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Incipient plasticity and dislocation nucleation in body-centered cubic chromium

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ABSTRACT

In this study, we conducted nanoindentation tests to explore the incipient plasticity (or onset of yielding) in a fully annealed coarse-grained Cr at loading rates between 0.02 and 2 mN/s and temperatures ranging from 295 to 423 K. The maximum shear stress at the first indentation pop-in were evaluated and found comparable to the theoretical shear strength for the yielding of dislocation-free Cr. The activation volume and activation energy for the onset of yielding were also evaluated using Schuh's statistical model, and further compared with that anticipated for homogeneous and heterogeneous dislocation nucleation processes. We concluded that heterogeneous nucleation was the favorite mechanism and the type of point defect that induced dislocation nucleation appeared to be interstitials rather than vacancies in the body-centered cubic Cr.

1. Introduction

A sudden displacement excursion or "pop-in" event during an indentation test was first observed in metals in 1968 [1] and was attributed to the onset of plasticity. In past decades, nanoindentation technique has been widely used to explore this phenomenon and its correlation with the incipient plasticity of crystals [2–6]. The advantages of having high resolution in both displacement ($\sim$nm) and load ($\sim\mu N$) enables a nanoindentation system to capture pop-in events while probing a small volume of sample that is free of lattice defects, in particular, dislocations. In such case, the incipient plasticity triggered by the nucleation of dislocation [7] can be described as a stress-assisted, thermally activated process [8,9]. Specifically, the energy barrier for the nucleation process can be overcome by thermal fluctuation assisted by applied stresses. Michalske et al. [10] and Chiu et al. [11] suggested the formation of dislocation loop was energetically favorable in a stressed volume based on the conventional continuum theory. The view of homogeneous dislocation nucleation was also supported by several atomistic simulations [12–14]. Bei et al. [15] found a higher theoretical strength for Mo alloy determined by nanoindentation than that by pillar compression, as a result of easier heterogeneous dislocation nucleation from free surfaces or corners in a rectangular pillar sample.

On the other hand, Mason et al. [16] developed a statistical approach to analyze the pop-in events in single-crystal face-centered cubic (fcc) Pt and obtained the activation volume (0.5 b^3) and activation energy (0.28 eV) for incipient plasticity. These activation values were distinctively lower than those expected for homogeneous nucleation [14,17], and they suggested possible heterogeneous dislocation nucleation from point-like defects. Relatively low activation parameters were also observed in GaN (0.85 eV) [18] and hexagonal-close-packed (hcp) Mg (0.2 b^3) [19]. Recent atomistic simulations showed that the presence of point-like defects (e.g., vacancies, di-vacancies, tri-vacancies, and interstitial atoms [20–22]) could play significant roles in dislocation nucleation. For example, Salehinia et al. [20] demonstrated in fcc-Ni that the presence of point-like defects could drastically reduce the stress required for pop-ins, although the exact atomic process for dislocation nucleation was not clear. Compared with fcc-metals [2,3,23,24], studies of incipient plasticity of the body-centered cubic bcc-metals [25,26] are limited. It will be intriguing to evaluate the activation parameters for the incipient plasticity of bcc-metals to shed lights on the dislocation nucleation, since the nucleation kinetics in bcc-metals are expected to be different from that in the fcc-metals, in which partial dislocations tend to dominate [4]. In this study, we carry out nanoindentation tests on bcc-Cr and explore the nature of incipient plasticity in the material.

2. Materials and experimental

In this study, a piece of Cr sample was sliced from a coarse-grained Cr bulk prepared by arc melting method (purity > 99.99%). The surface of the Cr sample was ground using 1200 and 2400-grit SiC, and then mechanically polished using alumina of 0.06 μm in

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grit size. To reduce existing dislocations and minimize possible surface damage resulting from the mechanical polish, the Cr specimen was initially sealed in a fused silica tube filled with pure Ar gas and, then, annealed at 1250 °C for 4 h [27]. As the final procedure, the annealed Cr was additionally electropolished in a solution (H_3PO_4:H_2SO_4=4:1) to remove possible oxidization layer during annealing. To measure the grain size and dislocation population in the Cr sample, an etchant consisting of 6 wt% nitric acid, 16 wt% ceric ammonium nitrate, and 78 wt% water was used and an etched surface is shown in Fig. 1. Dislocation density is estimated from the number of etched pits to be about 1×10^{10} m^{-2} and the grain size is determined to be about 0.5 mm.

Nanoindentation was performed in only one single Cr grain, the crystal orientation of which was determined by electron backscattering diffraction (EBSD), as indicated in the inverse pole figure (inset of Fig. 2). Indentation tests were all conducted on a Triboindenter (Hysitron, Minneapolis, MN). A Berkovich indenter tip supported by a low-thermal-expansion shaft was used in this study. The tip radius of this Berkovich indenter was estimated from the calibration tests on *W* single-crystal to be 210 nm. Load-controlled indentation tests at various loading rates (0.02 mN/s, 0.1 mN/s, 0.5 mN/s, 2 mN/s) were conducted to examine the effect of loading rate on incipient plasticity. Indentation tests were also performed at elevated temperatures (295 K, 333 K, 373 K, 423 K) at a constant loading rate of 2 mN/s to access the activation enthalpy for the incipient plasticity. A maximum load of 1 mN was used in all tests. To carry out indentation tests at elevated temperatures, a commercial heating stage was installed and the displacement transducer was shielded from the hot specimen and stage with a cooled copper fixture [28]. Before indentation, the sample was heated to the set temperature and held for at least 30 min and the tip was, then, brought to contact with the sample surface and equilibrated for another 30 min. The temperature of the specimen surface was monitored and controlled (± 0.2 K) using a J-type thermocouple in direct contact with the sample surface. At least 60 indentations were performed at each test temperature and loading rate. The distance between adjacent indents was set at 10 μm to minimize possible overlap of the indented zone. Thermal drift was estimated to be within 0.5 nm/s.

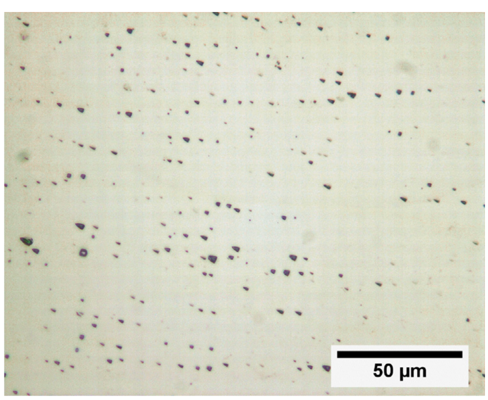

Fig. 1. Etched pits on the surface of the annealed Cr sample gives an averaged dislocation density of about 1×10^{10} m^{-2}.

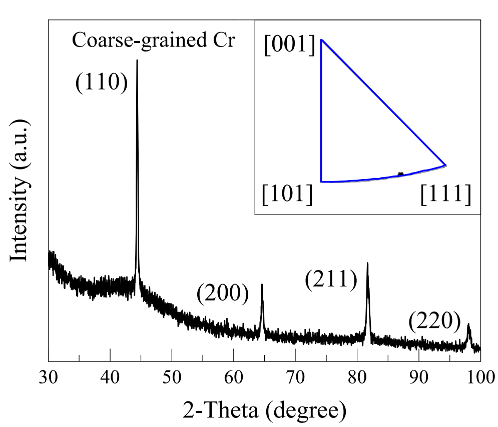

Fig. 2. XRD pattern of coarse-grained Cr and the crystalline orientation of the grain studied.

3. Results

3.1. Pop-in event and effect of surface condition

Representative *P–h* curves with apparent pop-in events during indentation are presented in Fig. 3(a). At a pop-in event, a sudden strain burst (or sink in of the indenter) at the constant load is generally recognized as the onset of dislocation motion (or incipient plasticity). In other words, it is the indicative of the transition from elasticity to plasticity. The elastic part of the *P–h* curves can be well described by the Hertzian relation [29]: $P=(4/3)E_rR^{1/2}h^{3/2}$, where in the equation R is tip radius of the indenter, h is the penetration depth, and E_r is the reduced Young's modulus. Insert proper parameters into the Hertzian relation, namely, $E_r=[((1-\nu^2)/E)_{Diamond}+((1-\nu^2)/E)_{Cr}]^{-1}$, where the Poisson's ratio ν for Cr is 0.21 [30], and the diamond properties [31] are E=1141 GPa and ν=0.07, we can plot a P–$h^{1.5}$ graph using a collection of *P–h* pairs at the pop-in obtained from a series of 184 indentations; this is shown in Fig. 3(b). The slope of the fitted curve is 4.509, from which E_r is deduced to be 236.12±1.59 GPa. The elastic modulus of Cr sample is thereafter determined to be 284.30± 1.52 GPa, which is slightly higher (2%) than the literature value of 279 GPa [32].

It has been reported that the occurrence of pop-in events is highly sensitive to the surface condition of a test sample [26,33]. In the present study, it is also demonstrated in Fig. 3(a) in which load–displacement (*P–h*) curves obtained from Cr samples prepared by both mechanical polishing and electropolishing are shown. It is evident that pop-in events occur in the electropolished Cr, but are absent in the mechanical polished sample. Similar results were also observed in single-crystal Mo [33], indicating that a mechanically damaged surface layer can artificially eliminate the pop-in events during indentation.

It is worthy to note that the size of the plastic zone beneath the Berkovich tip is scaled with the contact radius a (=71 nm), which is $a=(Rh)^{1/2}$, according to the Hertzian theory [29]. In the current Cr sample, the dislocation spacing, estimated from the dislocation density (Fig. 1) is about 10 μm, which is much larger than the size of plastic zone. It suggests that the observed pop-in is unlikely triggered by preexisting dislocations.

3.2. Effect of loading rates

The cumulative probability f is defined as the probability of pop-in event occurring under a given load/stress for a group of indentation tests. To perform the statistical analysis with the pop-in data obtained at different loading rates, cumulative probability of pop-in events f as a function of τ_{max}/μ, the maximum shear stress τ_{max} normalized by the shear modulus of Cr: μ, is plotted in Fig. 4. The maximum shear stress beneath the indenter, from the Hertzian contact theory, is

$$\tau_{max}=\frac{0.47}{\pi}\left(\frac{4E_r}{3R}\right)^{2/3}P^{1/3} \quad (1)$$