Grain-structure refinement in titanium alloy under different loading schedules

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The dependence of the kinetics of globularization of the lamellar structure in titanium alloy VT9 (Ti–6.6% Al–3.5% Mo–1.7% Zr–0.27% Si) on the character of high-temperature loading was investigated. It was found that monotonic methods of loading (tension, torsion, tension with simultaneous torsion) were more effective means of refining the structure than non-monotonic ones (reverse torsion, torsion alternate with tension, multiaxial forging).

1. Introduction

The transformation of a coarse-grain lamellar ($\alpha + \beta$) morphology into an equiaxed one (including submicrograin structure) in titanium alloys is usually effected by means of large plastic deformation treatment within the $(\alpha + \beta)$ phase field, for example during many-sided forging [1, 2]. Such treatment is, in general, very time-consuming. At the same time, the structure refinement is known to be an essential prerequisite for superplasticity. In addition, the fine equiaxed structures promise to show some improvements in mechanical properties. Therefore, the search for the most effective ways of developing the fine-grain microstructures using different kinds of mechanical work, is of interest. This process is being thoroughly investigated in order to determine the most effective methods of thermomechanical treatment [3-5]. The influence of the strain rate value has been studied in detail [3]. It was established that, in any case, the forging reduction should not be less than 50%. At the same time, approximately 25% rolling reduction leads to an analogous degree of structure transformation [4]. Thus the amount of plastic deformation required to transform the material structure strongly depends upon the deformation schedule.

On the other hand, it is very important to provide the structural uniformity of the deformed preform. With that in mind, the deformation in different directions, in particular, multiaxial forging, is usually used in practice [2, 5]. However, such processes have a nonmonotonic character. The deformation path does not follow a smooth curve, but represents a broken line. The influence of these factors on the process of structure transformation remains obscure. The purpose of the presented work was to determine the most effective way to transform the material structure. Thus, the kinetics of the transformation process of a lamellar microstructure into an equiaxed one in titanium alloy was investigated at various loading schedules.

2. Experimental procedure

In order to achieve this aim the following two methodical problems should be considered and solved.

The first problem is to determine a suitable parameter required to evaluate numerically the degree of structural transformation. The efficiency of the transformation process of a lamellar structure into an equiaxed one can be evaluated by comparison of the number of equiaxed α -particles, *n*, visible on a selected area of specimen cross-section at the same values of the accumulated strain, e, strain rate, ξ , and temperature. The value of *n* can be calculated as the ratio of the number of equiaxed α -particles to their total number visible on the specified area of the specimen crosssection. A specific α -particle is considered to be equiaxed if the ratio of its maximum size to its minimum one does not exceed 2. The calculation of the value of n for each specimen was carried out using 12 circular fields of vision, so that the total number of grains exceeded 1000. The value of the confidence interval was evaluated, beginning with the level of fiducial probability of 95%.

The second problem is concerned with the necessity of providing identical values of strain rate and strain under different deformation schedules. It is well



Figure 1 Deformation paths in the deformation space e_1e_2 , where e_1 corresponds to the tension component while e_2 corresponds to the torsion component: (a) tension, $v = 0.08 \text{ mm s}^{-1}$; (b) torsion, $\omega = 0.016 \text{ s}^{-1}$; (c) tension with simultaneous torsion, $v = 0.046 \text{ mm s}^{-1}$, $\omega = 0.014 \text{ s}^{-1}$; (d) reverse torsion $\omega = 0.017 \text{ s}^{-1}$; (e) tension alternately with torsion $v = 0.08 \text{ mm s}^{-1}$, $\omega = 0.017 \text{ s}^{-1}$.

known that in the case of large plastic deformations, the value of strain may be calculated in different ways.

The strain tensor describes the deformation state of any material. This tensor has six independent components. For an incompressible material, there are only five independent components, which is why the deformation state of any material may be described by a five-dimensional vector [6]. The locus of the end of this five-dimensional vector, corresponding to its changes during plastic deformation, represents the socalled "deformation path," which was introduced by Iljushin in 1963 [6]. The value of accumulated strain may be calculated by integrating the value of the strain rate along the streamline. The distance of the end of the five-dimensional vector from the origin of the coordinates represents the strain intensity. For a one-dimensional tension test, strain intensity coincides with the ordinary relative strain $\varepsilon = \Delta L/L$, while accumulated strain, e, is $\ln(L/L_0)$.

The different types of deformation path that may be realized by a two component tension/torsion testing machine are shown in Fig. 1. They may be characterized as follows. Paths (a) and (b) represent simple, single-component, monotonic processes. The difference between them is in the stress state. Path (c) represents a complex, two-component, monotonic process. Path (d) represents a complex, singlecomponent, non-monotonic process. Finally, path (e) represents a complex, two-component, non-monotonic process, having 90° breaks of the deformation path.

The value of the accumulated strain may be calculated in accordance with the following expressions. For the case of a simple tension test

$$e_1 = \ln\left(L/L_0\right) \tag{1}$$

For the case of a simple torsion test

(

$$e_2 = r_0 \omega t / (L_0 \, 3^{1/2}) \tag{2}$$

For the case of a tension with torsion test

ρ =

$$= 2(1 + \theta^{2}/3)^{1/2} - 2\{1 + \theta^{2}/[3(1 + \varepsilon)]\}^{1/2} + \ln(1 + \varepsilon) + \ln(1 + \theta^{2}/[3(1 + \varepsilon)]) + \{1 + \theta^{2}/[3(1 + \varepsilon)]\}^{1/2}) - \ln[1 + \theta^{2}/6] + (1 + \theta^{2}/3)^{1/2}]$$
(3)

where L_0 and L are initial and current length of the specimen, respectively, t is the time of deformation, ω and v, both constants, are the angular and axial velocity of the grip end, respectively, $\varepsilon = vt/L_0$ and $\theta = r_0 \omega/v$ are non-dimensional parameters.

Equations 1 and 2 are well known, while Equation 3 is derived by integrating the following relationship for the value of effective strain rate, ξ , for the case of a tension/torsion test [7]

$$\xi^2 = v^2 / L^2 + r^2 \omega^2 / (3L^2) \tag{4}$$

 $\varepsilon = vt/L_0$ represents an ordinary relative strain during simple tensile test. $\theta = r_0 \omega / v$ is the ratio of circular velocity $(r_0\omega)$ and linear velocity (v) for the outer specimen points at the initial moment of time. It reflects the relative role of torsion during the tension/torsion test. In particular, $\theta = 0$ corresponds to the simple tension test, while $\theta \ge 1$ corresponds to the case of a simple torsion test. Equation 3 includes the well-known expressions for the values of the accumulated strain for the simple tension test and for the simple torsion test as special cases of application. In fact, from Equation 3 with $\omega \rightarrow 0$ it follows that $e \rightarrow \ln(1 + \varepsilon)$, which coincides with Equation 1. At the same time, from Equation 3 with $v \rightarrow 0$, it follows that $e \rightarrow r \omega t/(L3^{1/2})$, which corresponds to the wellknown expression for the torsion test. At the same time, Equation 4 includes the following well-known expressions. With $\omega = 0$ from Equation 4 it follows that $\xi = v/L$ (simple tension test); with v = 0 from Equation 4 it follows that $\xi = r\omega/(L3^{1/2})$ (simple torsion test).

The material used was titanium alloy VT9 (Ti -6.6%Al -3.5%Mo -1.7%Zr -0.27%Si). Preforms made of this alloy were preliminarily deformed within the $(\alpha + \beta)$ region. The deformed preforms were subjected to the following heat treatment: 30 min annealing at 1030 °C (β -region), air cooling; 1 h annealing at 850°C, water quenching. An annealing at 1030°C together with air cooling enables relatively small matrix β -grains and sufficiently thin lamellae to be obtained. Additional annealing at 850 °C with water cooling provides non-equilibrium phase composition during subsequent deformation at 960 °C. Such treatment promotes lamellar structure transformation during deformation [8]. This is necessary in order to obtain the elongation of maximal value during the simple tension test. Specimens of 30 mm gauge length and 8 mm gauge diameter were deformed at the same temperature, 960 + 3 °C. The values of linear, v, and angular, ω , velocity of the grip end were selected in order to provide the same values of strain rate of the order of 10^{-3} s⁻¹ (near the outer specimen surface). The deformation was followed by water quenching.

Microstructure was investigated at the longitudinal section near the outer surface of the specimen.

It should be emphasized that all deformation paths, which are represented in Fig. 1, are flat. Therefore, it is necessary to realize a more complex deformation path, which is characterized by three-, four- or five-dimensional curves in Iljushin's deformation space. Unfortunately, it is impossible to realize such loading using a standard tension/torsion scheme of deformation. Moreover, it is easy to show that usual multiaxial forging of a cubic preform is characterized by a flat deformation path as well. Iljushin thus proposed the realization of such loading when all projections of the deformation path on each of the five axes in the deformation space were approximately equal in value. With that in mind, he offered to compress a cubic specimen many times in accordance with a special program. Dmitriev [9] has calculated such a program in detail. The program includes the following steps. A cubic preform is forged consequently along its three edges. Then a cubic sample is cut from the deformed preform in accordance with pre-determined angles. This procedure is repeated twice.

A sixth deformation path was realized as follows. The initial preform (cube $50 \text{ mm} \times 50 \text{ mm} \times 50 \text{ mm}$) with lamellar microstructure was subsequently compressed 18 times along its three edges in accordance with a pre-determined sequence [9]. The forging reduction was 20% at each of the 18 compressions - after that the preform was quenched in water. A new cubic sample was then cut from the deformed and quenched preform in accordance with pre-calculated angles [9]. The entire procedure was then repeated twice, so that the total number of compressions was 54. The loading was effected by an hydraulic press EU-100. The temperature was 960 \pm 20 °C, while the value of strain rate was about 10^{-3} s⁻¹. Microstructure was studied at the corners of all of three cubes and at the centre of the last cube.

3. Results and discussion

The results of calculating the number of equiaxed particles formed in the VT9 alloy under different loading conditions are represented in Figs 2 and 3. From Fig. 2 it can be seen that as the value of the accumulated strain increases, the number of equiaxed particles increases first quickly and then more and more slowly. The second, fading part of the n-e curve was not observed here because of neck development and the consequent specimen failure at e > 0.8. However, tension with simultaneous torsion provided a high degree of structure refinement n = 70% already at e = 0.8. At the same time, for the case of broken deformation paths (reverse torsion and tension alternately with torsion) the number of equiaxed particles, n, does not exceed 40% when the value of e increases to 1.6. The five-dimensional loading path, having considerably more breaks at the same values of accumulated strain, proved to be a still less effective process: $n \cong 20\%$ at e = 1.6. Moreover, even at e = 10, the number of equiaxed particles does not exceed 50%. Thus, all *n*–*e* curves may be divided into two groups:



Figure 2 The number of equiaxed particles, n(%), formed in titanium alloy VT9 with initial lamellar structure at $T = 960^{\circ}$ C and $\xi \approx 10^{-3}$ s⁻¹ versus the accumulated strain, $e: (\circ)$ tension; (x) torsion; (\triangle) tension with simultaneous torsion; (\Box) reverse torsion; (\bullet) tension alternately with torsion.



Figure 3 The number of equiaxed particles, n(%), formed in titanium alloy VT9 with initial lamellar structure during multiaxial forging in accordance with a special program, realizing a five-dimensional deformation path, versus the accumulated strain, e.

monotonic processes (a, b, c) and non-monotonic processes (d, e, f). It should be emphasized that the n-e curves from the first group lie perceptibly higher than those from the second group.

It should be noted that an extremely low efficiency of the process of structure transformation was observed for a broken deformation path not only in the above-mentioned experiments, but in our earlier unpublished investigation as well [10]. The cyclic deformation of titanium alloy VT9 was realized at approximately the same temperature and strain rate as in the above-mentioned experiments. It was found that after 50 tension-compression cycles with a magnitude of plastic deformation of about 10%, there was no visible structure refinement, though the value of accumulated strain was very significant. Analogous results were obtained elsewhere [11] during cyclic loading of the steel having a lamellar pearlite structure.

Thus, the results obtained enable us to conclude that the monotonic loading is a more effective method of structure refinement than non-monotonic loading. In addition, tension with simultaneous torsion, that is two-component loading, leads to better results (see Fig. 2). It is rather difficult to establish, unambiguously, the reason for the above-indicated features of the structure behaviour of the alloy under consideration, because a phase transition takes place during cooling after deformation. At the same time, the influence of the deformation path on the lamellar structure refinement may be accounted for by the following reasons. It is known [12-14] that the transformation of $(\alpha + \beta)$ lamellar structure in titanium alloys is closely related to the recrystallization development. In turn, a certain defect density should be created in the material to initiate the recrystallization processes. The character of the defect structure, including accumulated dislocation density, and their volume distribution, is determined by the active deformation mechanisms. In particular, it has been shown [15] that a relationship exists between the deformation mechanisms and features of the recrystallization processes. On the other hand, it is known that the deformation conditions influence the deformation mechanisms as well. For example, it is known [16], that α -Titan tends more towards twinning during torsion than during tension. Proceeding from these, one can suppose that the observed differences in the kinetics of lamellar structure transformation under different loading conditions (Fig. 2) are caused by the changes in the deformation mechanisms and, consequently, in the kinetics of recrystallization.

The negative role of the breaks in the deformation path during the development of the recrystallization processes and, correspondingly, the grain refinement, is most likely connected with their impedance of dislocation accumulation. Depending on circumstances, the above-indicated counteraction may be effected in different ways. A break in the deformation path after small deformation, when the dislocation density is insignificant, causes considerable increase in the number of movable dislocations. This takes place at the expense of some of immovable dislocations, possibly being able to move along new paths skirting old obstacles. Therefore, the number of generating dislocations needed to maintain the process of plastic deformation, turns out to be less than in the case of the absence of a break in the deformation path. All this leads not only to the smaller dislocation density in comparison with monotonic deformation but to some softening of the material as well. In fact, that the reduction in the value of yeild stress during inverse loading is known as Baushinger's effect [17].

Another possible mechanism of the dislocation density decrease owing to breaks in the deformation path may be that dislocations of opposite sign are generated after the break. This factor, in turn, promotes the annihilation of dislocations. It also reduces the tensorial dislocation density, which determines the crystal curvature and, correspondingly, the possibility of forming recrystallization nuclei. Both of these mechanisms seem to account for the low efficiency of the structural transformation during non-monotonic loading processes.

The above-described suggestions concerning the mechanisms of processes lamellar structure transformation should be experimentally verified in further investigations. However, the experimental results obtained in the present work may already be of help in promoting the development of advanced highly efficient methods of metal working, which enable parts of the needed sizes with fine-grained microstructure to be obtained.

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