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Materials Science and Engineering A 419 (2006) 238–248

MATERIALS SCIENCE & ENGINEERING A

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An experimental assessment of grain size effects in the uniaxial straining of thin Al sheet with a few grains across the thickness

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Received in revised form 21 December 2005; accepted 23 December 2005

Abstract

The mechanical behaviour under uniaxial tension of polycrystalline 99.999 at.% Al sheet with a thickness of a single or a few crystallites is investigated experimentally. The specimens are cold rolled to a thickness (t) of 100 up to 340 μm and the grain size (d) is varied by recrystallisation between 75 and 480 μm. All specimens have a similar texture and a regular grain structure. For $t/d < 1$ the flow stress varies only slightly with t/d, independently of t or d itself. For $1 < t/d < 3$, a strong increase in the flow stress is observed. The main explanation for this different behaviour in tension, stems from the grain boundaries parallel to the specimen surface, impacting the deformation and rotation of the grains. Furthermore, it is shown that a single Hall–Petch type relation does not apply to these kinds of specimen. Extrapolations towards specimens with columnar grains are made through the analysis.

Keywords: Size effects; Surface effects; Grain size; Texture; Uniaxial tension; Hall–Petch

1. Introduction

As a consequence of the ongoing miniaturisation in, for instance, the medical and the micro-electronics industry, ever thinner films and plates are being processed. As a result, only a few grains are often present in the thickness of the material. In mechanical loading, through e.g. forming, shaping, actuating and sensing (securing) of such materials, free surface and size effects are gaining importance and traditional continuum mechanics approaches may be losing their validity. Also, if only a few grains are present across the thickness of a component, the response to the applied forces may show great variation among components: hence, the reproducibility of the mechanical performance may become a problem. Overlooking the above effects in the industrial design of mechanics at small length scales may well lead to erroneous or too costly solutions. Many known phenomena in bulk metal mechanics, such as ductile failure, localised necking, texture development, etc., need to be reconsidered when the ratio between the component and the grain size becomes small. A number of effects are listed below that are important upon miniaturisation. A more detailed description of several of these effects can be found in [1].

1.1. Microstructural effects

The effect of grain size refinement on the strengthening of bulk metals has been thoroughly investigated in the literature. A linear dependency of the yield stress on the inverse of the square root of the grain size was reported [2,3], now well known as the Hall–Petch relation. A similar equation holds for the flow stress at constant strain [4]. As an explanation, a pile-up model was introduced [2,3] and a grain boundary dislocation source model was proposed [5]. However, there is clear experimental evidence that the Hall–Petch relation breaks down for very small grains, an opposite trend of softening was reported for copper below a grain size of 50 nm [6].

Second phase particles within the material impede dislocation motion (Orowan and Friedel effect). The interaction of the dislocation loop with obstacles not only depends on the obstacle size and spacing but also on their strength. The dislocations either bow out between obstacles or cut through. Changing the particle spacing or size will therefore change the mechanical response.

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doi:10.1016/j.msea.2005.12.029

Second phase particle effects are avoided in the present work by analysing a very pure metal.

1.2. Grain statistics effects

Crystals are anisotropic with respect to deformation. The response of a specimen to the applied forces will vary depending on the number of grains involved and on the distributions of grain size and grain orientation. The stronger and the sharper the crystallographic texture is, the smaller the variation will be. Although grain statistics is not the subject of this paper, it has to be considered carefully in the design of the specimens to be investigated in order to rule out its effect on the comparison between the different specimen series.

1.3. Strain-gradient effects

Size-dependence was also discussed at a scale of a micron to tens of microns [7]. Considering the results reported by Fleck et al. [8] and Stölken and Evans [9], it is proven that at this scale a non-homogeneous deformation implies "smaller is stronger". This so-called strain-gradient effect is essentially due to the appearance of lattice curvature, which has to be accommodated by geometrically necessary dislocations. The stresses created through that directly impact the yielding behaviour. In this work it is chosen to apply a macroscopically homogeneous deformation to the specimens by means of uniaxial tension. In doing so, (macroscopic) strain-gradient effects on the mechanical behaviour are ruled out.

1.4. Boundary and surface effects

In small structures or thin films, all material is relatively close to a physical boundary. This boundary was categorized as a dimensional constraint which plays a similar role as the microstructural constraints that govern the bulk behaviour (external boundaries versus internal boundaries/interfaces) [1]. For metals a hard oxide layer may exist, which might be (partially) impenetrable to dislocations. Reducing the specimen size the larger the contribution of this hard boundary layer becomes and the stronger the strengthening will be. In the present work a free surface will be considered, for which the effect of an oxide layer (even though present) is negligible.

In the range of specimen thicknesses of hundreds of microns, the flow stress of polycrystalline Al, Cu, Cu–13 at.% Al and Fe has been investigated in a certain range of grain sizes and specimen thicknesses [10]. It is shown that the flow stress decreases with decreasing specimen thickness when the ratio of specimen thickness (t) and average grain size (d) is smaller than a critical value. The critical value of t/d increases with decreasing grain size and stacking fault energy. Critical values of t/d were found between 3.5 and 15. Similar results were obtained on polycrystalline CuNi18Zn20 and CuZn15 alloys [11,12], on CuZn36 [13] and on Al 99.0–99.5% [14]. Although the lines of reasoning in these contributions for the explanation of the observations are different, the following can be stated: grains in the surface region have a different deformation behaviour than grains within the volume of the material. Because 'surface' grains are less constrained to deformation by surrounding grains, surface grains are therefore weaker. At decreasing sheet thickness the fraction of grains with a free surface increases and a decreasing flow stress results.

Experimental work on specimens with t/d less than or equal to one is scarce. Tensile experiments were performed on Al–2.5 wt.% Mg (A5052) specimens with constant thickness (250 μm) and varying grain size to find a Hall–Petch like relation for the flow stress dependency on the grain size [15]. Among the specimens, there were two with $t/d < 1$, namely t/d equal to 0.083 and to 0.8. Tensile tests were performed on three pure Al (99.999%) specimens with varying thickness and grain size, such that t/d equals 0.36, 0.6 and 1.1, to investigate the effect of specimen size on the fracture strain [16].

The tensile data of four of these five specimens seem to imply that for $t/d \leq 1$ a variation of t/d does not considerably change the flow stress. Yet, no conclusions in this direction were drawn in these papers. Because of lack of data Miyazaki et al. [10] extrapolated, for the purpose of their model, their results for $t/d > 1$ to $t/d = 0$, assuming a monotonic decrease of the flow stress. This might be in contradiction with the above indication of almost no flow stress change when there is a change of t/d (<1). Evidently, there is a serious need for systematic research on the mechanical behaviour of specimens with $t/d < 1$.

1.5. Geometrical effects

The effects of varying the specimen shape were investigated by Miyazaki et al. [17]. Again, it is shown, for rod and plate specimens, that the flow stress decreases at decreasing diameter (D) or thickness. However, the decrease in flow stress starts earlier for the rod specimens than for the plate specimens when D/d or t/d are decreased. This is a logical consequence of the larger volume fraction of surface crystals in rod specimens than in plate specimens when the diameter and the thickness are equal. However, when the cross section areas are equal the reverse holds.

In the above-mentioned experimental work on surface effects, it was found that stresses decrease with decreasing t/d, i.e. with an increasing volume fraction of grains with a free surface. Van der Giessen and Richelsen [18] have pointed to the possibility that, despite the free surface effect, the reverse might also occur in specimens where the crystallographic texture varies across the thickness (which is not uncommon in e.g. rolled materials), since the surface crystals may exhibit a texture that delivers a higher mechanical strength than the texture of the bulk crystals.

In the experimental study of the free surface effect the ratio t/d is varied in order to be able to separate the free surface effect from other types of (size) effects. By preparing specimens with different t and/or d there is a serious risk that differences can occur in, for example, dislocation density, oxide layer and crystallographic texture. These differences cause variations in mechanical properties that disturb the quantitative analysis of the free surface effect. To date, this is not sufficiently taken into