-temperature strength of the alloy. The strength of the SMC alloy is lower than that of the MC alloy at temperatures above 300°C. The creep resistance of the SMC alloy is slightly lower than that of the MC alloy already at 250°C. Such behavior of the SMC alloy is not fully unpredictable.<sup>17)</sup> The decrease of grain size leads to a significant increase in grain boundary volume, which means that grain boundaries play an important role in

the deformation especially at elevated temperatures. These boundaries act as sources and sinks for dislocations, and facilitate such mechanisms by the sliding of grain boundary. A drastic increase in total elongation as well as intense softening observed in the SMC alloy at the short-term test above 400°C indicates that superplastic deformation is likely to occur.<sup>4,18)</sup>

The increase in volume fraction of grain boundary also leads to the predominance of the Coble creep, which is controlled by diffusion of vacancies along grain boundaries, over the Nabarro-Herring creep, which is controlled by diffusion of vacancies through the grain volume.<sup>17)</sup> Obviously, grain boundary diffusion is much faster compared to that through grain volume. Obviously, that is why the creep rate of the SMC alloy is higher than that of the MC alloy above 250°C. Further increase in temperature to 350°C leads to a drastic softening of the SMC alloy, which is most probably associated with the operation of grain boundary sliding.

## 5. Conclusions

The present paper has investigated mechanical properties of the Ti–6Al–4V alloy with SMC structure produced by severe plastic deformation where the grain size is as small as about 0.4 µm. The properties have been compared with those of a heat-strengthened MC alloy with the grain size of about 10 µm. The major results can be summarized as follows:

- (1) In comparison with the MC alloy, the alloy with SMC structure has shown increases in yield stress from 960 to 1180 MPa, ultimate tensile stress from 1050 to 1300 MPa, area reduction from 32 to 60% and fatigue limit from 580 to 690 MPa at room temperature.
- (2) In short-time tests at high-temperatures the higher strength of the SMC condition continues up to 300–400°C. However, the creep resistance of the SMC alloy is five percent lower than that of the MC alloy at 250°C. At 350°C the difference in creep resistance between the SMC and MC conditions increases to by a factor of 1.6.
- (3) An upper working temperature at which the benefit of the SMC condition of the Ti–6Al–4V alloy appears is

150–200°C with a possibility of short-time heating to 400°C.

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