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Dominating deformation mechanisms in ultrafine-grained chromium across length scales and temperatures

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ABSTRACT

The microstructure influence on the thermally activated deformation behaviour of chromium is investigated for a more fundamental understanding of the deformation mechanisms contributing to plasticity in bcc metals. Therefore, scale-bridging experiments at variable temperatures and varying strain-rates are performed, encompassing macroscopic compression tests in direct correlation to local in-situ SEM micro-compression experiments on taper-free pillars and advanced nanoindentation testing. For the first time, it is demonstrated that, independent of stress state, sample volume and surface fraction, a distinct temperature-dependent transition of the dominating deformation mechanism occurs. While at low temperatures the lattice resistance dominates, exceeding a critical temperature the dislocation interaction with grain boundaries becomes the rate limiting step. Finally, based on the vastly different fractions of grain boundaries in the tested sample volumes, a comprehensive model on the deformation of bcc metals, in particular at small scales or for confined volumes is derived.

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1. Introduction

Over the last decades, investigations on the elemental deformation mechanisms in different metals, in particular body-centred cubic (bcc) ones, were extended from originally coarse-grained (cg) states [1,2] to single-crystalline (sxx) [3,4], ultrafine-grained (ufg) [5–7] and even nano-crystalline (nc) [8,9] microstructures. Since macroscopic tests average deformation characteristics over several length scales, testing of limited sample volumes [10] offers the premise to assess specific intrinsic material behaviour. In these small dimensions, individual plasticity mechanisms, such as dislocation motion, dislocation interactions, dislocation pile-ups [9], or diffusion-mediated processes such as grain boundary (GB) sliding [11,12] can potentially be isolated, thereby allowing identification and analysis of specific deformation modes.

A common approach to gain insight into the thermally activated deformation behaviour of bcc metals is to determine the strain-rate sensitivity (m) and the corresponding activation volume (v) [13]. Therefore, constant strain-rate tests or/and strain-rate jump (SRJ) tests are conducted and the rate-dependent stress responses are used to determine m. Since in this work the aim is to bridge macroscopic experiments to small volumes, it is important to note that recent work on nc Ni, ufg Al and ufg Nb [14–16] revealed direct comparability of constant strain-rate tests and SRJ tests performed by nanoindentation, small-scale tension and compression experiments with macroscopic data.

Conventional cg or sxx face-centred cubic (fcc) metals exhibit m-values in the order of 10^{-3} [5,17–20]. If internal length scales for dislocation interaction become smaller, e.g. by decreasing the grain size, m increases by about one order of magnitude [9,17]. The corresponding v (indicated in multiples of the cubed Burger's vector b) decreases from values well above 100 b^3 (cg microstructure) to a couple of 10 b^3 in ufg fcc metals [9,18]. This indicates a transition from forest dislocation interactions to dislocation-GB interactions. Further decreasing the grain size to the nc regime leads to low v of ~1 b^3. These values are classically attributed to diffusion-driven processes [9].

To investigate the GB contribution in confined sample volumes, Zhang et al. [19,20] performed micro-compression tests on sxx and ufg Cu pillars and reported that m is strongly dependent on the sample diameter to grain diameter (D/d) ratio. While sxx and macroscopic polycrystalline Cu samples show low m-values of ~0.002, they reported ~0.10–0.15 for D/d ranging between 3 and 10. Thus, the high number of interfaces in the deformed volume

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strongly influences the deformation behaviour [5,9,21] as well as the yield stress [22–25] of pillars in the fcc case.

While the situation in fcc metals is quite well understood, the situation is less clear in bcc structures [26,27]. This prevails especially for cases where microstructure, microstructural constraints, loading conditions and variable sample sizes are taken into account. Moreover, the flow stress in bcc metals consists of two parts, namely the temperature independent athermal component (σ_a), which arises from long-range stresses caused by obstacles to dislocation motion, such as impurities or GBs, and the temperature and strain-rate dependent thermal component of the flow stress (σ_{th}), which stems from the resistance of the lattice itself, called the Peierls potential [1,28]. In the latter case, the movement of screw dislocations via the kink-pair mechanism becomes the dominating thermally activated contribution during low temperature deformation [29].

The temperature dependency varies with respect to a critical material specific temperature (T_c), upon which thermal activation eases the movement of screw dislocations with increasing temperature. Eventually, the lattice friction diminishes once T_c is reached at ~$0.2 \cdot T_m$ [29], with T_m being the melting temperature of the respective metal. Above this temperature (for Cr ~160 °C [29]), the rate-dependent characteristics in bcc and fcc metals are comparable since the Peierls potential contribution vanishes. Therefore, screw dislocations exhibit a similar mobility as edge dislocations [28], which consequently leads to low m and corresponding high v typical for fcc metals, as only long range stresses prevail.

In literature, investigations on bcc metals were conducted by Wei et al. [30–34] performing macroscopic compression tests and Zhou et al. [35] and Wu et al. [8] performing nanoindentation tests, addressing the rate-dependent deformation behaviour on V, W, Mo, Ta and Cr. Increasing m with increasing grain size was reported, opposite to reports on fcc metals [5,9,21].

More recently, Maier et al. [6,7] investigated the deformation mechanisms in sxx and ufg bcc metals by means of advanced nanoindentation techniques at room temperature (RT) for Cr and W [7], and at elevated temperatures for Cr [6]. For Cr, m at RT of ~0.07 in sxx samples was attributed to a strong contribution of the Peierls potential and a low mobility of screw dislocations [28] which govern the deformation process at low homologous temperatures underneath T_c via the kink pair mechanism [1,28,29]. Comparably lower m at RT of ~0.02 in ufg samples was referred to a prevailing contribution of the Peierls potential in combination with an increased athermal contribution due to GB strengthening. Overcoming T_c in the ufg state, a further increase of m was measured and related to a diminishing contribution of σ_{th} accompanied by an emerging dominant thermally activated dislocation-GB interaction. σ_a remains mostly constant with increasing temperature due to a thermally stable microstructure [6].

In this work, focus is placed towards a more comprehensive scale-bridging understanding of the deformation characteristics of bcc Cr by examining contributing factors such as microstructure, sample size, temperature and stress state. The corresponding effects on the deformation behaviour over four orders of magnitude concerning the sample size, taking into account rate effects at ambient as well as non-ambient conditions, are analysed in this study. Therefore, uniaxial macroscopic compression tests, in-situ SEM micro-compression experiments, as well as multiaxial advanced nanoindentation experiments, using different tip geometries, were performed at variable strain-rates between RT and 400 °C to determine m-values and activation volumes. The stress-strain response and occurring deformation mechanisms were compared with sxx Cr to assess the impact of GB contributions. Moreover, the rate-dependent properties and microstructural evolution will be related to the loaded material volume with respect to testing temperature (T_{test}) and fraction of GBs present within the specimen to assess size-dependent mechanism transitions.

2. Material processing

The as-received polycrystalline ultra-high-purity Cr sheets (Cr-265, Plansee SE, Reutte, Austria) were cut by Electrical Discharge Machining (EDM, Brother HS-3100) to a cylinder with a diameter of ~30 mm and a height of ~7 mm. Subsequently, this cylinder was deformed via High Pressure Torsion (HPT) [36,37] using a rotational speed of 0.5 rpm and a pressure of 4.2 GPa at 200 °C to an equivalent strain of ~360 (50 rotations) to reach an ufg microstructure. Subsequently, the Vickers hardness (Buehler MicroMet 5104, load of 500 gf) of ufg Cr was measured over the whole disk radius (r) and disk thickness on polished samples and is shown in Fig. 1a on top of the HPT disk cross section.

2.1. Compression testing

The EDM-cut macroscopic compression samples indicated in Fig. 1a were subsequently ground and polished with SiC paper to achieve a smooth sample surface with final sample dimensions of $2 \cdot 2 \cdot 3$ mm^3. They were machined in axial disc direction, where elongated grains from the HPT process are oriented perpendicular to the compression axis. The samples were held between two WC-

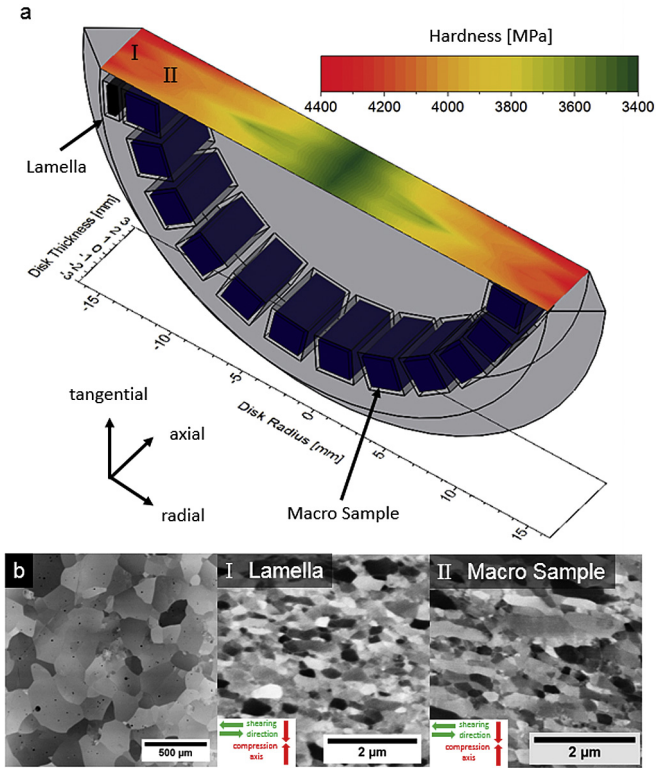

Fig. 1. Half of a HPT-deformed Cr disk with corresponding hardness map and BSE images of the microstructure. a) EDM was used to cut a lamella (black – left side) from the HPT disk for FIB pillar preparation from a disk radius of 14 mm and several macroscopic compression samples from a radius range between 12 mm and 14 mm. The compression axis is the axial direction of the HPT sample. b) Microstructures before and after HPT deformation. The left image shows the as-received Cr (d~200 µm). I and II represent the microstructure after HPT deformation at a disk radius of 14 mm (lamella for pillar preparation, d~160 nm) and at a disk radius of ~12 mm (for macroscopic samples, d~300 nm), respectively.

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