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Size-dependent creep of duralumin micro-pillars at room temperature

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ABSTRACT

The strength of aluminum alloy 2025 (duralumin) micro-pillars is known to be significantly higher than that of pure Al micro-pillars of comparable sizes since the precipitates present act as obstacles to trap dislocations within the small sample volume. In this work, the creep behavior of precipitate-hardened duralumin micro-pillars of sizes ~1 μm to ~6.5 μm is investigated by compression experiments at room temperature. The effects of an internal grain boundary were also investigated by comparing the creep behavior between single crystalline and bi-crystalline micro-pillars. The results reveal that peak-aged duralumin pillars, in which the produced precipitates can efficiently block mobile dislocations, show increasingly significant creep with increasing pillar size, with a typical creep rate of $\sim 10^{-4}\ s^{-1}$ which is drastically larger than that of bulk at room temperature. The bi-crystalline pillars creep even faster than the single crystalline counterparts. TEM examination of the deformed microstructures reveals that the creep rate depends on the residual dislocation density, indicating that dislocations are the agents for creep. Theoretical modeling suggests that the steady-state creep rate is proportional to the lifetime of mobile dislocations, which rises with specimen size in the microns range due to the fact that the dislocations are not easily pinned in this range. As the pillar size increases in this range, the dislocations spend longer time in viscous motion across the specimen, hence the retained dislocation density is higher which leads to a higher strain rate according to the Orowan equation. It is expected that this trend of creep rate with specimen size will be reversed for larger specimens, probably in the tens of microns range, when dislocations experience a higher chance of being pinned and immobilized by the precipitates.

1. Introduction

It is well known that micro- and nano-sized metallic single crystals exhibit size-dependent deformation behavior. The most investigated phenomena are the inverse relationship between strength and specimen size (Dimiduk et al., 2005, 2007; Dou and Derby, 2009; Frick et al., 2008; Greer and De Hosson, 2011; Han et al., 2010; Kim et al., 2010; Kim et al., 2012; Lee et al., 2009; Ng and Ngan, 2008b; Sun et al., 2011; Volkert and Lilleodden, 2006; Ye et al., 2011), and jerky flow behavior (Dimiduk et al., 2005, 2007, 2006; Greer and Nix, 2006; Greer et al., 2005; Ng and Ngan, 2008a, b; Shan et al., 2008; Uchic et al., 2004). With the assistance of advanced techniques such as *in situ* transmission electron microscopy (TEM) (Hemker and Nix, 2008; Oh et al., 2009; Shan et al., 2008) and Laue micro-diffraction (Maass and Uchic, 2012; Maass et al., 2009; Zimmermann et al., 2012), the dislocation structure evolution in nano- and micro-specimens can be visualized, and the physical mechanisms of the unusual deformation behavior are explainable by a number of models. One widely

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accepted model considers a "dislocation starvation" condition pertinent to submicron crystal volumes (Greer and Nix, 2006; Greer et al., 2005), in which mobile dislocations would easily glide through the crystal leaving the latter in a continuous dislocation-depleted state. The "source truncation" (Parthasarathy et al., 2007; Rao et al., 2007) and "exhaustion hardening" (Akarapu et al., 2010; Norfleet et al., 2008; Rao et al., 2008) models consider that in larger crystals that are microns in size, due to the distribution and operation of dislocation sources in the confined volume, a mean-field condition for forest hardening could not be met.

In order to smooth out the jerky flow, a number of strategies have been proposed to restore the mean-field condition for dislocation interactions by introducing means to trap dislocations within the specimen, including coating (El-Awady et al., 2011; Gu and Ngan, 2012; Jennings et al., 2012; Lee et al., 2013; Ng and Ngan, 2009b; Xu et al., 2013), grain boundary (Burek et al., 2011; Gu et al., 2012; Jang et al., 2011; Kunz et al., 2011; Ng and Ngan, 2009a; Rinaldi et al., 2008; Zhang et al., 2013), nano-twinning (Afanasyev and Sansoz, 2007; Deng and Sansoz, 2009; Jang et al., 2011, 2012), and precipitates(Gu and Ngan, 2013). Somewhat surprisingly, in addition to achieving a smoother flow behavior, the rapid accumulation of dislocations within a small volume can often lead to tremendous strain hardening and therefore elevation of the flow stress (Afanasyev and Sansoz, 2007; El-Awady et al., 2011; Gu and Ngan, 2012, 2013; Jang et al., 2011; Jennings et al., 2012; Ng and Ngan, 2009a,b; Zhang et al., 2013), although in the case of introducing a grain boundary to form a bi-crystal, a weakening effect was observed in nano-sized pillars (Kunz et al., 2011) whereas strengthening was observed in micron-sized counterparts (Ng and Ngan, 2009a). Our recent study on precipitated duralumin micro-pillars (Gu and Ngan, 2013) shows that strength is improved while size dependence is reduced by the precipitates, when compared with pure Al pillars. However, significant creep was also found in these precipitated duralumin micro-pillars, particularly in relatively larger pillars.

Understanding time-dependent plasticity is very important in engineering applications, and so far, the room temperature (RT) creep behavior of micro-pillars has been studied in a number of materials. Compression experiments on Ni_3Al micro-pillars at RT have revealed an extremely high creep rate relative to that of bulk, which is thought to be due to the surface diffusion at the pillar heads (Afrin and Ngan, 2006). However, single crystalline Al micro-pillars were found to exhibit creep rates two orders of magnitude lower than polycrystalline Al bulk, and this is attributed to the much lower contents of dislocations which are the agents for dislocation creep (Ng and Ngan, 2007). The RT creep of nanocrystalline nickel pillars has also been found to exhibit a reverse relation with the specimen size, with a much larger creep rate than that of bulk due to an increased contribution of free surface (Choi et al., 2013). These observations as a whole suggest that both the external specimen size and internal microstructures can significantly affect the creep behavior of small crystals. However, the effects of these two factors on creep have not been systematically studied yet. In the present study, using duralumin micro-pillars as a model system, the coupled influence on the RT creep of a controlled precipitated microstructure, an introduced grain boundary, as well as external specimen size, was investigated.

2. Experimental details

Two bulk slices of aluminum alloy 2025 (Al-4 wt% Cu-1.3 wt% Mg-1.3 wt% Ag-0.6 wt% Mn) were annealed at 500 °C for 24 h for homogenization initially. The specimens were then solution heat-treated at 520 °C for 3 h in an air furnace, followed by immediate water quench in icy water. The bulk specimens were stretched by ~2% in a tensile machine before aging treatment. Then, one of the specimens, labeled as the 'RT-aged' hereafter, was exposed at RT for several days without any artificial aging; the other, labeled as 'peak-aged', was aged at peak condition at 170 °C for 15 h in a furnace (Gu and Ngan, 2013). The two pieces were mechanically polished, and then electro-polished in a 1:4 mixture of nitric acid and methanol at −30 °C and 15 V for 30 s. Afterwards, Electron Back Scattered Diffraction (EBSD) was carried out on both pieces to identify large grains with diameter > 80 μm and orientation ~[101], in which micro-pillars were fabricated by a Quanta 200 3D Dual Beam FIB/SEM (FIB) system. Micro-pillars of diameters ~6.5 μm, ~3.5 μm, and ~1.0 μm, all with a diameter-to-height ratio of ~1:3, were made by a series of concentric annular-patterned FIB milling with decreasing currents at an ion beam voltage of 30 kV. The fabricated micro-pillars were slightly tapered ~3° due to the defocused ion beam milling, and the diameters of pillars were taken at mid-height. While most pillars machined this way were single crystalline, some were bi-crystalline with an initially subsurface grain boundary passing through the body of pillar. All pillars were subjected to creep testing at RT in an Agilent G200 Nanoindenter with a flat-ended diamond punch of diameter 8.5 μm, which was fabricated from a diamond Berkovich tip also by FIB milling. The creep tests were performed in a load controlled manner, in which the applied load was raised to a peak value and held for 200 s before unloading. The loading and unloading rate was the same at ~12 $MPa s^{-1}$, so that the strain rate during this stage could remain at ~10^{-3} s^{-1}. Thermal drift was controlled at within 0.5 nm/s. The deformed appearances of micro-pillars after creep tests were imaged by scanning electron microscopy (SEM) in a LEO1530 microscope. Finally the deformed duralumin pillars were characterized by transmission election microscope (TEM) in order to examine their crystal structures as well as dislocation distributions. Longitudinal TEM samples running along the long axis of the pillars were prepared by FIB milling on both sides of the pillars to reduce the thickness into ~1 μm, then welded onto an *in situ* Omniprobe by tungsten deposition, followed by cutting off from the bulk. The samples were welded into TEM copper grids by tungsten deposition and cut from the Omniprobe, and finally thinned down to ~150 nm thickness by FIB milling for electron transparency. TEM characterization was carried out in a Philips Tecnai microscope operating at 200 kV.