Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 25(2015) 3240−3246
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Effect of indentation size and grain/sub-grain size on microhardness of high purity tungsten

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Received 29 January 2015; accepted 13 May 2015

Abstract: Hardness of materials depends significantly on the indentation size and grain/sub-grain size via microindentation and nanoindentation tests of high-purity tungsten with different structures. The grain boundary effect and indentation size effect were explored. The indentation hardness was fitted using the Nix−Gao model by considering the scaling factor. The results show that the scaling factor is barely correlated with the grain/sub-grain size. The interaction between the plastically deformed zone (PDZ) boundary and the grain/sub-grain boundary is believed to be the reason that leads to an increase of the measured hardness at the specific depths. Results also indicate that the area of the PDZ is barely correlated with the grain/sub-grain size, and the indentation hardness starts to stabilize once the PDZ expands to the dimension of an individual grain/sub-grain.

Key words: high purity tungsten; indentation hardness; indentation size effect; grain boundary; plastic deformation zone

1 Introduction

Indentation tests have widely been used as an effective and economical method for measuring the mechanical properties of materials, including hardness and elastic modulus [1], elastic properties [2] and creep properties [3,4]. It has well been recognized that indentation hardness depends significantly on the indentation size or depth [5−12]. This phenomenon is referred to the indentation size effect (ISE) and is attributed to the evolution of geometrically necessary dislocations (GNDs) beneath the indenter, which gives rise to the strain gradients [13,14]. The plastically deformed zone (PDZ) and its evolution that are correlated to the hardness values were widely investigated [15−17].

In last several decades, many explanations have been proposed to describe the ISE, including the presence of oxides or chemical contamination near the surfaces [18], characteristic size plastic deformation [19] and a critical thickness layer [20]. Among all the explanations, Nix−Gao model [21] based on GNDs and Taylor dislocation model [22] might be the most important one, which can be expressed as

$$(H/H_0)^2 = 1 + (h^*/h) \quad (1)$$

where H is the real hardness, H_0 is generally called the macroscopic hardness, corresponding to the hardness that results from statistically stored dislocations (SSDs) in the absence of GNDs, and is always obtained when the indentation depth (h) becomes infinitely large; h^* is a characteristic length that characterizes the dependence of the hardness on the indentation depth [23]. It is suggested that in the Nix−Gao model the total dislocation density represents the total coupling between GNDs and SSDs, both of which play a significant role in the hardening mechanism. Although the Nix−Gao model could certainly agree well with many experimental results [21], some nanoindentation results [24−26] showed that it cannot predict the hardness with a small indentation size/depth precisely. FENG and NIX [25] suggested that the Nix−Gao model overestimated the hardness of MgO for small indentation and pointed out that it might be caused by a slight expansion of the PDZ [25]. It should be noted that the PDZ was not considered in the Nix−Gao model, in which the radius (a_{pz}) of the PDZ is regarded as the contact radius (a_c) between the indenter and the materials. DURST et al [24] also indicated that plastically deformed volume used to store the GNDs is larger than the volume defined in the Nix−Gao model, and they proposed a correction by considering the ISE, in which the radius of the PDZ is

Foundation item: Project (51174235) supported by the National Natural Science Foundation of China
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DOI: 10.1016/S1003-6326(15)63958-9

calculated by a_{pz}−fa_c approximately, where f is a factor dependent on the material. Moreover, ABU Al-Rub [27] formulated a micromechanical-based model that can be used to predict the ISE for both microindentation and nanoindentation simultaneously. This model is based on the evolution of GNDs beneath the indenter that is nonlinearly coupled (linearly coupled in the Nix−Gao model) with the evolution of SSDs through the Taylor's hardening law. A couple of parameters, H_y and β, were introduced by ABU Al-Rub et al [27].

$$[(H-H_y)/(H_o-H_y)]^{\beta}=1+(h^*/h)^{\beta/2} \quad (2)$$

where H_y is the hardness due to the initial yield stress (friction hardness) and β is considered as a material constant (interaction coefficient). β is normally used to assess the prediction sensitivity that the coupling between the SSDs and GNDs is enhanced during the plastic deformation process. It should be noted that if both SSDs and GNDs are coupled in a linear sense, i.e. β=2 and the friction hardness is neglected, i.e. H_y=0, the model of ABU Al-Rub et al [27] (Eq. (2)) returns to the commonly-used Nix−Gao model. However, all these models do not consider the interaction between the dislocations and grain boundaries, except for the work of YANG and VEHOFF [28], in which it was suggested that for large grains the hardness always decreases with increasing the indentation depth by using the nanoindentation tests in the center of individual grains to study the ISE and grain size effect (GSE). For small grains the hardness exhibited a behavior opposite to that of the coarse grains because of the grain boundary effect (GBE). In this work, the indentation hardness of high purity tungsten (W) with different structures was performed, in order to further understand the ISE and GBE during indentation.

2 Experimental

High purity W (99.95%) with two types of structures (fully dense coarse-grained structure and fine-grained structure with residue pores after sintering) was received from market and fabricated by using W powder with spark plasma sintering (SPS) technique at a sintering temperature of 1700 °C. All the specimens were mechanically polished by using emery paper and fine diamond (particle size of 0.5 μm) to meet the requirements for indentation tests using an ultra nano- (with effective radius of about 50−100 nm at the tip) and micro-indentation tester with Berkovich tips (with an effective radius of 100−200 nm at the tip). The indents were displacement/depth-controlled and the indentation hardness was obtained by Oliver and Pharr method [1]. Specifically, all the samples were pressed with the same penetrating rate of 5 nm/s to different maximum displacements, followed by holding at the maximum displacement for 10 s, and then the load was completely unloaded in 10 s. Three intents were tested for each process and the average value was used to plot the load−depth (p−h) curves and to calculate the indentation hardness. It should be noted that the W surface obtained through mechanical polishing is rather even. This can be well demonstrated by the fact that the p−h curves look the same as the depth increased (see Fig. 1). No electrolytic polishing was performed to remove the mechanical surface layer from the sample based on the following considerations. Firstly, even though the working hardened layers produced by mechanical polishing in W can lead to an increase in the hardness of the surface layer to some extent, the surface stress layer produced by friction between the W and the emery paper is extremely thin due to the high hardness of W [29]. Thus, the effect of surface stress and thin harder surface layer on the hardness was neglected in this work. Secondly, it is generally recognized that exposure to corrosive environments may form a chemically modified layer on the surface, which can influence the hardness if it is sufficiently thick [5]. To exclude this factor, only mechanical polishing without any electrolytic polishing was used to obtain the required surface. Macroscopic hardness of the full densification structured W was tested by using 500RMA hardness testing machine, and the microstructures were characterized by using a FEI Nova nano230 scanning electron microscope (SEM) and electron back-scattering diffraction (EBSD) technique in conjunction with SEM.

3 Results and discussion

Figure 1 shows the load−depth curves of the full densification structured W under nanoindentation and microindentation, respectively. The maximum depths for nanoindentation and microindentation are in the range from 50 to 600 nm (Fig. 1(a)) and from 600 to 5000 nm (Fig. 1(b)), respectively. It can be seen from Fig. 1 that the p−h curves appear to be the same with increasing the depth, particularly for microindentation (see Fig. 1(b)). Good repeatability of the p−h curves under the same indentation process validates the effectiveness of the experimental results. Most importantly, when more attention is paid to the very beginning of the p−h curve (rectangle section in Figs. 1(a) and (b)), it is found that no pop-in behavior was observed. It is generally recognized that the pop-in behavior at the beginning of the loading process is the result of the transition from elastic to plastic deformation and is attributed to the homogeneous nucleation of dislocations during indentation [30−32]. A distinct pop-in will be detected if indentation is performed on the perfectly prepared