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Size effects and temperature dependence on strain-hardening mechanisms in some face centered cubic materials

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The mechanical behavior of face centered cubic metals is deeply affected when specimen dimensions decrease from a few millimeters to a few micrometers. At room temperature, a critical thickness (t) to grain size (d) ratio $(t/d)_c$ was previously highlighted, under which the softening of mechanical properties became very pronounced both in terms of Hall–Petch relation and work hardening mechanisms. In this work, new experimental results are provided concerning the influence of temperature on this size effect for copper, nickel and Ni–20 wt.%Cr, representative of a wide range of deformation mechanisms (*i.e.* dislocation slip character). It is shown that multicrystalline samples $(t/d < (t/d)_c)$ are not deeply affected by an increase in temperature, independently of the planar or wavy character of dislocation glide. For pronounced wavy slip character metals, surface effects in polycrystals $(t/d > (t/d)_c)$ are not significant enough to reduce the gap between polycrystal and multicrystal mechanical behavior when the temperature increases. However, a transition from wavy slip to planar glide mechanisms induces a modification of the polycrystalline behavior which tends toward multicrystalline one with a moderate increase in temperature. This work demonstrates that surface effects and grain size influence can be successfully disassociated for the three studied materials using an analysis supported by the Kocks–Mecking formalism. All these results are supported by microscopic investigations of dislocation substructures and compared to numerical simulations using a strain gradient plasticity model.

1. Introduction

Designing small technical systems often means dealing with confined plasticity at scales below 10^{-4} m, which requires additional knowledge of the material work hardening properties. At macroscopic scale, for instance, micro-components working with metallic parts of typical dimensions in the range [1–500] μm are often processed by traditional ways: forging, rolling or extrusion processes (Fu and Chan, 2014). For these forming processes, it is necessary to take into account the size effects (Janssen et al., 2008) to correctly predict the force, the final product geometry, and the possible appearance of fracture or necking (Fu et al., 2013; Lai et al., 2008). However, the strong variability of the mechanical responses to the external loadings can imply a lack of reliability of these micro-systems (Geiger et al., 2001). This may lead to an early or unexpected failure due to the development of damage consecutive to their forming process (Fu et al., 2013; Molotnikov et al., 2012; Fu and Chan, 2012).

Previous works have clearly shown a so-called "smaller is softer" trend for samples with critical dimensions in the

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range of 1 to 500 μm (Fu et al., 2001; Janssen et al., 2006; Keller et al., 2009; Weiss et al., 2002; Yang et al., 2012), triggered by the t/d parameter (*i.e.* the thickness (t) to grain size (d) ratio). When this parameter decreases, the flow stress, the hardening behavior and the fracture strain of face centered cubic (fcc) metals are strongly modified as reported by numerous researchers both in terms of experimental studies (Janssen et al., 2006; Keller et al., 2009; Weiss et al., 2002; Klein et al., 2001; Miyazaki et al., 1979; Simons et al., 2006) and numerical simulations (Lai et al., 2008; Geißdörfer et al., 2006). This modification is observed either by changing grain size for constant thickness (deviation from the Hall–Petch relation for the larger grain size levels Keller et al., 2011) or by modifying the thickness for constant grain size (Yang et al., 2012; Hug and Keller, 2010). Three kinds of mechanical behavior have been highlighted for fcc structure when the t/d ratio is reduced: polycrystalline, multicrystalline and quasi-single crystalline (Keller et al., 2011), separated by two critical t/d ratios (Janssen et al., 2006). The typical value $(t/d)_c$ separating the polycrystalline behavior from the multicrystalline one reported in the literature ranges between 3 and 15, mainly depending on the stacking-fault energy. The second critical t/d ratio separating the multicrystalline from the quasi-single crystalline behavior is close to the unity.

The physical origin of this softening can be found in the surface effects occurring in multicrystalline samples during the strengthening. These surface effects involve a stress gradient between core and surface grains of around 30%, directly linked to the different mean dislocation cell size observed in these two regions, especially during the second work hardening stage for samples with t/d lower than $(t/d)_c$ (Keller et al., 2010). Activating cross-slip mechanisms in the third work hardening stage leads to a progressive decrease of these stress gradient effects and therefore materials become less sensitive to size effects.

The transition from a polycrystalline to a quasi-single crystalline state modifies both mechanical behavior and forming ability (Gau et al., 2007, 2013; Geng et al., 2013; Raulea et al., 2001). In order to improve the formability of micro components in the dimension range [1–500] μm, a moderate modification in temperature could be an interesting option. However, very few experimental data are available in literature concerning size effects and temperature. Eichenhueller and co-workers (Eichenhueller et al., 2007) studied the microforming of brass and stainless steel microparts from room temperature up to 673 K. They clearly show a homogenizing effect of the temperature on the main parameters of microforming processes as upsetting or extrusion. In a recent study devoted to copper for two extreme t/d values (Hug et al., 2013), one higher and the other below the critical value $(t/d)_c$, we reported that polycrystalline behavior tends toward multicrystalline one with a moderate increase in temperature, approximately above a value of 373 K. These experimental results were correlated to the progressive predominance of the third work hardening stage and associated dislocation cross-slip. The origin of this phenomenon remains nevertheless unclear. Indeed, two mechanisms could be responsible for this observation: (i) a modification of the grain size dependence with a change in temperature, and (ii) a progressive generalization of surface effects in the entire volume of the samples even for polycrystalline samples. These two mechanisms may also act simultaneously.

The objective of this paper is, hence, to contribute to the understanding of the temperature dependence of the size effects in small dimension metallic samples. The contribution of the two mechanisms cited above will be analyzed for a large range of temperature and t/d values. Experimental results concerning size effects in a temperature range from 203 K to 773 K are presented for nickel, copper and Ni–20 wt.%Cr alloy characterized by different slip character. The Hall–Petch relation, the strain hardening and the free surface influence are experimentally investigated using a work hardening formalism based on Kocks–Mecking works which help to disassociate the grain size and surface effects. The discussion of these results is supported by two complementary tools. From a microstructural point of view, Transmission Electron Microscopy (TEM) observations are performed in order to quantify the dislocation substructures which develop during the plastic straining in temperature. From a predictive point of view, the physical mechanisms as dislocation glide or stress gradient localization across the thickness are studied by numerical simulations using a previously described single crystal strain gradient plasticity model (Keller et al., 2012). The evolution of the size effects in fcc materials with temperature will finally be correlated with the corresponding deformation mechanisms (wavy or planar slip character).

2. Experimental procedures and numerical tools

2.1. Material description, samples preparation and microstructural analysis

In this study, three distinct metals were employed: high purity polycrystalline nickel and copper (purity >99.98 wt%) and polycrystalline Ni–20 wt.%Cr. These metals are commonly used in small mechanical parts in biomedical or electronic devices for instance. The values of the stacking fault energy γ_{sfe} (obtained from literature) of each one are summarized in Table 1. The slip character of the material is linked to the stacking fault energy through the ratio $\gamma_{sfe}/\mu b^2$ (μ is the shear modulus and b the modulus of the Burgers vector of dislocations) which is inversely proportional to the dissociation distance between two dislocations. Cross-slip is then easily activated for materials with large values of $\gamma_{sfe}/\mu b^2$ whereas planar slip metals are characterized by corresponding low values. The values of this ratio for Ni, Cu and Ni–20 wt.%Cr are indicated in Table 1. Cross-slip is then easy for nickel, involving wavy slip character whereas planar slip is dominant for Ni–20 wt.%Cr. Copper presents an intermediate value ensuring deformation mechanisms with cross-slip.

Samples consisting of rolled sheets of thickness t ranging from 12.5 μm to 3.2 mm were annealed in a secondary vacuum ($P_{O2} < 10^{-5}$ mbar) in order to obtain various grain sizes without oxide layers. Details of the experimental procedures and associated microstructural characterization