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Low temperature in-situ micro-compression testing of iron pillars

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ABSTRACT

An in-situ nanomechanical cooling system has been developed to study the temperature dependency of local mechanical properties and slip behavior of bcc α-Fe. Uniaxial compression tests with Focused Ion Beam (FIB) fabricated pillars with a diameter of 1 µm, were performed in the single slip orientations $[\bar{2}35]$ and $[\bar{1}49]$ at room temperature and 198 K. The testing was conducted inside a Scanning Electron Microscope (SEM) equipped with a nanoindenter. Slip trace analyses revealed occurrence of slip in the $\{112\}<111>$ family of slip systems for $[\bar{2}35]$ pillars at both room temperature and 198 K while the predominantly slip systems governing the deformation on $[\bar{1}49]$ pillars were $\{110\}<111>$ for both test temperatures. The stress-strain response showed an increased strength with decreasing temperature for the $[\bar{2}35]$ pillars, in contrast to $[\bar{1}49]$ pillars, where only a weak temperature dependence is observed. Furthermore, for $[\bar{2}35]$ pillars, the appearance of slip is less prominent at 198 K, indicating that the temperature strongly influences the relative motion of screw and edge dislocations. Molecular Dynamics (MD) simulations performed at 15 K and 300 K, was used to study dislocation mechanisms for the two orientations. $[\bar{1}49]$ pillars exhibit a change in deformation mechanisms at low temperature and the evolution of dislocation density during deformation, display distinct differences for the two loading orientations.

1. Introduction

High Strength Low Alloy (HSLA) steels are used in a number of structural applications in the arctic offshore industry. However, the ductile-to-brittle transition in steels makes it vulnerable at low temperatures. In arctic conditions this important concern may lead to catastrophic failures, since structures operate at subzero temperatures. In practice, the mechanical properties of steels are usually determined by testing macroscopic specimens within the ductile-to-brittle transition temperature regime. Macroscopic tests have revealed a shift in mechanical properties at lower temperatures. This accentuates the need to understand how the mechanical properties depend on temperature and crystallographic orientation at the micro level, as this also strongly influences the cleavage fracture toughness. Bcc iron is widely used in structural applications as the main component in steels, and it is important to understand the fundamental physical mechanisms of the mechanical properties. The micromechanical testing and atomistic modeling is now being applied as new tool to gain more insight in these governing deformation mechanisms of the critical microstructural constituents.

Understanding material deformation and failure mechanisms at micro- and nanoscale has become a major interest in material science for a large number of applications, and the rapid developments within nanotechnology have made it possible to get a detailed characterization of materials with new available tools such as Focused Ion Beam (FIB) and nanoindentation. A large amount of publications regarding small-scale plasticity are available today. In particular, the "size effect" has been observed in metallic materials, where the flow stress usually increases with decreasing size [1–5]. An efficient way to quantify the mechanical behavior on the micrometer scale is by microcompression testing [1,6–8]. Experiments on body-centered cubic (bcc) and face-centered cubic (fcc) pillars have reported a more pronounced size effect for fcc metals compared to bcc metals correlated to the different contribution of lattice resistance to plastic flow [5,9–11]. There are fundamentally different plasticity mechanisms for bcc metals compared to fcc, that arises from the intrinsic behavior of screw dislocations which dominates the plastic deformation in bcc metals [12]. Bcc metals have a complex non-planar dislocation core with a threefold symmetry, allowing screw dislocations to interact with several planes of the $<111>$ zone axis. As a consequence, the bcc metals exhibit complicated slip modes, i.e. breakdown of Schmid law, and a strong temperature -and orientation dependency of yield -and flow stresses [5,9,12–15].

A recent investigation [9] of fcc Au and bcc Mo, reported a nearly twice as large size effect for Au than Mo. Dislocation Dynamics (DD) and Molecular Dynamics (MD) simulations [9,16] has verified these

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findings and show that different plasticity mechanisms are operating in the two metals. In fcc metals, the dislocations quickly leave the crystal, resulting in a dislocation-starved condition that requires high stresses to nucleate new dislocations. While in the bcc crystal, a dislocation-starved condition are unlikely due to a longer residence time of dislocations in the sample, and the ability of a single dislocation to multiply before it exits the pillar surface. Moreover, the weaker size effect observed in bcc metals is found to correlate with their critical temperature, T_c [11,15], i.e. temperature at which screw and edge dislocation obtain equal mobility and flow stress is nearly insensitive to temperature and strain rate [17]. Below this temperature, the mobility of screw dislocations decreases in contrast to edge dislocations and their mobility is a function of the test temperature relative to T_c [15]. Micropillar tests by Schneider et al. [11] showed that the critical temperature was found to have a strong influence on the deformation morphology for larger pillars (> 1 µm). The bcc pillars with high T_c, i.e. W (T_c=800 K) and Mo (T_c=480 K) consisted of wavy slip traces after deformation, typically related to cross-slip of screw dislocations. While those with lower T_c, like Nb (T_c=350 K) and Ta (T_c=450 K) displayed defined and localized slip behavior, indicating activation of a few slip systems [11].

The existing studies on bcc metals have revealed valuable insight into the plasticity mechanisms of small-scale bcc samples. However, the different studies have tried to understand the role of size effects in bcc metals with dependence of their critical temperature, more than the plasticity mechanisms relative to the test temperature and orientation. The majority of pillar compression experiments have been conducted at room temperature due to less demanding equipment as subzero testing demands vacuum-conditions to avoid the influence of ice deposits. Development of experimental techniques revealing small-scale plasticity at lower temperatures is necessary in order to address local properties causing failures in materials. So far, only a few studies have performed micropillar compression tests of bcc pillars at different temperatures. Abad et al. [18] investigated the plasticity structures of Ta and W submicro and micropillars systematically from room temperature to 673 K. Schneider et al. [19] examined the size dependent deformation behavior of Mo at 300 K and 500 K. Both studies suggested that at higher temperature, the plasticity mechanisms were consistent to what is commonly seen in fcc pillars, as a result of the increased mobility of screw dislocations. Only one study, to our knowledge, have performed compression test of bcc pillars at subzero temperatures: Lee et al. [20] studied the temperature-dependent size effect on the mechanical behavior of W and Nb pillars at room temperature and 165 K. Together with dislocation dynamics simulations it was suggested that the surface-controlled dislocation multiplication was easier achieved at low temperature when screw dislocation mobility was lower. Moreover, a significant larger lattice resistance and yield stresses occurred at low temperatures.

The effect of temperature on the mechanical strength has not yet been investigated in α- Fe microsamples. To the authors knowledge, this work represents the first attempt to elucidate the temperature-dependent plasticity mechanisms and mechanical behavior on FIB-machined α-Fe pillars at subzero temperatures. We report results of uniaxial compression testing of 1 µm diameter single crystalline Fe pillars at 198 K and room temperature. A cooling system interfaced with an Environmental SEM (ESEM) was developed, with a liquid nitrogen source connected to a Hysitron PI-85 nanoindenter. Suitable grains were identified with EBSD analysis and subsequently $[\bar{2}35]$ and $[\bar{1}49]$ oriented Fe pillars were fabricated by Focused Ion Beam (FIB)-milling. The temperature and orientation dependence was further evaluated by using MD simulations to provide insight into the complex behavior of dislocations and mechanisms related to the deformation process at a fundamental level. We further find that loading orientation governs the competition between twinning and dislocation slip for the MD simulated Fe nanopillars.

2. Experiments and method

2.1. Specimen fabrication

A high purity (99.99%) iron sample with the dimensions 8×8 mm^2, was mechanically polished, heat-treated and subsequently electrochemically polished to remove surface deformations. A more detailed description of the sample preparation is found in [21]. Cylindrical micropillars with a top diameter of 1 µm, were milled out using Ga$^+$ ions with 30 keV energy in a FIB/SEM DualBeam™ system (FEI Helios NanoLab™). A milling current of 90 pA was used for a final polish of the pillar surface, resulting in a taper angle between 2° and 3.5° and a h/d_t ratio (pillar height (h) and the top diameter (d_t) of the pillar) of 3.5. Prior to pillar fabrication, Electron Backscatter Diffraction (EBSD) was used to identify grains of interest for location of the micropillars. The two loading orientations are visualized in the standard triangle as solid circles in Fig. 1a and in Fig. 1b and c, oriented in the unit cell.

2.2. Micromechanical testing at low temperatures

Uniaxial in-situ compression tests were performed inside a FEI Quanta Environmental SEM (ESEM) at room temperature and 198 K with a Hysitron PI-85 nanoindenter applied with TriboScan software. In order to perform the compression tests at low temperatures, a cooling nanomechanical testing system was developed. The cooling system consist of the PI-85 nanoindenter, interfaced in the ESEM and a liquid nitrogen container mounted on a SEM port further connected to the sample and indenter tip via a copper coldfinger (thermal conductivity 385 W/mK), in order to cool down the sample and tip simultaneously to ~198 K, Fig. 2. The temperature was measured by attaching a thermocouple (type K) directly to both the samplestub and the tip. The thermocouples were connected to a temperature-measuring instrument via a thermocouple extension and compensation cable (TC cable) fed through a SEM flange with a vacuum compatible feedthrough part for the TC cables. In order to minimize thermal drift, experiments were performed after the temperature had been stabilized.

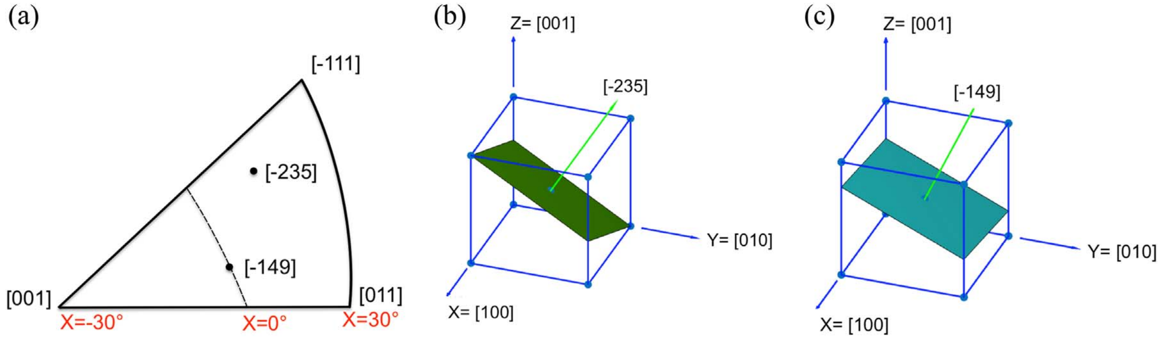

Fig. 1. (a) Standard triangle where the uniaxial loading orientations investigated are marked with black dots and in the unit cell showing the (b)$[\bar{2}35]$ and (c) $[\bar{1}49]$ oriented plane.