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Grain-structure development in heavily cold-rolled alpha-titanium

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ABSTRACT

High-resolution electron back-scatter diffraction (EBSD) analysis was employed to establish mircostructure evolution in heavily cold-rolled alpha-titanium. After thickness reductions of 75% to 96%, significant microstructure and texture changes were documented. The surface area of high-angle grain boundaries was almost tripled, thus giving rise to an ultra-fine microstructure with a mean grain size of 0.6 μm. Moreover, orientation spread around typical 'split-basal' rolling texture substantially increased. These effects were suggested to be related to the enhancement of pyramidal $\langle c+a \rangle$ slip.

1. Introduction

The mechanical properties of metallic materials may be improved considerably by microstructure refinement. To attain a very fine structure with a grain size less than one micrometer, a variety of techniques based on severe deformation at low temperatures, such as high-pressure torsion, equal-channel angular extrusion, and 'abc' forging, are commonly used [1]. For commercial-purity titanium, it has been found, however, that the refinement imparted by these techniques is comparable to that produced by conventional cold rolling [2]. Such a finding has spurred a renewed interest in microstructure evolution during cold rolling of this material.

Microstructure evolution during rolling to relatively low thickness reductions ($\leq$40%) has been shown to be markedly influenced by mechanical twinning [3–8]. Extensive development of both compressive $\{11\bar{2}2\}\langle 11\bar{2}\bar{3}\rangle$ and tensile $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ twins during rolling leads to rapid grain refinement [3–7]. On the other hand, grains favorably oriented for prism slip tend to remain untwinned [4,6]. This behavior gives rise to a bimodal grain structure consisting of fine-grained (twinned) and coarse-grained (untwinned) areas [6].

At relatively large thickness reductions ($\geq$40%), grain refinement suppresses twinning activity, and slip becomes the dominant deformation mode [9,10]. Microstructure formation at these large strains has been hypothesized to be largely controlled by the development of deformation-induced *dislocation* boundaries [5,8], but the details of this process are not clear. The objective of the present investigation, therefore, was to develop an understanding of microstructure evolution during cold-rolling of alpha titanium to large strains.

2. Material and procedures

The material used in the present investigation was commercial-purity titanium whose nominal chemical composition is given in Table 1. The as-received material was pre-conditioned by severe 'abc' forging at temperatures in the range of 600–400 °C followed by annealing at 800 °C for 1 h. This produced a fully recrystallized structure with a mean grain size of ~35 μm, a large fraction (85%) of high-angle boundaries, and a moderate crystallographic texture with (0002) basal planes inclined by ~45° to a rolling plane (supplementary data, Fig. S1).

Rolling samples measuring 30 (length) × 10 (width) × 3 (thickness) mm^3 were machined from the recrystallized material. They were rolled at ambient temperature using a thickness reduction

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per pass of 10%. to an overall total reduction of 25%, 50%, 75%, or 96%. using a rolling speed of 30 mm/s in a cluster mill with 65-mm diameter work rolls. To maintain consistency with the scientific literature, the typical flat-rolling convention was adopted in this work; i.e., the rolling, long-transverse, and thickness/normal directions were denoted as RD, TD, and ND, respectively.

Microstructure characterization was performed primarily via electron backscatter diffraction (EBSD) examination of the mid-thickness rolling plane (containing the RD and TD). For this purpose, samples were prepared using conventional metallographic techniques followed by long-term (24 h) vibratory polishing with a colloidal-silica suspension. EBSD analysis was conducted with a JSM-7800F field-emission gun, scanning electron microscope (FEG-SEM) equipped with a TSL OIM™ EBSD system. To examine microstructure and texture evolution at different scales, several EBSD maps were acquired for each material condition (Table 2). For each diffraction pattern, nine Kikuchi bands were used to index the orientation thus minimizing the possibility of errors. The average confidence index (CI) for EBSD maps ranged from 0.2 to 0.64 (Table 2). By comparison, experiments on face-centered cubic materials have shown that the fraction of correctly indexed patterns with CIs greater than 0.1 is 95% [11]. Non-indexed data points as well as points with low CI (≤ 0.1) were usually associated with grain-boundary regions. Grains comprising 3 or fewer pixels were automatically cleaned in the EBSD maps using the grain-dilation option in the TSL software. In addition, to eliminate spurious boundaries caused by software limitations, a lower-limit boundary misorientation cut-off of 2° was used.

A 15° criterion was used to differentiate low-angle boundaries (LABs) and high-angle boundaries (HABs). Because the microstructures developed at large strains are frequently characterized by a complex mixture of HABs and LABs, there is often confusion in the definition of grains. To clarify this issue, the term 'grain' in the present work was applied to denote a crystallite bordered by a continuous HAB perimeter. In all cases, the grain size was quantified by the determination of the area of each grain and the calculation of its circle-equivalent diameter, i.e., the so-called grain-reconstruction method [12].

To obtain an additional insight into microstructure evolution and material flow, the Vicker's microhardness was measured using a load of 500 g for 10 s. At least 25 measurements were made in each case to obtain an average value.

Table 1
Nominal chemical composition (wt%) of program material.

Al	Fe	O	Si	C	N	H	other	Ti
0.30	0.30	0.20	0.10	0.07	0.04	0.01	0.30	Bal.

Table 2
EBSD measurements.

Material condition	Scan step size (μm)	Map area (mm^2)	Average confidence index	Number of grains
Unrolled material	5.0 0.1	64.40 0.08	0.55 0.64	52,689 243
25% reduction	1.0 0.1	6.00 0.09	0.56 0.56	29,060 7970
50% reduction	0.5 0.1	1.00 0.09	0.41 0.45	65,304 16,052
75% reduction	0.5 0.1	1.03 0.02	0.18 0.32	130,940 7299
96% reduction	0.2 0.1	0.15 0.01	0.20 0.23	145,839 17,317

3. Results

3.1. Microhardness

The influence of rolling strain on microhardness is illustrated in Fig. 1a. It is seen that the hardness doubled after rolling to 96% thickness reduction. On the other hand, the approximate hardening rate $\delta Hv/\delta\varepsilon$ (where Hv is microhardness and ε is true strain) was found to drop rapidly after a true strain of 50% (Fig. 1b). This effect may be related to the suppression of twinning at large strains [13] as well as with texture changes, discussed below. Furthermore, the strain hardening rate approached almost zero at the maximum level of deformation (Fig. 1b). This behavior may promote instability of material flow and the formation of deformation/shear bands.

3.2. Structure morphology and grain size

Selected portions of low- and high-resolution EBSD maps illustrating the grain structures which developed during rolling to different thickness reductions are summarized in Figs. 2–4. In the orientation maps shown in Figs. 2 and 3b, grains are colored according to the orientation of the local sample normal direction relative to the crystal coordinate system. In the grain-boundary

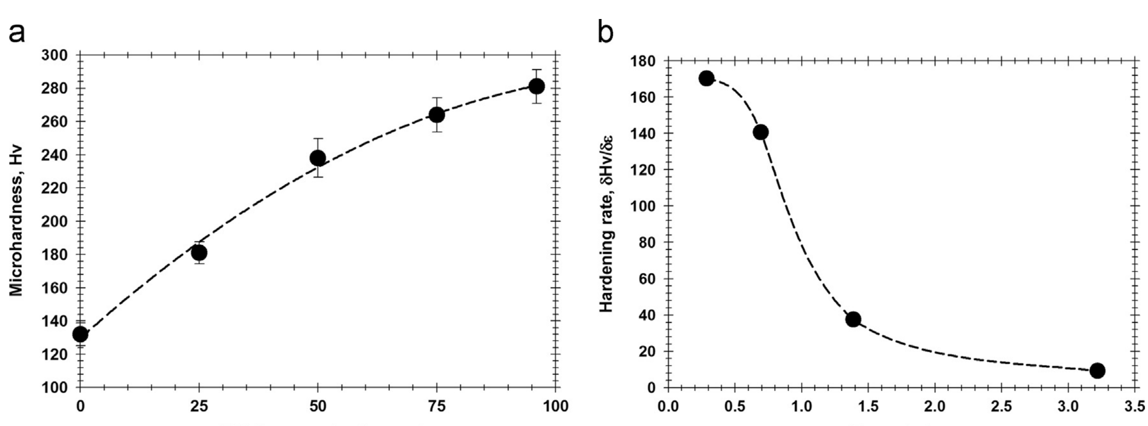

Fig. 1. Effect of rolling strain on microhardness (a) and hardening rate (b). In (a), error bars show standard deviation.