Analysis of Microstructure and Its Effect on Yield Strength of Pure Alpha-Titanium Consolidated by Equal Channel Angular Pressing

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The recycling value of chips of pure alpha-Ti was demonstrated with eco-friendly equal channel angular pressing (ECAP) to replace energy intensive melting and casting. Electron backscatter diffraction (EBSD) was used to reveal the microstructure formed after single- or multi-pass ECAP, characterized by a heterogeneous structure containing high- and low-angle grain boundaries (HAGBs, with misorientation ≥15°, and LAGBs, <15°). Grain formation was analyzed by considering the shear strain due to tooling features of ECAP die, and thermal effect because of elevated temperature. The strength was properly predicted by the Bailey-Hirsch relation, consistent with the analysis of the contributions from alpha-matrix, HAGBs, and LAGBs, respectively. The paper makes clearly the underlying physical metallurgy principles to achieve high yield strength in pure alpha-Ti. [doi:10.2320/matertrans.M2018033]

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1. Introduction

A sustainable development through the recycling of metallic resources has become one of the critical problems of the 21st century. Machining is widely conducted with Ti and alloys in aerospace industry and biomedical engineering, but much of the material may be removed to valueless chips. In this study, chips of commercial purity alpha-Ti were recycled by environmentally-benign equal channel angular pressing (ECAP), as a substitute to energy intensive melting and casting.

To date, some studies have been reported about the processing of pure alpha-Ti by ECAP.1−4 Of technical significance is to understand the microstructure in hard-to-deform Ti suffering from its hexagonal close-packed lattice, and to address the question as to how it was strengthened. Generally, a couple of ways were adopted to harden pure alpha-Ti. One is by the presence of interstitials such as O and N. The other is by refining alpha-grains via thermomechanical processing, e.g., forging and extrusion of high extent of strains.5 In this paper, electron backscatter diffraction (EBSD) was used to reveal high- and low-angle grain boundaries (HAGBs, with misorientation ≥15°, and LAGBs, <15°).6 The Hirsch-Bailey relation and the Hall-Petch relation were verified to analyze the yield strength of pure alpha-Ti.

2. Experimental Procedure

Chips of commercial purity alpha-Ti (ASTM grade 2) were consolidated by ECAP using a die with two equal channels in diameter (~11 mm) intersecting at 90°. Lined with a piece of steel foil having its outside attached with a layer of graphite paper (as lubricant), the entrance channel was charged with chips whose cubic sizes were averagely less than 1 mm in thickness, and several millimeters in width and length, respectively. Then, under a hydrostatic pressure (with an extent up to ~0.7 GPa), cold consolidation took place causing very intimate contacts between chips. Subsequently, a heating device enabling stabilization of temperature within ±1°C was designed to heat the die to 450°C or 590°C. Then, hot consolidation was occurred with a forward plunger moving at ~5 mm/min and providing high pressure up to ~1.0 GPa, and a back plunger providing a back-pressure ~50 MPa. A very efficient ECAP route termed route C was adopted to refine grains by 180° rotations clockwise between passes about the sample’s longitudinal axis.7,8 After ECAP the sample was quenched immediately with cold water to maintain the microstructure hot deformed. Based on the Archimedes’ principle, a Mettler Toledo 33360 density determination kit was used to confirm that full density (~4.51 g cm−3) was readily obtained. An entire inspection by scanning electron microscopy (SEM) was conducted to further confirm the absence of any visible porosity. The sample for metallographic characterization was chopped, and ground using SiC abrasive grinding papers with P-Grades up to 2500. Polishing of the sample was conducted with a solution of CH3OH (600 ml), HClO4 (60 ml) and C6H5OCH2CH2OH (360 ml) at 30 V for 20 s at −10°C, and in a colloidal silica suspension using a vibratory polisher (VibroMet Buehler) at 220 VAC 50 Hz. EBSD was conducted with JEOL JSM 7001F FEGSEM and FEI NOVA NanoSEM 230. Scanning step sizes 0.05 to 0.1 μm was used depending on the pre-estimate of average grain sizes, and a tilt angle 70° was chosen for the detection by EBSD. A subroutine HKL Channel 5 TANGO was used to analyze the scanning results. To evaluate yield strength, samples of sizes 4 × 4 × 6 mm were compressed at a strain rate 1 × 10−3 s−1 by a mechanical testing system equipped with a standard non-contacting laser extensometer. The compression is not convincing until unfavorable effects of friction and buckling are appropriately handled. Vaseline was smeared as lubricant to reduce the friction on the ends of samples. According to Hosford,9 a height-to-width ratio ~1.5 (i.e., 6:4) was designed for compressive samples to overcome the dead metal and a barrel profile caused by the friction.

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3. Results and Discussion

As shown in Fig. 1, the microstructure of pure alpha-Ti subjected to a single-pass ECAP was resolved into HAGBs (the black lines) and LAGBs (the white lines), respectively. For the single-pass ECAP, only the temperature $\sim 590^\circ$C was selected since at any temperature lower than this point, it is reluctant to eliminate pores after only one pass of consolidation. LAGBs were discontinuously presented before ECAP (Fig. 1(a)). However, as enclosed by HAGBs, grains were nearly equiaxed despite that some of them were distorted under hydrostatic pressure. By the subroutine HKL Channel 5 TANGO, the average circle equivalent diameter ($d$) of equiaxed grains (prior to ECAP) were determined as $\sim 7.4$ µm. After one pass, the networking of LAGBs became pronounced (Fig. 1(b)), the reason for which is attributed to dynamic recovery stimulated by shear strain. Moreover, rather than subdivided, coarse grains were elongated to form layered microstructure with an average lamellar intercept $\sim 4.0$ µm. Geometric analysis of tooling feature was conducted to explain these findings. At an intersecting angle $2\phi (= 90^\circ)$, the shear strain for one pass is $\gamma = 2\cot\phi$. Thus, $\gamma = 2$ at $\phi = 45^\circ$. The elongation of grains is described by $\left(\begin{array}{c} x_2 \\ y_2 \end{array}\right) = \left(\begin{array}{cc} 1 & 0 \\ 0 & 1 \end{array}\right)\left(\begin{array}{c} x_1 \\ y_1 \end{array}\right)$, where $(x_1, y_1)$ or $(x_2, y_2)$ is the coordinate before and after one pass. By the matrix transformation, the shape of grains was converted from circle to ellipse. As shown in Fig. 1(b), being the elliptic minor axis, the intercept between lamellas ($\lambda$) is given by $\lambda = d\left(\sqrt{1 + 2\cot^2\phi + \sqrt{\cot^2\phi + \cot\phi/2}}\right)^{-1}$, and the inclination angle between the major axis and the direction of exit channel (arrowed in Fig. 1(b)) is estimated with $\beta = \tan^{-1}(\sqrt{1 + \cot^2\phi - \cot\phi})$. By the formula, $\lambda = 3.6$ µm and $\beta = 22.5^\circ$ as per $\phi = 45^\circ$ and $d = 7.4$ µm, consistent with the observation that $\lambda \approx 4.0$ µm and $\beta = 20$–25°, respectively.

Furthermore, with the increase of passes, route C endowed more complex shear pattern because of 180° rotations about sample axis between passes, which implies a varied orientation of shear plane pass by pass, and thus allows more equiaxed grains to form after a multiple number of passes. In contrast, Route A (without sample rotation between passes) was used by Garcia-Infanta and co-workers to deform hypoeutectic Al–7Si alloy (in mass%). By route A, primary dendrite cells and eutectic constituent were simply stretched with an inclination angle (to the sample’s longitudinal axis) gradually reduced from $\sim 22.2$ to $\sim 3.5^\circ$ for repetitive ECAP with the number of passes increasing from 1 to 8.

As indicated by Figs. 2 and 3, fine grains (enclosed by HAGBs) were produced after multi-pass ECAP. Because it
was a number fraction that was used to estimate the mean grain size, very small average grain size was achievable despite there existed some coarse grains embedded in the “ocean” of a large number of ultrafine grains. Another pronounced feature, according to statistical distribution of grain boundary misorientation (see Fig. 4), is to form a lot of LAGBs. Accordingly, Table 1 summarized microstructure parameters such as grain size ($d$), mean misorientation ($\theta_{\text{LAGB}}$) and linear intercept ($L$) between LAGBs. Moreover, some grains were partially surrounded by HAGBs, and in part, surrounded by LAGBs, due to the operation of continuous dynamic recrystallization (CDRX), by which new grains were progressively formed through converting gradually LAGBs into HAGBs by increasing misorientation.\(^\text{14}\) Unlike a static recrystallization that occurs in post-annealing, CDRX happens during ECAP, and is dynamically driven by thermal effect and the strain energy accumulated with the exertion of a multiple number of passes.

To evaluate the yield strength achieved by multi-pass ECAP, compressive true stress ($\sigma$) vs. true strain ($\varepsilon$) curves are presented in Fig. 5. The dependence of 0.2% proof stress ($\sigma_{0.2}$) on $\rho^{1/2}$ obeys the Bailey-Hirsch relation\(^\text{15}\) (see Fig. 6):

$$\sigma_y = \sigma_0 + k_{B-H} \rho^{1/2}$$

(1)

where $\sigma_y$ is yield stress, $k_{B-H} (= 20.93 \text{ MPa \mu m})$ is the slope...
that measures the strengthening capability due to dislocation multiplication, $\rho$ is dislocation density estimated by $\rho = (3b\eta_{\text{LAGB}}/b)L^{-1.16,17}$ where $b$ is the Burgers vector length ($= 0.347$ nm for Ti), and $\sigma_0$ ($\sim 129$ MPa) represents the contribution by alpha-matrix (free of dislocation) calculated with the extrapolation at $\rho = 0$ $\mu$m$^{-2}$. The estimate of dislocation density of LAGBs (sub-boundaries) is derived from geometrical theory of dislocation indicating that LAGBs are alignments of dislocations.\(^{18}\) So, the estimate is versatile to various types of lattice. However, the constituent of LAGB is sensitive to processing method, e.g., ECAP, accumulative roll bonding (ARB), or rolling. After ARB or rolling, incidental dislocation boundaries (IDBs) were observed interconnecting the lamellae of geometrically necessary boundaries.\(^{19-21}\) In this study, more equiaxed grains and the absence of IDBs were identified for the condition of ECAP, consistent with the finding by Mishin and coworkers, as reported elsewhere.\(^{22}\)

Moreover, it is of interest to compare $k_{\text{BH}}$ with the slope ($k_T$) in the Taylor formula,\(^{23,24}\) i.e., $\tau_T = \sigma_0 + M_{\text{GB}}g_{\text{LAGB}}^{1/2}$, where $M$ is the Taylor factor ($\sim 3.0$), $\sigma = 0.2$, and $G$ is the shear modulus ($= 45.6$ GPa for titanium). The reason for $M = 3$ is explained by the analysis using the subroutine HKL Channel 5 TANGO. An average Schmid factor ($\langle \hat{n} \rangle$) on the preferred primary (110)[1120] slip system was determined as $\sim 0.31$ over all grains about the tensile axis. Thus, the Taylor factor that converts the critical resolved shear stress on the primary system, $\tau_{\text{CRSS}}$, into yield stress ($= M_{\text{CRSS}}$), was in practice estimated by $M = \langle \hat{n} \rangle^{-1.25}$. By inserting $\langle \hat{n} \rangle \sim 0.31$ to this equation, one arrives at $M \approx 3$. From this analysis, $k_T = M_{\text{GB}}g_{\text{LAGB}} = 9.49$ MPa $\mu$m, only about half the value of $k_{\text{BH}}$. Therefore, the Taylor strengthening was overestimated because it only represents the sum of the contributions by alpha-matrix ($\sigma_0$) and LAGBs. In fact, $\sigma_0$ has been properly estimated by the Bailey-Hirsch relation (Fig. 6), implying a total contribution by alpha-matrix, LAGBs, and HAGBs.

On the other hand, $\sigma_0$ can be theoretically evaluated by
\[
\sigma_y = \sigma_0 + \sigma_{\text{LAGB}} + \sigma_{\text{HAGB}}
\]
where, $\sigma_{\text{LAGB}}$ is the contribution by LAGBs;\(^{16,17,26}\) $\sigma_{\text{HAGB}}$ is the strengthening by HAGBs following the Hall-Petch equation: $\sigma_{\text{HAGB}} = k_{\text{HAGB}} \cdot d^{-1/2}$, where $k_{\text{HAGB}}$ ($\sim 0.33$ MPa $\mu$m$^{1/2}$) is the slope for Ti.\(^{25,27}\) Obviously, this theoretical estimate is in agreement with 0.2% proof stress ($\sigma_{0.2}$) (Fig. 6).

As resolved by EBSD, a grain consists of grain boundary (referred to as HAGB) and grain interior. The latter is constitutive of three components, i.e., the network of LAGBs (by which sub-grains are defined), randomly (freely) distributed dislocations (e.g., statistically stored dislocations, SSDs) within sub-grains, and very short-range obstacles in lattice including the Peierls-Nabarro stress, very fine particles (the Orowan mechanism), and interstitials such as O and N. However, it is hard to evaluate SSDs because they share very small intra-granular misorientation angle that is already beyond the display limit of EBSD. Actually, the contribution of SSDs is very small because usually they are afloat once an external stress is exerted. Rather, much more significant is the effect of LAGBs (the Taylor hardening). Also, the effect of HAGBs is synonymous with that of grain boundaries obeying the Hall-Petch relation (the hardening by grain refinement). Lastly, $\sigma_0$ is relevant to material category. For example, it mainly comes from interstitials (O and N) in alpha-Ti,\(^{19}\) or from the Peierls-Nabarro stress in very hard molybdenum disilicide (MoSi$_2$).\(^{28}\)

4. Conclusion

The microstructure of commercial purity alpha-Ti is characterized by substantial amounts of HAGBs and LAGBs after single- or multi-pass ECAP. The variation of yield strength obeys the Bailey-Hirsch relation, consistent with the sum of contributions by alpha-matrix, HAGBs, and LAGBs, respectively. This paper demonstrates that as a matter of fact, the consideration of the contribution of LAGBs is unavoidable to estimate the yield strength as a whole.

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