



Effect of stress path on the miniaturization size effect for nickel polycrystals



C. Keller^{a,*}, E. Hug^b, A.M. Habraken^c, L. Duchêne^c

^a Groupe de Physique des Matériaux, UMR 6634, CNRS, Université de Normandie, INSA de Rouen, avenue de l'Université, 76800 Saint-Etienne du Rouvray, France

^b Laboratoire Crisamat, UMR 6508, CNRS, Université de Normandie, Ensicaen, 6 bvd du maréchal Juin, 14050 Caen, France

^c Département ArGEnCo, Division MS²F, Université de Liège, Chemin des Chevreuils 1, 4000 Liège, Belgium

ARTICLE INFO

Article history:

Received 26 March 2014

Received in final revised form 2 June 2014

Available online 18 July 2014

Keywords:

A. Dislocations

A. Grain boundaries

A. Strengthening mechanisms

B. Metallic material

Hall–Petch

ABSTRACT

The mechanical behavior of metallic materials deeply depends on the size of samples. For specimen dimensions decreasing from a few millimeters to a few micrometers, the general observed trend is a softening of the mechanical behavior in tension which affects the stress level and the strain hardening. The objective of this work is to provide new experimental results in order to analyze the miniaturization size effects for various stress paths without strain gradients across the thickness of the samples. To this aim, experimental tensile tests, large tensile tests and shear tests have been performed on Ni sheets with various grain sizes. Results show that the miniaturization softening is affected by triaxiality, the larger is this parameter, the lower is the mechanical softening. These features seem to be linked to surface effects which are larger for low triaxiality stress paths. From an industrial point of view, it is hence possible to improve the forming of microparts using suitable stress paths.

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1. Introduction

For approximately 20 years, the mechanic of materials community has paid a great effort to understand the mechanical behavior of metallic materials at small scales. This effort has been motivated by the huge demand of miniaturized parts from the automotive, medical or electronic industries for micro-device production. Due to the low dimensions of these micro-systems, metallic parts with typical dimensions ranging from 0.5 μm to 500 μm are used. As a consequence, the size of the microstructure is generally no longer negligible with respect to the specimen dimensions and size effects occur modifying the mechanical properties. If these size effects are not properly considered during the design and forming of micro-parts, then several issues can appear such as low reliability or high production costs (Geiger et al., 2001).

These size effects involve two different trends depending on the dimension range of the parts. The transition from micro-sized samples to nano-sized ones leads to a “smaller is stronger” effect widely characterized first for thin films (Nix, 1989; Venkatraman and Bravman, 1992; Espinosa et al., 2004) and more lately observed for micro-pillars (Uchic et al., 2004; Greer et al., 2005; Greer and Nix, 2005). This effect, characterized by a strong increase in yield stress, is linked to the role played by dislocation sources on surfaces, the truncature of dislocation segments (Parthasarathy et al., 2007) and the dislocation starvation phenomenon (Greer et al., 2005). A general description of the “smaller is stronger effect” can be found in various recent reviews (Kraft et al., 2010; Uchic et al., 2009; Greer and Hosson, 2011). From macro specimens to micro-sized

* Corresponding author.

E-mail address: clement.keller@insa-rouen.fr (C. Keller).

specimens, a “smaller is softer” trend, less investigated than the “smaller is stronger” effect, is generally observed for metallic materials. This effect is associated with a general softening of the mechanical behavior.

For this “smaller is softer effect”, if specimen dimensions are reduced in the micro-range, the surface over volume ratio becomes significant. In this case, the surface regions, which behave differently due to their proximity with the solid boundaries, dominate the global response of the specimen. From a microscopical point of view, this mechanism has been extensively studied for Cu and Al single crystals during the 1970s (Fourie, 1967, 1970; Mughrabi, 1970, 1971; Kitajima et al., 1967, 1968; Kramer, 1963, 1967; Basinski, 1977; Nabarro, 1977). These authors reported a general softening of the surface areas of Cu single crystals due to dislocation annihilation on free boundaries. In particular, free surfaces promote planar slip conditions even for large stacking fault energy materials (Fourie, 1967). For Ni polycrystals strained in tension, a softening of the surface areas similar to the one characterized for single crystals has been observed by transmission electron microscopy (Keller and Hug, 2008). Miniaturization size effects are hence enhanced by the same mechanisms for single and polycrystals.

From a macroscopical point of view, surface effects modify the mechanical behavior and samples cannot be considered as a representative volume element. As a consequence a strong scattering of the mechanical properties may appear due to a weakest link effect. For polycrystals, this miniaturization size effect is triggered by the number of grains across the thickness (characterized by the ratio of the sample thickness t to grain size d also called t/d ratio) (Janssen et al., 2006; Miyazaki et al., 1979; Keller et al., 2009, 2012). When this parameter is lower than a critical value depending on the material, on the strain (Keller et al., 2009; Miyazaki et al., 1979) and on temperature (Dubos, 2013; Hug et al., 2013) then the softening of the mechanical properties occurs. This critical value represents, hence, a transition between the so-called multicrystalline behavior (low t/d ratios) and the polycrystalline behavior (large t/d ratios) (Keller et al., 2011). Moreover, for multicrystalline samples, stress and strain distributions are no longer homogeneous. As reported in a previous paper (Keller et al., 2012), when the t/d ratio is reduced below the critical value, strong stress gradients due to surface effects form across the thickness involving the softening of the overall sample.

As a consequence, for microforming, the existence of the lower bound for the polycrystalline behavior must be taken into account together with the softening of the mechanical properties. However, up to now, most of the analyses of the miniaturization effect on the mechanical behavior have been performed for tensile conditions at room temperature involving stress paths far from those employed for microforming. New developments are hence necessary to improve the microforming processes. For the effect of temperature, a recent investigation reported a reduction in the softening of the mechanical behavior for copper samples strained at moderate temperatures (Hug et al., 2013). For the effect of the stress path, so far, a few articles have been published on the mechanical behavior of small samples submitted to different complex loadings (Dubos et al., 2013; Michel and Picart, 2003; Saotome et al., 2001). The different published results show that the miniaturization effect also occurs for complex stress paths. In particular, it seems that the critical strain level which promotes the size effect strongly depends on triaxiality with an increase in the critical strain for an increase in triaxiality. Moreover, the softening of the mechanical behavior seems to be reduced for low triaxiality stress paths (Dubos et al., 2013). However, these results have been obtained thanks to Nakazima tests involving different sample geometries and strain gradients across the sample thickness. When the sample thickness is reduced, the contribution of strain gradients on the mechanical behavior can be important and interact with the size effect.

This work is hence focused on the investigation of miniaturization size effect for different stress paths avoiding strain gradients. The objective is to characterize the role played by triaxiality on the softening of the mechanical properties for complex stress paths close to microforming ones. To this aim, a comparison is made between the mechanical behavior of Ni sheets with various thickness to grain size ratios submitted to three different mechanical tests with different triaxiality: tensile test, large tensile test and shear test. The results show that triaxiality plays an important role on the softening of the mechanical properties, the softening being larger for low triaxiality stress paths. These phenomena are then discussed in terms of surface effects and of dislocation mean free path which seem to depend on triaxiality.

2. Material study and experimental procedure

Three different mechanical tests with different stress paths were employed to study the effect of triaxiality on the miniaturization size effect: tensile test, simple shear test and large tensile test. Note that at the start of the simple shear test, the pure shear state is present. As strain level is low (for instance, less than 0.3 in Fig. 3) in most of the cases analyzed, the pure shear assumption will be applied within the discussion. The first mechanical test was performed using a classical strain controlled tensile machine coupled with an extensometer. The other two mechanical tests were achieved thanks to a bi-axial servo-hydraulic machine designed by the University of Liège. This machine, illustrated in Fig. 1(a), is characterized by two actuators, a vertical one able to perform axial displacements and a second horizontal one able to carry horizontal displacements. The two tests performed using the bi-axial machine are displacement controlled and strains are optically measured using a digital image correlation software. For the three tests, the strain rate was set up to 10^{-4} s^{-1} . A more detailed description of this mechanical testing device can be found elsewhere (Flores et al., 2010).

Samples with constant thickness (0.5 mm) and various grain sizes were employed to ensure various thickness to grain size ratios. Thanks to this moderate thickness, shear tests can be performed without buckling. Two different kinds of samples were machined from 0.5 mm nickel rolled sheets. For tensile conditions, dog bone shape samples were employed with a gauge section of $20 \times 10 \text{ mm}$ (length \times width) as represented Fig. 1(b). This first kind of sample was gripped in the hatched

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