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Three-stage relationship between flow stress and dynamic grain size in titanium in a wide temperature interval

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ABSTRACT

The relationship, $\sigma \propto D^{-N}$, between the flow stress (σ) and the size (D) of deformation-induced grains has been established for commercial-purity titanium in the interval 77–1123 K. The relationship has three parts with different grain size exponent (N). The N values of 0.83, 0.38, 0.95 were obtained in the intervals of low stresses (hot deformation), moderate stresses (warm deformation) and high stresses (cold deformation), respectively. The changes in the values of N were suggested to be associated with different mechanisms of microstructure evolution.

1. Introduction

The microstructural control during plastic deformation is of considerable importance in the thermo-mechanical processing of the structural metallic materials because of desirable properties of the products. The microstructure evolution during hot/warm plastic working is generally discussed in terms of dynamic recrystallization (DRX) [1]. The characteristic stress–strain curves and new grains evolved during DRX have been studied in detail as a function of temperature–strain rate conditions, which can be represented by the Zener–Hollomon parameter Z [1]:

$$Z = \dot{\varepsilon}\exp(Q/RT), \quad (1)$$

where $\dot{\varepsilon}$ denotes the strain rate; R is the universal gas constant; T is the temperature and $Q = -R[\partial \ln \dot{\varepsilon}/\partial(1/T)]_{\sigma}$ is the apparent activation energy of DRX. Depending on Z, DRX in materials with low-to-medium stacking fault energies develops via either continuous (high Z, low temperature domain) or discontinuous (low Z, high temperature domain) mechanisms [1–3]. The discontinuous DRX is associated with conventional nucleation and growth of new recrystallized grains [4–6]. In case of continuous DRX, the new grains form due to increase in the subgrain misorientations as a result of progressive accumulation of dislocations in sub-boundaries [2,7]. At the steady-state flow, the size of recrystallized grains (D) can be expressed by a power law function of the flow stress (σ_s) as

$$\sigma_s = KD^{-N}, \quad (2)$$

where K and N are constants [1,8]. In turn, Z can be related to σ_s, as: $Z = c_1 \sin h(c_2\sigma_s)^n$, where c_1, c_2, and n are constants. It has been shown that N in Eq. (2) takes on the values of approx. 0.7 or 0.4 for discontinuous DRX (high temperature domain) or continuous DRX (low temperature domain), respectively [9–12]. The transition temperature from discontinuous to continuous DRX for a commercial-purity titanium was found to be ~673 K [11].

The small value of the grain size exponent, N, in the low temperature domain suggests rapid decrease in size of the dynamically recrystallized grains with decreasing the deformation temperature. It should be noted that the subgrain size can also be related to the flow stress through Eq. (2) with the subgrain size exponent of about 1. Therefore, a decrease in the deformation temperature leads the dynamic grain size to approach the size of deformation subgrains. Since the grain size cannot be smaller than the subgrain size, the above relationships suggest an apparent limit for grain refinement by cold working, when the grains and subgrains approach the same size. However, the relationship between the flow stress and the dynamic grain size, when grains become comparable with subgrains, has not been studied in detail, in spite of a great interest in production of ultrafine grained or nanostructured materials. The aim of the present work is to study the relationship between the flow stress and the dynamic grain size in a wide range of deformation temperatures down to cryogenic

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deformation. Commercial-purity titanium was selected as a promising structural material for advanced medical applications.

2. Experimental details

Commercial-purity titanium (impurities in wt% less than 0.01 Al; 0.02 Si; 0.12 Fe; 0.14 O; 0.004 C; 0.003 N; 0.0008 H; others – 0.077) was supplied in the form of 30-mm diameter hot-rolled bar with recrystallized microstructure with grain size of 30 μm. Compression tests were used to establish the effect of temperature and strain on the microstructure and mechanical behavior of the commercial-purity (CP) titanium at elevated temperatures. Cylindrical samples measuring $\varnothing 7 \times 10\ mm^2$ were compressed isothermally in air in the interval 673–1123 K using a Schenk mechanical testing system at a nominal strain rate of $10^{-3}\ s^{-1}$ to a true strain of 1.2. Soaking of the specimens prior to deformation was 15 min. At temperatures below 673 K, stable microstructure was produced by plane rolling. Rectangular samples measuring $10 \times 30 \times 4\ mm^3$ were rolled at 623 K, 293 K or 77 K using a fixed rolling speed of 30 mm/s. Unidirectional multipass rolling to a total nominal strain of 2.6 was performed using a reduction (draft) per pass of 0.2–0.07 mm. An isothermal rolling mill was used for rolling at 623 K. Before rolling and between each rolling pass the specimens were soaking at 623 K for 10 min. Prior to cryorolling, each preform was encapsulated between sacrificial titanium sheets which were joined by spot welding. The pack was then cooled to 77 K in liquid nitrogen and rolled between room-temperature rolls. The temperature of the canned workpiece during such pack rolling process did not increase by more than 20 K. To ensure nearly isothermal condition, each pack was cooled in liquid nitrogen between each rolling pass. Details of the experimental procedure can be found elsewhere [12]. To determine mechanical properties of the specimens with the stable microstructures produced by rolling, tensile tests were conducted at 623 K, 293 K and 77 K for specimens rolled at respective temperatures. For this purpose, the flat specimens with gauge dimensions of 16 mm length × 3 mm width × 1.5 mm (or less, depending on the final sheet thickness after rolling) thickness were machined and pulled at a constant crosshead speed of 1 mm min^{-1} in a screw-driven test machine to fracture. The specimen and the grips were immersed in an open-top vessel filled with liquid nitrogen whose level was continuously monitored and adjusted to ensure adequate temperature control for the tensile tests at cryogenic temperature. The microstructural analysis was performed in the central portion of each specimen using a JEOL JSM-840 scanning electron microscope and a JEOL JEM-2100FX transmission electron microscope.

3. Results and discussion

3.1. Warm to hot deformation behavior and microstructures

A series of the stress–strain curves for compression of CP titanium at 673–1123 K is shown in Fig. 1a. All curves obtained by compression show continuous increase of flow stress. The flow stresses rapidly increase at initial stage of deformation to strains of approximately 0.2. Upon further deformation, the rate of strain hardening gradually decreases leading to apparent steady state flow at strains of 0.2–0.5. Then, the deformation to strains of $\varepsilon > 0.5$ (~40%) is accompanied by progressive increase in the flow stress that is usually associated with increasing contribution of friction force between the die and the specimen surfaces. The effect of deformation temperature on flow stress at apparent steady state corresponding to $\varepsilon \sim 0.4$ is represented in Fig. 1b in $\log(\sigma)$ vs $1/T$ scale. It is clearly seen that the temperature dependence of flow stress changes at a temperature of about 923 K, when the flow stress is approx. 150 MPa. Under hot deformation conditions, i.e., below 150 MPa, the flow stress shows strong temperature dependence. In contrast, the flow stress exhibits quite weak temperature dependence in the range of warm deformation, i.e., above 150 MPa. The different deformation behavior during warm or hot working at 673–823 K or 923–1123 K indicates a difference in the mechanisms of microstructure evolution in the corresponding temperature domains.

Typical deformation microstructures that develop in the CP titanium during hot deformation at 1023 K are shown in Fig. 2. The deformation microstructures that developed at relatively small strains consist of pan-caked original grains with frequently corrugated boundaries (Fig. 2a). Such boundary corrugation promotes the bulging mechanism for discontinuous DRX nucleation [13]. The new fine grains rapidly develop at original boundaries and at triple junctions (Fig. 2a). The number of new fine grains evolved at original grain boundaries increases with increasing the strain leading to a necklace-like microstructure consisting of coarse remnants of the original grains separated by the fine grained layers (Fig. 2b). Note here that such kind of microstructure was frequently observed during the discontinuous DRX development [3,14,15]. The fine grained layers progressively spread out and consume the original grains as the strain increases (Fig. 2c). Therefore, the new grains result from conventional discontinuous DRX involving nucleation and grain growth during hot working at elevated temperatures.

Decreasing the deformation temperature slows down the grain boundary mobility and, therefore, hinders the development of discontinuous DRX at relatively low temperatures. As a result, the DRX mechanism changes at a temperature of about 0.5 Tm from discontinuous to continuous one, which is characterized by a gradual transformation of strain-induced subboundaries into grain boundaries when the subboundary misorientations increase to the values typical of conventional grain boundaries during deformation [3,10,16]. Typical microstructures that evolve in the CP titanium under conditions of warm deformation accompanied by the development of continuous DRX are shown in Fig. 3. An early deformation brings about high-density dislocations, which are rather homogeneously arranged in cells and dense dislocation walls passing through a grain (Fig. 3a). These deformation substructures are characterized by low-angle misorientations as suggested by the single-spot diffraction pattern obtained from a selected area of 1.4 μm diameter. An increase in the strain leads to the development of well-defined subgrains throughout the initial grain interiors (Fig. 3b). Finally, the uniform ultrafine-grained microstructure with an average grain size of 350 nm develops at sufficiently large strains in place of preceding subgrains (Fig. 3c). The developed ultrafine grains are bounded by high-angle boundaries (many spots diffraction pattern in Fig. 3c was obtained from a selected area of 650 nm) with equilibrium triple junctions. The equilibrium triple junctions form owing to local grain boundary migration that is essential feature of the continuous DRX [17].

3.2. Cold worked microstructures and flow stresses

Typical cold-worked microstructures that develop in the CP titanium during deformation at 77 K are shown in Fig. 4. A distinctive feature of the grain refinement during cold working is the operation of multiple deformation twinning resulting in rapid grain subdivision at relatively small strain (Fig. 4a). Then, the sequence of structural changes during the cold working is similar to that observed under conditions of warm deformation. Namely, the ultrafine grained structures result from gradual transformation of the high density dislocation substructures as the subgrain misorientations increase during cold deformation (Fig. 4b and c).