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Creep of micron-sized Ni_3Al columns

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Abstract

Compression experiments were carried out using a nanoindenter at room temperature on micron-scale Ni_3Al pillars produced from focused ion-beam milling. Extremely high creep rates of the order of $10^{-5}\ s^{-1}$ were observed. The stress exponent measured is close to unity, which suggests that the creep mechanism is likely to be diffusion.
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1. Introduction

The influence of physical dimensions on mechanical properties has been of long-term interest to the materials science community. In metals, the increment of strength with grain refinement according to the Hall–Petch relationship [1,2] has been well documented. The effects of sample size on initial yield strength were first exploited in the 1950s using metallic whiskers [3–6]. At that time, the exploration was limited within the millimeter size range presumably due to instrumental limitations. With the advent of nanomechanical testing instruments, this subject received renewed interest in the submicron regime. Using nanoindenters the first sets of such data were reported from thin films showing the hardness varies inversely with the film thickness [7–9]. Nanoindentation also showed that the hardness of crystalline materials increases with decreasing indentation size, a phenomenon known as the indentation size effect (ISE) [10–13]. In fact the ISE was observed much earlier in micro-hardness tests, but was often confused due to measurement errors and was treated as an extrinsic property of material. After being reconfirmed using nanoindentation, the ISE was explained in terms of the strong strain gradients when deformation is limited to a small region [14–16].

Recently, the inverse relationship of yield strength with sample dimension was recognized from a series of compression tests on micron-sized columns fabricated using focused ion-beam (FIB) milling [17]. The experiments were performed on columns with varying diameters in 0.5–40 μm range, made of pure Ni, Ni_3Al–Ta and Ni-based super alloy, using a nanoindenter for the compression. The yield stress of these columns was found to increase drastically when the column size was below 20 μm. This report has created enormous scope for exploration of various mechanical properties as a function of the sample size. The purpose of the current investigation was to study the creep behavior of micron-sized pillars under different compressive loads.

2. Experimental

Pure Ni and Al at 74:26 atomic ratio was melted in an induction furnace and then cast into a small ingot. The ingot was homogenized at 1200 °C for 5 days in a vacuum furnace at 10^{-5} mbar and this resulted in enlarged grains of 1 mm average size. A small piece of sample from the core of the ingot was mechanically polished and then electropolished in a solution containing 10% perchloric acid in ethanol. The final composition of the sample was confirmed

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by energy dispersive X-ray (EDX) analysis to be in the Ni_3Al phase field in the Ni–Al binary phase diagram.

All the indentation experiments were conducted within a large single grain of 1.5 mm diameter to eliminate orientation effects. Inside that grain 2 μm diameter pillars of 3:1 aspect ratio were fabricated by milling out a circular crater of ∼20 μm diameter, leaving the pillars at the center of the crater, using FIB milling with Ga ions. The craters were large enough to ensure that the indentation probe would not make contact with the rims of the craters during compression of the pillars. Three such circular craters each containing one pillar at their centers is shown in Fig. 1. The compositions of the bulk material and the fabricated pillars as measured by EDX are shown in Table 1. The pillar composition shows a small amount of Ga implantation due to the FIB fabrication process.

Indentation experiments were carried out using a Hysitron nanoindentation transducer mounted on a ThermoMicroscopes CP scanning probe microscope. This system allows both nanoindentation and atomic force microscopy (AFM) imaging to be performed on the same platform. Since the *X*–*Y* position of the sample during nanoindentation can be controlled very precisely by the piezoelectric AFM scanner, this system is very suitable for the present experiments because of its high precision in locating the

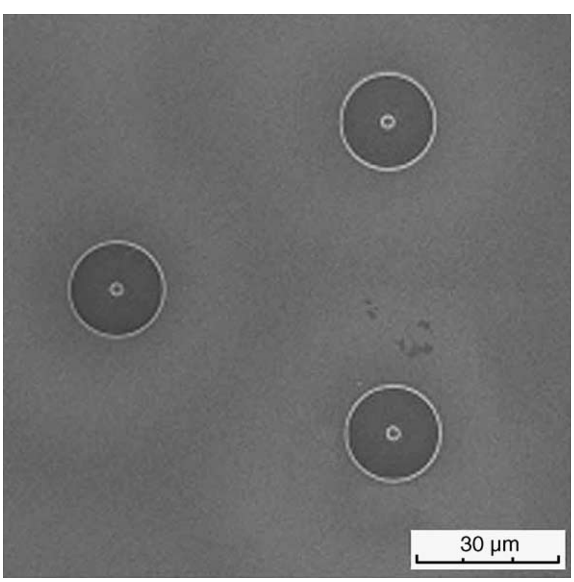

Fig. 1. Top view of circular craters, each containing one pillar at the center.

Table 1
Composition of bulk material and fabricated pillars

Element	At.% of bulk material	At.% of micro-pillars
Al K	26.37	26.13
Ni K	73.63	73.23
Ga K	–	0.64
Total	100.00	100.00

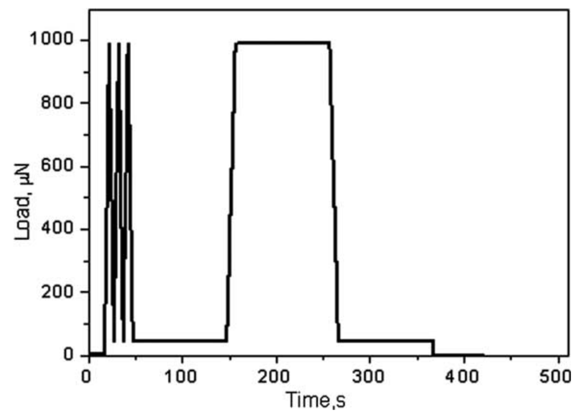

Fig. 2. Loading Cycle.

point for nanoindentation. A Berkovich tip was used as the indenter, and the tip radius was 200–300 nm, as determined from direct scanning electron microscopy (SEM) imaging of the tip. Before testing, the machine compliance was calibrated using a standard sample of fused quartz, and efforts were made to achieve a condition with very small thermal drifts. The loading schedule shown in Fig. 2 was applied onto the pillars. It contained a few cycles of loading and unloading at 100 μN/s to a peak load of either 1000 μN or 2000 μN to produce initial plastic deformation. The fact that the rather sharp indenter tip might punch into the heads of the pillars does not matter here because our aim is to investigate the subsequent creep behavior of these pillars after the injection of crystal plasticity into the pillars. The initial cycles were followed by a holding period at a low-load of 50 μN for 100 s to monitor the drift rate. The load was ramped up again and held at the peak load for 100 s, during which the creep response of the sample was monitored. This was followed by another low-load holding at below 70–100 μN for drift measurement before complete unloading. The creep displacement data presented below have all been corrected for thermal drift effects. In a control experiment, the same nanoindentation procedure was applied onto a flat area of the same Ni_3Al grain without FIB milling. AFM imaging was used to identify the area and to ensure the surface condition for indentation.

3. Results

Fig. 3(a) and (b) show the load and displacement curves produced from the loading cycle shown in Fig. 2 on a bulk surface and on micro-pillars at a peak load of 2000 μN. The curve from the bulk surface (Fig. 3(a)) shows a typical parabolic elasto-plastic behavior during the first load ramp, followed by significantly elastic recovery during the subsequent unloading and reloading steps, with little hysteresis between unloading and reloading. On the other hand, the micro-pillar (Fig. 3(b)) showed an initially rather linear load–displacement behavior followed by a convex turn at higher loads. Such a form of the load–displacement

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