

# Formation of submicrocrystalline structure in the titanium alloy VT8 and its influence on mechanical properties

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It is shown that the formation of microstructure with grain size up to  $0.06\ \mu\text{m}$  may occur during the course of plastic deformation of the Ti–6Al–3.2Mo ( $\alpha + \beta$ )-alloy with the initial coarse-grained lamellar structure. The formation of submicrocrystalline structure results from the development of dynamic recrystallization concurrent with the process of spheroidization. The temperature of superplastic deformation significantly decreases while strength characteristics at room temperature sharply increase in the alloy with such a microstructure.

## 1. Introduction

Metals and alloys with grain sizes of a few tenths or a few hundredths of a micrometre are of special interest because of their unique physical and mechanical properties [1–5]. Thus, in such materials, strength characteristics are sharply increased with the retention of high ductility at room temperature, while temperature significantly decreases or the rate of superplastic (SP) deformation increases [4, 5].

Materials with grain sizes less than  $1\ \mu\text{m}$  (with submicrocrystalline (SMC) structure) are mainly obtained by either sintering, or by microstructure refinement taking place during the process of plastic deformation, providing for the development of dynamic recrystallization (DR), and in this case the size can be regulated over a rather wide range. This has made it possible to suppose that under certain temperature–rate conditions of deformation, a microstructure with grain size close to the minimum size of subgrains, amounting to a few hundredths of a micrometre, is formed in the material [7].

The aim of the present work was thus to investigate the possibility of SMC structure formation under plastic deformation, and to study its mechanical behaviour using the VT8 titanium alloy.

## 2. Experimental procedure

( $\alpha + \beta$ )-titanium alloy, VT8, in the form of 150 mm diameter rod with the chemical composition, (wt %) Al 6.0; Mo 3.2; Fe 0.4; Si 0.3; O<sub>2</sub> 0.15; H<sub>2</sub> 0.015; N<sub>2</sub> 0.05; C 0.1; Ti balance, was used in this investigation. The polymorphic transformation temperature was  $1000^\circ\text{C}$ . Before tests the alloy specimens were annealed at  $1020^\circ\text{C}$  for 1 h, then furnace cooled. Mechanical tests were carried out on specimens of 10 mm diameter  $\times$  15 mm, which were subjected to compression in the temperature interval  $500\text{--}900^\circ\text{C}$  at

strain rates of  $5 \times 10^{-4}$  and  $5 \times 10^{-3}\ \text{s}^{-1}$ . Compression was performed in the special unit for hardening specimens at strain completion.

The alloy microstructure was studied in the central part of the strained specimens using scanning and transmission electron microscopes “JSM-840” and “JEM-2000EX”. The grain size was determined according to the standard procedure [8], the relative error amounting to 5%.

In order to estimate the mechanical behaviour of the alloy with SMC structure, specimens with gauge length diameter 3 mm and length 17 mm were tension tested. The specimens were cut from the central part of massive blanks after upsetting for 80%.

## 3. Results

Fig. 1 shows the  $\sigma$ – $\epsilon$  dependences for the VT8 alloy deformed in the temperature range  $600\text{--}900^\circ\text{C}$  at the initial rate  $5 \times 10^{-4}\ \text{s}^{-1}$ . A maximum value of flow stress was observed on the curves at all test temperatures, while after continuation of deformation a steady state flow was observed. It should be noted that we failed to plot the  $\sigma$ – $\epsilon$  dependence for  $500^\circ\text{C}$  because of the specimen's fracture after 20% straining.

In the initial state, the alloy microstructure was lamellar, the size of  $\beta$ -transformed grains amounting to  $400\text{--}500\ \mu\text{m}$ . The plates of  $\alpha$ -phase,  $1\text{--}2\ \mu\text{m}$  thick and  $40\text{--}70\ \mu\text{m}$  long were separated by  $\beta$ -interlayers  $0.1\text{--}0.2\ \mu\text{m}$  thick. The analysis of microstructure of the specimens after 75% straining showed that under all loading regimes the coarse-grained lamellar microstructure was transformed into fine grained equiaxed microstructure. The grain size of the phases decreased with decreasing strain temperature, and within the temperature range  $600\text{--}700^\circ\text{C}$ , the submicrocrystalline structure with grain sizes from  $0.1\text{--}0.4\ \mu\text{m}$ , respectively, was formed in the alloy (Fig. 2). In addition,

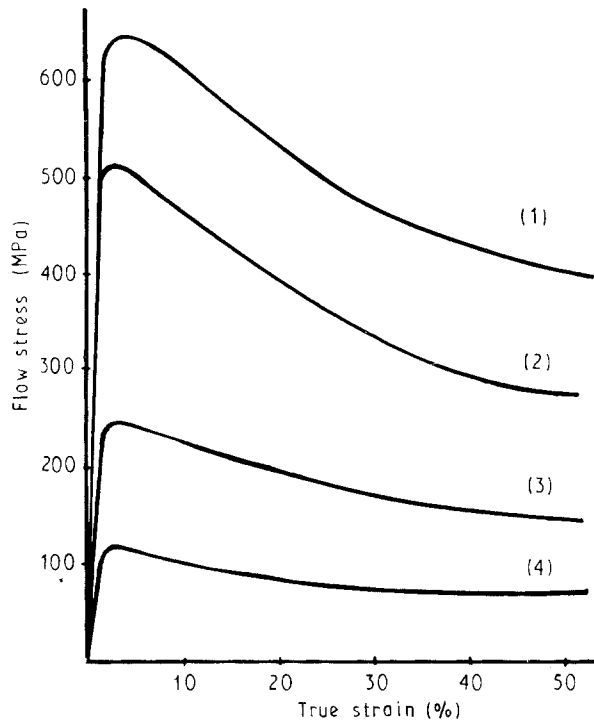


Figure 1 The dependence of flow stress on the degree of strain for VT8 alloy. (1)  $t = 600^\circ\text{C}$ , (2)  $t = 700^\circ\text{C}$ , (3)  $t = 800^\circ\text{C}$ , (4)  $t = 900^\circ\text{C}$ .

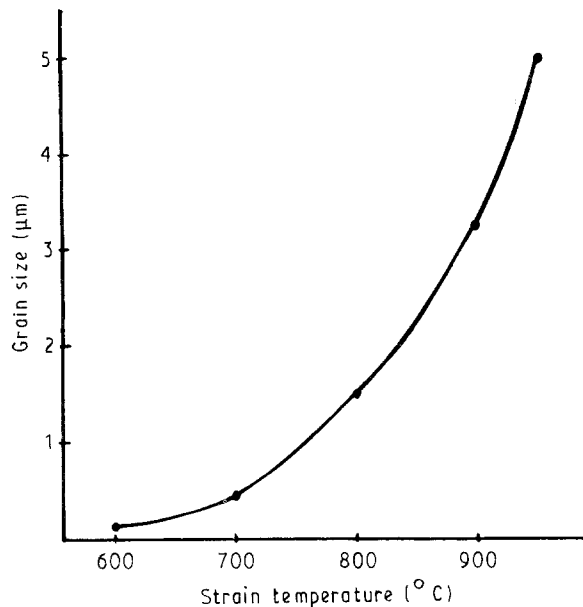


Figure 2 The dependence of recrystallized grain sizes on strain temperature.  $\varepsilon = 75\%$ ,  $\dot{\varepsilon} = 5 \times 10^{-4} \text{s}^{-1}$ .

the grain size depends on the strain rate: thus at  $T = 700^\circ\text{C}$  for  $\dot{\varepsilon} = 5 \times 10^{-4} \text{s}^{-1}$ ,  $d = 0.4 \mu\text{m}$ , and for  $\dot{\varepsilon} = 5 \times 10^{-3} \text{s}^{-1}$ ,  $d = 0.2 \mu\text{m}$ .

Investigation of the evolution of alloy microstructure was carried out on specimens strained with different degrees at  $700^\circ\text{C}$  and  $\dot{\varepsilon} = 5 \times 10^{-4} \text{s}^{-1}$ . In the initial stage of deformation the curvature of the plates and their bending in the direction of metal-plastic flow was observed, while at  $\varepsilon = 50\%$ , metallographic texture is formed in the alloy (Fig. 3a). During the course of further deformation, the formation of equiaxed grains was observed, their volume fraction

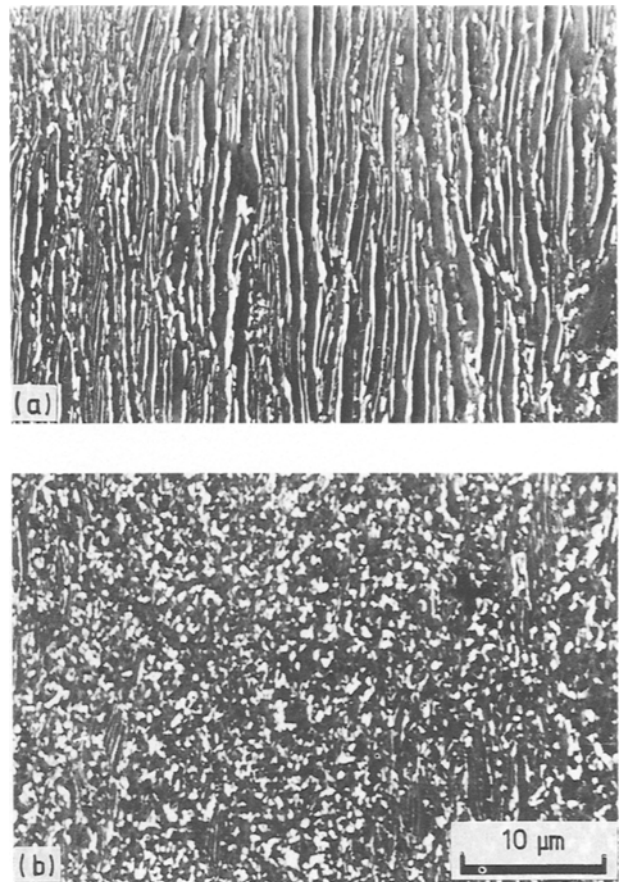


Figure 3 The microstructure of VT8 alloy strained at  $t = 700^\circ\text{C}$  and  $\dot{\varepsilon} = 5 \times 10^{-4} \text{s}^{-1}$ . (a)  $\varepsilon = 50\%$ , (b)  $\varepsilon = 75\%$ .

growing with increasing  $\varepsilon$ . In this case, the displacement of plate fragments relative to each other and the subsequent spheroidization of their fragments were observed. When strain reaches 75%, the metallographic texture is blurred, due to the formation of microstructure with mostly equiaxed grains (Fig. 3b).

The fine structure investigations have shown that dislocations are practically absent in the initial state in  $\alpha$ - and  $\beta$ -plates. With the onset of deformation ( $\varepsilon = 5\%$ ), the density of dislocations in the alloy increased, dislocation pile-ups being formed in  $\alpha$ -phase, while only single dislocations were observed in  $\beta$ -phase. On increasing  $\varepsilon$  up to 15%, subgrains  $0.3\text{--}0.4 \mu\text{m}$  in size were formed in  $\alpha$ -phase; these subgrains were found both in the vicinity of interphase boundaries and in the plate body. After 50% straining, the equiaxed grains with sizes of  $0.3\text{--}0.5 \mu\text{m}$  appeared in the  $\alpha$ -plate, the volume fraction of equiaxed grains increasing with  $\varepsilon$ .

After 15% straining, transverse subgrains were formed in  $\beta$ -plates, and simultaneously the entry of lattice dislocations into them was observed (Fig. 4a). Lattice dislocations were observed in the interphase boundaries as well. When strain reaches 50% transversal high-angle boundaries are formed in  $\beta$ -phase, the banded contrast on their image testifying to it. Grooves are formed in  $\beta$ -plates at the sites where interphase boundary gets out at the interphase surface. Interphase boundaries evidently change their structure too. If at the initial stage of deformation they

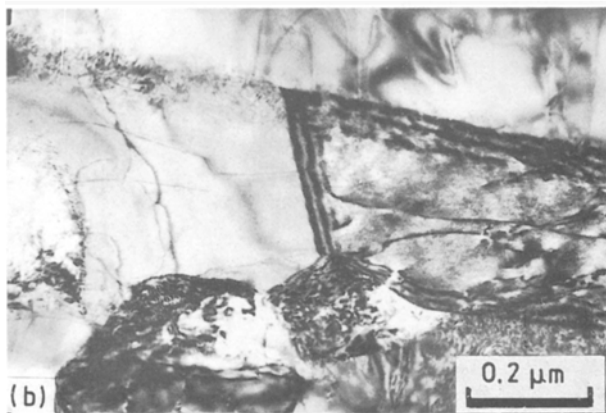
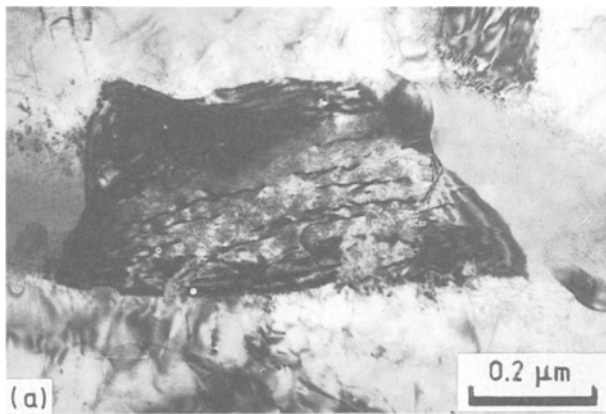


Figure 4 The fine structure of VT8 alloy strained at  $t = 700^\circ\text{C}$  and  $\dot{\epsilon} = 5 \times 10^{-4} \text{s}^{-1}$ . (a)  $\epsilon = 15\%$ , (b)  $\epsilon = 50\%$ , (c)  $\epsilon = 75\%$ .

have diffusion contrast which is apparently due to their semi-coherent structure [9], after  $\epsilon = 50\%$  the banded contrast, characteristic of the high-angle boundaries, appears on many of them (Fig. 4b). After 75% straining only single dislocations are observed in the body of equiaxial grains, while trapped lattice dislocations occur in their boundaries (Fig. 4c).

To estimate the possibility of obtaining still finer grains it was necessary to enhance the deformability of the material at lower temperatures. With this in mind a microstructure with a grain size  $\approx 0.2 \mu\text{m}$  was prepared. These blanks were used for cutting out specimens which then were deformed at  $500^\circ\text{C}$  and  $\dot{\epsilon} = 5 \times 10^{-4} \text{s}^{-1}$  by 80% without any signs of fracture. The microstructural investigations showed that,

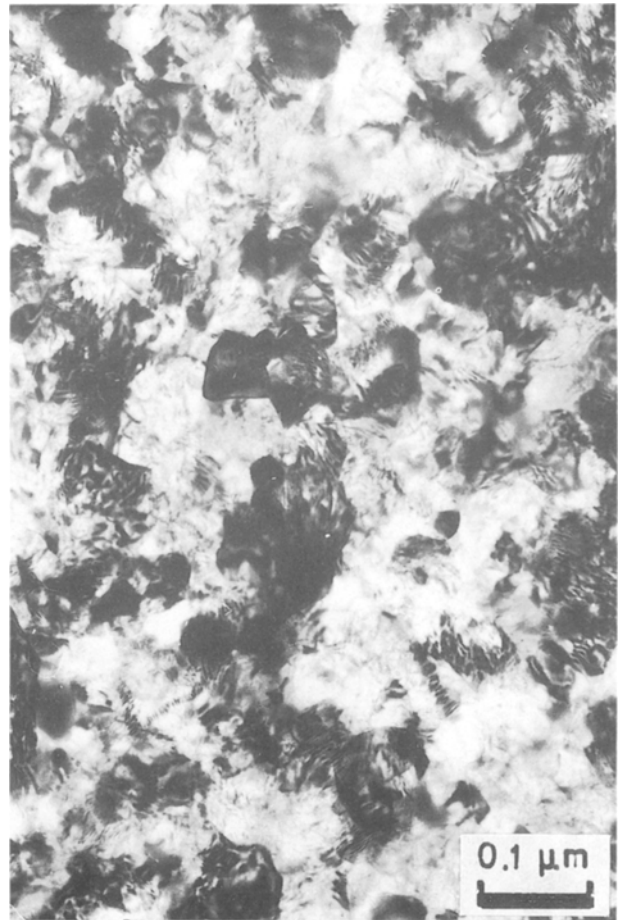


Figure 5 The fine structure of VT8 alloy strained at  $t = 500^\circ\text{C}$ ,  $\dot{\epsilon} = 5 \times 10^{-4} \text{s}^{-1}$ ,  $\epsilon = 80\%$ .

in this case, the microstructure with average grain size of  $0.06 \mu\text{m}$  was formed in the VT8 alloy (Fig. 5).

At the same time the subgrains with sizes of  $0.05\text{--}0.06 \mu\text{m}$  have been observed in the microstructure. Further decrease of strain temperature results in a sharp decrease of ductility which does not exceed 40%, this is probably why we have failed to obtain grains with size  $< 0.06 \mu\text{m}$  in this work.

Fig. 6 shows the dependences of relative elongation,  $\delta$ , and flow stress,  $\sigma$ , on the deformation temperature for the alloy with grain size of  $0.06 \mu\text{m}$  at the rate of  $5.6 \times 10^{-4} \text{s}^{-1}$ . At the temperature  $> 500^\circ\text{C}$  a sharp decrease in flow stress and an increase in elongation are observed, thus  $T = 550^\circ\text{C}$ ,  $\delta = 160\%$ , while  $\sigma = 280 \text{MPa}$ . For this temperature the effect of strain rate on the values of relative elongation and rate sensitivity coefficient has been investigated (Fig. 6b). It is seen that when  $\dot{\epsilon} = 1 \times 10^{-4} \text{s}^{-1}$  the values of  $m$ ,  $\delta$  and  $\sigma$  are 0.3, 260% and 205 MPa, respectively, i.e. SP features are observed in the alloy.

The influence of grain size on the characteristics of SP alloy has been studied at  $600^\circ\text{C}$  and  $\dot{\epsilon} = 5 \times 10^{-4} \text{s}^{-1}$ . The test results are given in Table I, and one can see that the microstructure refinement results in the considerable growth of  $\delta$  and  $m$  values and a decrease of  $\sigma$ . Thus, with the grain-size refinement from  $0.4 \mu\text{m}$  to  $0.06 \mu\text{m}$ , the flow stresses decrease from 390 MPa to 150 MPa, while the relative elongation increases from 225% to 600%.

TABLE I Mechanical properties of VT8 alloy with different grain sizes;  $\dot{\epsilon} = 5 \times 10^{-4} \text{ s}^{-1}$

Grain size ( $\mu\text{m}$ )	Strain temp. ( $^{\circ}\text{C}$ )	Coefficient $m$	Flow stress at $\epsilon = 40\%$ (MPa)	Specific elongation (%)	Tensile strength (MPa)
0.06	600	0.35	150	600	—
0.1		0.33	230	480	—
0.4		0.30	390	225	—
5		0.10	650	35	—
0.06	20	—	—	20 (53) <sup>a</sup>	1400
0.1		—	—	18 (46) <sup>a</sup>	1350
0.4		—	—	15 (35) <sup>a</sup>	1310
5		—	—	20 (45) <sup>a</sup>	1050

<sup>a</sup> The value of specific reduction is given in parentheses.

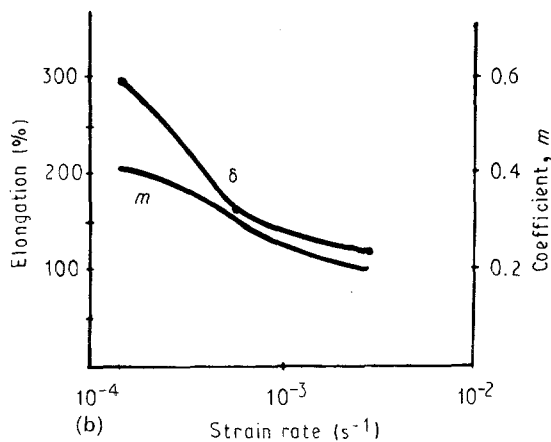
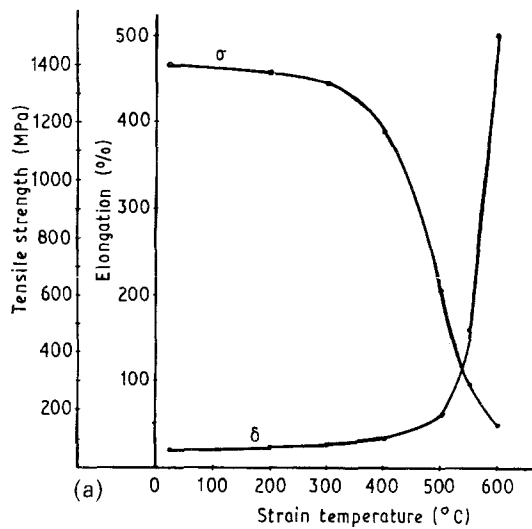


Figure 6 (a) Temperature dependence of specific elongation and flow stress for VT8 alloy with grain size of  $0.06 \mu\text{m}$ ,  $\dot{\epsilon} = 5.6 \times 10^{-4} \text{ s}^{-1}$ . (b) Strain-rate dependence of strain-rate sensitivity coefficient and specific elongation for the alloy with grain size of  $0.06 \mu\text{m}$ ,  $t = 550^{\circ}\text{C}$ .

The formation of SMC structure makes it possible to increase sharply the alloy strength, retaining high characteristics of plasticity at room temperature (see Table I). The decrease in grain size from  $5 \mu\text{m}$  to  $0.06 \mu\text{m}$  results in an increase of  $\sigma$  and  $\psi$  values by 45% and 17%, respectively, while the relative elongation remains at the same level.

#### 4. Discussion

The two-phase titanium alloy VT8 has been used to show the possibility of obtaining microstructure with a grain size up to  $0.06 \mu\text{m}$  by plastic deformation under certain temperature–rate conditions, i.e. the obtained grain size is rather close to that of nanocrystalline materials. However, unlike the latter, such a microstructure has been obtained in rather massive blanks.

Mechanical behaviour, characteristics of structural changes and the dependence of grain size on the temperature–rate conditions of deformation indicate that the formation of SMC structure in the alloy takes place as a result of dynamic recrystallization [10] development which occurs alongside the process of spheroidization. The mechanism of transformation of the alloy's initial lamellar structure into the equiaxed one at temperatures of  $600\text{--}700^{\circ}\text{C}$  has much in common with that observed in two-phase titanium alloys during deformation in the upper interval of the  $(\alpha + \beta)$  region [11, 12], although unlike high temperatures, the amount of  $\alpha$ -phase does not exceed 8%–9%.

Strain hardening is observed in the alloy at the initial stage of deformation, the accumulation of dislocations taking place more intensively in  $\alpha$ -plates than in  $\beta$ -phase, due to the lower strength of  $\alpha$ -phase at  $700^{\circ}\text{C}$  and below [13]. Later on, with the development of the dynamic recovery processes, the subgrain structure is formed in it. In  $\beta$ -phase only transverse subgrains are formed, due to the small thickness of the plates. It should be borne in mind that as shown earlier [14], in order to transform the plates into the equiaxed grains, the rearrangement of the initial semi-coherent interphase boundaries into the incoherent ones is necessary. During the course of subsequent deformation, this process, as well as the formation of high-angle boundaries in  $\alpha$ - and  $\beta$ -phases, is apparently performed by the interaction of sub-boundaries and semicoherent interphase boundaries with lattice dislocations [15]. The transformation of plates at low temperatures is accompanied by the weak formation of grooves on the interphase surface, while at high strain temperatures, deep grooves are formed, the weak formation of grooves being the main feature of spheroidization. Meanwhile, during deformation, the displacement of  $\beta$ -plate fragments relative to each

other is observed. The development of grain-boundary sliding along the transverse interphase boundary probably leads to the division of plates into fragments, thus providing for their further fast spheroidization. The important role of this mechanism in the spheroidization of plates has been pointed out previously [11]. As a result, the equiaxed submicrocrystalline structure is formed in the final stage of deformation. The character of the alloy's fine structure allows us to suppose that it is SP flow that is realized at this stage of deformation.

Therefore, SMC structure with a grain size which is close to the minimum possible subgrain size, can be obtained by plastic deformation of the VT8 alloy under conditions of dynamic recrystallization. Further microstructure refinement by this method is probably limited by the ability of dislocations to form dislocation shapes necessary for subgrain formation.

The formation of SMC structure in the VT8 alloy allows a sharp increase in its strength properties at room temperature, retaining high ductility, and reducing the SP deformation temperature. It is shown that SP deformation features were realized at  $t = 550^\circ\text{C}$  ( $0.42T_m$ ), while in the alloy with a grain size of  $5\ \mu\text{m}$ , SP was observed at  $t = 900\text{--}950^\circ\text{C}$  ( $>0.6T_m$ ) [15]. According to the theoretical notions, SP manifestation at such low (for SP) temperatures is caused by the considerable activation of diffusion processes due to the significant increase in the extent of boundaries. This is confirmed by the fact that at  $600^\circ\text{C}$ , in the VT8 alloy with decreasing grain size and, consequently, with increasing boundary extent, the flow stress decreases and the relative elongation increases.

## 5. Conclusions

1. It has been established that plastic deformation of the Ti-6Al-3.2Mo alloy at  $0.4T_m$  results in the formation of a microstructure with a grain size of  $0.06\ \mu\text{m}$  in the alloy.

2. The formation of submicrocrystalline structure in the alloy takes place as the result of concurrent development of dynamic recrystallization and spheroidization processes.

3. In the Ti-6Al-3.2Mo alloy with a grain size of  $0.06\ \mu\text{m}$ , the temperature of superplasticity decreases to  $550^\circ\text{C}$  ( $0.42T_m$ ) which is lower by  $300^\circ\text{C}$  than the usual temperature of superplasticity. A sharp increase in strength is observed at room temperature, high characteristics of ductility being retained.

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