Accepted for publication in Zeitschrift fur Kristallographie (2005).

Burgers Vector Populations in hot rolled titanium determined by X-ray Peak Profile Analysis

I. C. Dragomir[1], G. A. Castello-Branco[2, 3], G. Ribarik[4], H. Garmestani[1], T. Ungar[4] and R. L. Snyder[1]

[1] School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332-0245, USA; [2] Materials Science and Engineering, University of Florida P.O. Box 116400, Gainesville, Florida 32611-6400, USA; [3] Centro Federal de Educacao Tecnologica, CSF-RJ, Rio de Janeiro, RJ, Brazil, 22710; [4] Department of General Physics, Eötvös University Budapest, H-1518, P.O.B 32, Budapest, Hungary.
[*] Contact author I. C. Dragomir, e-mail: iuliana.cernatescu@mse.gatech.edu.

Keywords: titanium, X-ray Peak Profile Analysis, dislocations, Burgers Vector Population

Abstract. Commercially pure alpha-titanium was deformed by hot rolling at 268° C for reduction levels of 40%, 60% and 80%. The dislocations densities and types in terms of Burgers Vector Populations at different deformation rates were established using X-ray Peak Profile Analysis. The dislocation model of anisotropic broadening of peak profiles was used to determination the Burgers vector distribution, and their evolution as the deformation proceeds to higher degrees in alpha-titanium samples presented here. It was found that the <a> dislocation type dominate at all deformation levels investigated in this study. The population of the <c+a> dislocation type is significant after 40% reduction and it becomes marginal for higher deformation rates. The <c> type dislocation population was found to be marginal at all deformation levels.

Introduction

Due to their outstanding properties titanium and titanium alloys are very attractive in a large variety of applications: aerospace, biomedical, and commercial applications [1, 2]. In order to improve and to control the mechanical proprieties of titanium it is important for understanding and explaining the formation of certain microstructure during deformation. On the other hand to explain and understand the deformation process information such as dislocation density, dislocation type and slip system activation are critical. Alpha-titanium has a hexagonal close-packed structure. Unlike the cubic crystals where usually one major type of slip system is activated during deformation in the hexagonal crystals, there are several fundamentally different slip systems types that are contributing simultaneously to the deformation [3-5]. The determination of the dislocations slip systems type by using conventional

techniques such as TEM is rather difficult when the dislocation density reaches values as high as $10^{10}/cm^2$. On the other hand throughout the sample preparation process necessary for the TEM experiments the original microstructure may change. Other alternatives on investigating the microstructure are X-ray and neutron diffraction techniques. Recent works have shown that by using the method of X-ray Peak Profile Analysis information about the dislocation densities and dislocation type can be extracted from the X-ray pattern [6-10].

In the present study commercially pure alpha-titanium was deformed by rolling at 268° C for the following reduction rates: 40%, 60% and 80%. X-ray Peak Profile Analysis technique was employed to determine the changes in dislocation density and dislocation types with deformation.

Experimental procedures

Titanium specimens were rolled at 268° C to a reduction of 40%, 60% and 80%. A Lindberg/Blue model BF51800 electrical furnace was used to heat the samples. In order to obtain a homogeneous deformation a step-size of 5%, for reduction was applied. After each pass of 5% the sample was immediately returned to the furnace for reheating and again deformed to an additional reduction of 5%. The procedure was repeated until the desired reduction level was achieved. After the final step the samples were cooled in air. In order to remove the formed oxide layer before the X-ray diffraction experiments each sample was chemically etched.

The diffraction profiles necessary for the X-ray Peak Profile Analysis were measured using an Alpha-1 PANalytical Diffractometer set up in Bragg-Brentano geometry. Symmetrical incident beam Johansson monochromator allowed only the Kα1 component of Cu radiation to be used. In order to reduce the instrumental broadening effect $1/4^o$ divergent slit, $1/2^o$ anti-scattering slit and 0.02 rad soller slits was used on the incident beam peath. On diffracted beam side a 5.0 mm anti-scattering slit and a 0.02 rad. soller slit was used. A mask of 5mm was used to adjust the size of the probing X-ray spot. The profile data acquisition was done using a solid-state position-sensitive ultra-fast detector (X'Celerator, PANalytical). For each sample the following reflections were measured: 0002, $10\bar{1}1$, $10\bar{1}2$, $11\bar{2}0$, $10\bar{1}0$, $10\bar{1}3$ and 0004. The instrumental broadening was measured using NIST SRM (660a). As the measured profile is a convolution of the physical with the instrumental profile the Stokes-correction based on the Fourier transforms of the profiles was used to determine the physical line profiles [11]. Background and instrumental profile correction were done with the MKDAT program described elsewhere [10].

Burgers Vector Population

The X-ray peaks are broadened due to the small crystallite size and to the crystal lattice imperfections in materials. It has been shown by Wilkens that in a dislocated crystal the distortion Fourier coefficients of the physical profiles can be written in terms of dislocation density and strain anisotropy. The latest can be taken in account by introducing the contrast factors of dislocation [12, 13]:

$$A_L^D = \exp\left[-\ 2\pi^2 L^2 g^2 \left(\rho \overline{Cb^2}\ /4\pi\right) f(\eta)\right], \qquad (1)$$

where g is the absolute value of the diffraction vector, L is the Fourier variable, ρ is the dislocation density, $\overline{Cb^2}$ is the average contrast factor of the dislocations present in the sample multiplied by the square of the dislocations Burgers vector and $f(\eta)$ is the Wilkens function [12]. In the case of hexagonal crystals the average contrast factor of dislocation can be written as follows [9]:

$$\overline{C}_{hk.l} = \overline{C}_{hk.0}\,[1 + q_1 x + q_2 x^2], \qquad (2)$$

where $x = (2/3)(l/ga)^2$, q_1 and q_2 are parameters which depend on the elastic properties of the material, $\overline{C}_{hk.0}$ is the average contrast factor corresponding to the *hk.0* type reflections, a is the lattice constant in the basal plane, g is the diffraction vector and l is the last index of the *hk.l* reflection for which the $\overline{C}_{hk.l}$ is evaluated. Since the contrast factor is a measurable parameter and it depends on the relative orientation of the diffraction vector and the line- and Burgers vectors of the dislocations, the X-ray peak profiles of the specimen can provide information about the dislocation types in the specimen. By inserting equation (2) in (1) it can be seen that only two parameters related to the contrast factor can be determined experimentally: q_1 and q_2. The measured values of q factors, $q_1^{(m)}$ and $q_2^{(m)}$, can be written in terms of numerically calculated average contrast factors of i number of slip systems considered in the evaluation:

$$q_1^{(m)} = \frac{1}{P}\sum_i h_i \overline{C}_{hk.0}^{(i)} b_i^2 q_1^{(i)}\ ,\ q_2^{(m)} = \frac{1}{P}\sum_i h_i \overline{C}_{hk.0}^{(i)} b_i^2 q_2^{(i)} \text{ and } \sum_i h_i = 1, \qquad (3)$$

where h_i is the fraction of dislocations which slips in the i-th slips system, $\overline{C_{hk.0}^{(i)}}$ is the theoretical value of the average contrast factor corresponding to the i-th slips system for the *hk.0* reflection, $q_1^{(i)}$ and $q_2^{(i)}$ are the q-factors of the i-th slip system, $P = \sum_i h_i \overline{C}_{hk.0}^{(i)} b_i^2 = \overline{b^2 C_{hk.0}}^{(m)}$ and $0 \le h_i \le 1$.

According to Honeycomb [14] and Klimanek & Kuzel [15] in a hexagonal crystal there are eleven possible slip systems. By taking this into account, the possibility of measuring q_1 and q_2 offers three independent equations and eleven unknowns [9]. This means that equations (3) can give an exact solution only by making certain assumptions about the activated dislocation slip systems. In the present work it is assumed that a particular Burgers vector type has random (or uniform) distribution in the different slip systems. In this case h_i terms in equations (3) become the fractions of dislocations with the three fundamental Burgers vectors types defined in the hexagonal systems: $\boldsymbol{b_1}$=1/3<$\bar{2}$110>, $\boldsymbol{b_2}$=<0001>, and

$\boldsymbol{b_3}$=1/3<$\bar{2}$ 113>. At the same time $\overline{C_{hk.0}^{(i)}}$, $q_1^{(i)}$ and $q_2^{(i)}$ become an averaged over the slip systems, each corresponding to the same Burgers vector type. The set of values for h_i obtained from equations (3) by using the above-mentioned assumption is called Burgers Vector Population [9]. In order to solve equations (3) the numerically calculated values of $\overline{C}_{hk.0}$, q_1 and q_2 for all slip systems are required. The theoretical values of $\overline{C}_{hk.0}$, q_1 and q_2 for titanium in the case of the most common slip systems were calculated and published previously in reference [9].

Results and Discussion

In the present work the Multiple Whole-Profile (MWP) fitting procedure was used to evaluate the q parameters in equation (2) and the dislocation densities from the X-ray diffraction pattern. In this procedure the Fourier coefficients of the measured physical profiles are fitted all at once by the product of the theoretical functions for size and strain caused by distortion in the crystal. In this evaluation process it is assumed that the peak broadening is caused by the smallness of the crystallites and by strain due to the dislocations. Details of the MWP method can be found in reference [10]. The usage of the average contrast factor of dislocations in the X-ray Peak Profile Analysis assumes that within a slip system type every slipping direction has the same probability. This assumption is not anymore valid when the specimen exhibits strong crystallographic texture. Due to the rolling deformation process the titanium samples studied here show a pronounced texture. In order to overcome this difficulty a random polycrystalline specimen was emulated for each sample by mixing the strongest diffraction peaks from the three different faces of the orthogonal sample to form a full diffraction pattern [16, 17]. These mixed patterns were then used in the MWP evaluation. The results for the dislocation densities and the arrangement parameter, M, are listed in table 1. for the titanium specimens deformed at 40%, 60% and 80% reduction levels. M is defined by Wilkens as the dislocation arrangement parameter [12]. The value of M gives the strength of the dipole character of dislocations: the higher the value of M, the weaker the dipole character and the screening of the displacement fields of dislocations [13]. The values of M in table 1. show that the dislocations in the titanium samples studied here are quasi-homogeneous distributed and exhibits a week dipole character. It can be observed from table 1. that the increment of ρ is small in accordance with a dynamic recovery effect, where dislocation annihilation that may occur due to the high deformation temperature. The solutions of the equations (3) for Burgers Vector Population are illustrated in figure 1. The results show that: I) in the specimen deformed at 40% reduction the <c+a> and <a> types of dislocation are dominating the dislocation population present in the sample; II) at higher deformation levels the population of <a> type dislocations increases and the presence of <c+a> dislocations decrease; III) at all deformation levels the fraction of <c> dislocations type is marginal. The results are in good agreement with the well known fact that dislocations with $\boldsymbol{b}$=<0001> Burgers vector are sessile, thus their presence is practically unchanged in the deformation range studied here [18]. The activity of <c+a> dislocations plays an important role in dynamic recovery. Screw dislocations of <c+a> type can move to the next slip planes by double cross slip followed by dislocation annihilation, which leads to a decrease of <c+a> dislocations at higher strains.

The results presented here are in good correlation with previous TEM studies, where the <a> dislocation types are most frequently observed in deformed titanium and <*c*+*a*> and <*c*> types are also reported [18, 19].

Table 1. Dislocation densities and arrangement parameter, M, obtained from MWP evaluation for different rolling reductions.

Rolling Reduction [%]	40% reduction	60% reduction	80% reduction
M	2.9	1.7	1.1
$\rho[1/cm^2]$	$5x10^{10} \pm 5\%$	$8x10^{10} \pm 10\%$	$10^{11} \pm 7\%$

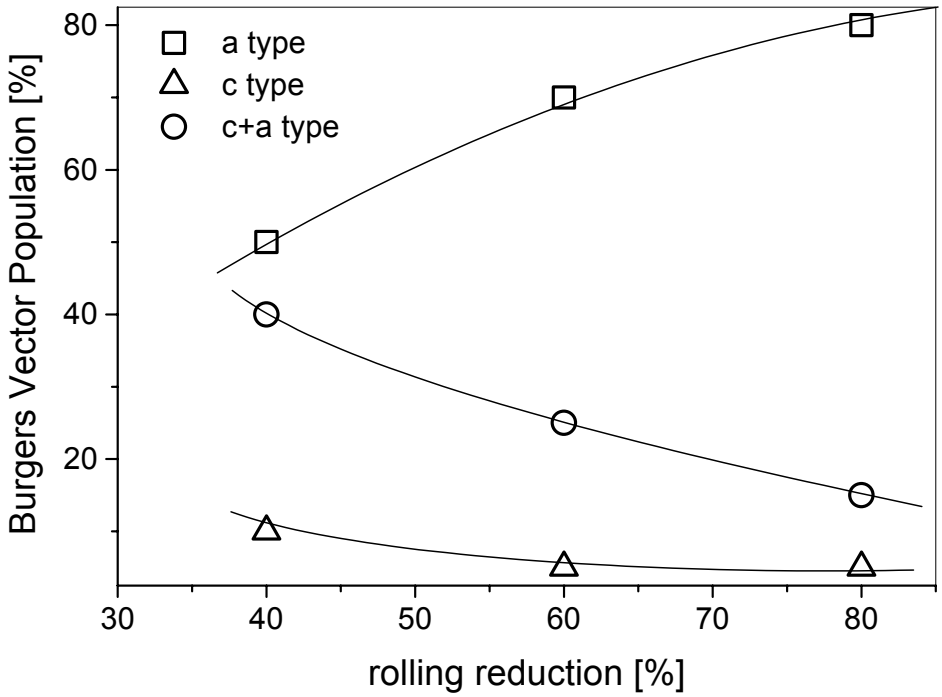

Figure 1. The h_i fractions (see the text) of the three fundamental Burgers vector types, <a>, <c> and <c+a>, as a function of rolling reduction percentage. Note that in the figure the solutions to equations (3), the h_i fractions, were transformed in percentages.

Conclusions

By using Peak Profile Analysis methods the evolution of dislocation density and the Burgers Vector Population has been evaluated as a function of rolling reduction in hot rolled titanium. It was found that the <a> types of dislocations are dominating the whole deformation range studied here. The percentage of <c+a> dislocation type is present significantly at 40% reduction level, but decreases as the deformation proceeds to higher values. The fraction of <c> dislocation type is minimal during the whole deformation process. It was also established that the dislocation density slightly increases as the reduction degree increases.

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Acknowledgements. We gratefully acknowledge the support of PANalytical Inc.