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Thermally activated dislocation plasticity in body-centered cubic chromium studied by high-temperature nanoindentation

In-Chul Choi, Christian Brandl*, Ruth Schwaiger

Institute for Applied Materials, Karlsruhe Institute of Technology (KIT), Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

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ABSTRACT

The deformation behavior of Chromium (Cr) was investigated with the goal to understand and quantify the thermally-activated dislocation plasticity and the transition to the temperature-/rate-independent regime in body-centered cubic (*bcc*) metals. High-temperature nanoindentation experiments were utilized to characterize the deformation behavior of *bcc* Cr from room temperature to 673 K. To validate the indentation method itself at elevated temperatures, we systematically studied the temperature-dependent indentation elastic modulus, which clearly shows a discontinuity at the magnetic phase transition at 308 K, which is quantitatively consistent with literature data. We characterized the kinetics of dislocation plasticity by analyzing the strain-rate sensitive behavior of the hardness at different temperatures. The observed signatures of the plastic relaxation mechanisms are discussed in the context of screw dislocation mobility governed by thermally-activated kink-pair nucleation, kink-drift or dislocation-impurity interaction.

1. Introduction

The materials science challenge for energy conversion systems is the development of novel structural metallic materials, which maintain sufficient strength and stability under challenging operating conditions, in addition to sufficient toughness and ductility at ambient temperatures to ensure structural integrity. For example, higher operating temperatures in gas turbines would improve both performance and efficiency [1], which, however, pushes the structural materials to new extremes in temperature, corrosion, and mechanical loading. However, the melting temperatures of currently used Nickel-based superalloys limit the operation temperatures in the energy conversion process. A notable improvement in efficiency requires novel high-temperature (HT) structural materials. Candidate materials that easily come to mind are refractory metals because of their high melting points [2]. While chromium should in principle be an excellent material for high temperature oxidation because of its oxidation and corrosion resistance, it is generally not used as base metal but rather as a reinforcing material due to its low fracture toughness and low ductility at room temperature (RT) [3]. Numerous studies have focused on improving the ductility and toughness of Cr [3] through, for example, purification [4,5], alloying [6], and microstructure refinement [7]. However, before reliably using Cr-based alloys as structural components in HT applications, it is necessary to fully understand the deformation behavior and mechanisms of body-centered cubic (*bcc*) Cr at both RT and HT, particularly in the context of defect and impurity contents.

The most prominent characteristics of *bcc* metals are the rapid decrease in the flow stress with increasing temperature [8,9] and the pronounced rate-sensitive deformation behavior observed at RT, which is closely related to the limited mobility of screw dislocations due to their non-planar dislocation core structure in *bcc* metals [8–15]. *Bcc* metals behave clearly differently from face-centered cubic (*fcc*) metals, whose mechanical properties become time-dependent at elevated temperatures. Only few experimental studies of the mechanical behavior have focused on pure Cr [16–22]. Moreover, the relationship between the mechanical behavior and the deformation mechanisms as well as the interplay with impurities have not yet been established. Recently, this fundamental question of temperature- and rate-dependent deformation behavior of Cr [19,23] has re-emerged, probably also because advanced mechanical characterization techniques have become readily available. For example, high temperature nanoindentation [24] allows the local probing of single crystal behavior of a usually polycrystalline sample thereby uncovering the intrinsic deformation mechanisms.

* Corresponding author.
E-mail address: christian.brandl@kit.edu (C. Brandl).

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2. Experimental details

The experiments were conducted on commercial polycrystalline Cr with high purity (>99.8%, procured from Goodfellow). The amount of impurities was carefully analyzed including nitrogen and oxygen by carrier gas heat extraction (TC600, Leco), and carbon and sulfur by combustion analysis (CS600, Leco). The results of the chemical analysis are summarized in Table 1.

The microstructure of the specimen was characterized by electron microscopy (SEM) and electron backscatter diffraction (EBSD) (Nova NanoLab 200, FEI Inc., Hillsboro, OR, equipped with a HKL EBSD-system, now: Oxford Instruments). The samples were mechanically ground and polished with silicon carbide paper and diamond suspensions, respectively. Subsequent electrolytic polishing removed the deformed surface layer resulting from the mechanical preparation. Fig. 1a shows representative microstructures of the Cr sample with equiaxed grains having an average grain size of ~35 μm and no preferred crystallographic orientation.

In this study, nanoindentation with a sapphire Berkovich indenter tip was performed using a Nanoindenter G200 XP (Keysight Technologies, Chandler, AZ) with continuous stiffness measurement (CSM) and a laser heating stage (Surface Tec, Hückelhoven, Germany) for elevated temperature experiments. In this setup, both the indenter tip and the sample are heated independently [24], which minimizes thermal drift effects and establishes a well-defined and stable contact temperature. The thermal drift of the system was less than 0.1 nm/s at all temperatures. The correction of the load frame stiffness and the determination of the tip area function were performed according to the procedure outlined by Oliver and Pharr [25]. The values of hardness were calculated from the measured load divided by the projected tip area at the respective penetration depth of the indenter into the Cr sample. All indents were positioned inside individual grains (cf. Fig. 1b). Different types of experiments were conducted, i.e. constant indentation strain rate (CSR) as well as strain rate jump (SRJ) tests, while the temperature was varied from room temperature to 673 K. The morphology of the imprints was evaluated by SEM and an UV-laser scanning microscope (LSM, VK-9710K, Keyence, Osaka, Japan).

We performed a series of HT nanoindentation experiments on high-purity Cr to understand and quantify temperature-dependent deformation behavior. The results are discussed in the context of screw dislocation movement governed by a thermally-activated process.

3. Results

3.1. Temperature-dependent elastic properties

The Cr sample was loaded to a maximum indentation depth of ~2 μm at constant indentation strain rate $\dot{P}/P$ of 0.05 s^{-1}, where P is the indentation load. The temperature T was varied from 298 K to 673 K in steps of 2.5 K (298 K $\le T \le$ 323 K), 5 K (328 K $\le T \le$ 373 K), 10 K (383 K $\le T \le$ 473 K), and 25 K (498 K $\le T \le$ 673 K) to cover the magnetic phase transition and a finite temperature regime, in which plasticity does not show any rate dependence. At even higher temperatures creep effects are expected to dominate the

Table 1
Impurity content of the Cr sample.

Element, wt. %	C	N	O	S
Average	0.0056	0.0138	0.176	<0.0005
Standard deviation	0.0005	0.0002	0.002	
Limit of quantification	0.001	0.0003	0.01	0.0005

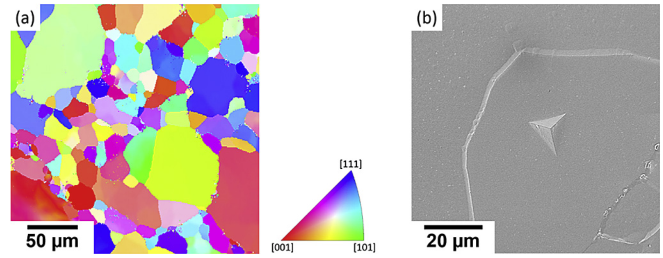

Fig. 1. Microstructure of the polycrystalline Cr: (a) EBSD image exemplifying the randomly oriented grains, and (b) SEM micrograph of an indent positioned in the center of a grain.

plastic deformation. The elastic modulus E was determined according to

$$E = \frac{(1-\nu^2)\cdot E_i E_r}{E_i - \left(1-\nu_i^2\right)\cdot E_r} \quad (1)$$

where ν is Poisson's ratio of the specimen, E_i and ν_i are the elastic modulus and Poisson's ratio, respectively, of the sapphire indenter, and E_r is the reduced modulus. The values $\nu = 0.24$ and 0.21 were assumed for sapphire [26] and Cr [18], respectively, which are well-approximated as temperature-independent [27]. The elastic modulus of sapphire is temperature dependent and has been well-characterized by a resonance frequency technique [28]. For the analysis of our data, the values of E_i were incorporated according to [28].

Fig. 2 shows averaged load-displacement curves obtained at 298 K, 473 K, and 673 K at $\dot{P}/P = 0.05$ s^{-1}. The maximum load clearly decreases with increasing temperature. The elastic modulus was constant over the indentation depth as shown in the inset for 673 K. The modulus values were averaged in the indentation depth range of 1500–2000 nm for the different temperatures and are compared with existing temperature-dependent elastic constants, i.e. from dynamic tensile tests of polycrystalline Cr [29], self-oscillatory system of polycrystalline Cr [30], and ultrasound measurements in Cr single crystals [31] (cf. Fig. 3). The absolute values of E at a given T obtained in this study, are in general lower than those of polycrystalline Cr [29,30] and single-crystalline Cr

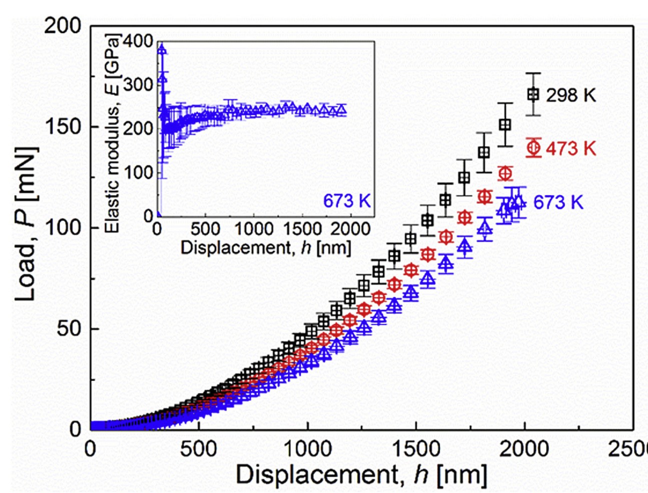

Fig. 2. Averaged load-displacement curves at $\dot{P}/P = 0.05$ s^{-1} at different temperatures. The average of six tests is shown with error bars representing one standard deviation. The elastic modulus is constant over the indentation depth as shown in the inset for 673 K.