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In situ transmission electron microscopy study of deformation of an aluminum alloy tribolayer

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Nanoscale deformation in the tribolayer of an Al–Mg alloy is studied using an in situ mechanical probe in a transmission electron microscope. The sample is strained locally at room temperature and the deformation is observed in real time. It is observed that when the tungsten probe comes into contact with the tribolayer, the material exhibits further hardening followed by material removal.
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In applications involving moving surfaces in contact with each other, the structure and the properties of the material close to the contacting surface will determine its friction and wear characteristics [1]. The structure is modified by the tribological process and evolves to a steady-state tribolayer. This study reports the microstructural evolution in the tribolayer leading to material removal using a specially designed in situ deformation holder inside a transmission electron microscope (TEM). For this study we have chosen the automotive-grade strain-hardenable Al–Mg (AA5754) alloy that comes into contact with the die surface during stamping operations.

In the past few years a number of in situ TEM nanoindentation holders capable of localized loading have been built [2–4] and the material behaviour studied. For example, Bobji et al. have studied indentation mechanics of Cu–Be alloy [5], Minor et al. have observed grain boundary rotation in Al–Mg thin films during in situ nanoindentation [6], De Hosson and Soer have studied interaction between dislocation and grain boundary [7], and Soer et al. have studied the effect of Mg on grain boundary motion [8] in Al–Mg thin films using in situ holders.

This paper reports the in situ TEM observation of deformation processes and mechanisms accompanying material removal when a hard asperity comes into contact with a softer tribolayer. A newly designed mechanical probe mounted in a TEM holder is used to observe the microstructural evolution in the tribolayer. A wedge-shaped electron-transparent sample prepared by mechanical polishing to introduce large deformation mimicking the tribolayer is indented using a tungsten probe inside the TEM. The sequence of deformation events in the tribolayer of a softer substrate coming into contact with a hard asperity which culminates in decohesion and material removal are imaged along with displacement–time data during deformation.

The in situ experiments were carried out in a JEOL 2000FX microscope using a holder modified to mechanically probe the sample. Similar to the one used by Bobji et al. [4], this holder has a two-stage positioning mechanism. Coarse positioning is achieved with a three-axis compact inertial slider drive and the fine positioning is achieved with a resolution of 0.1 nm through a four-quadrant piezoelectric tube operating in open-loop mode. The fine positioner acts as a pure displacement generator with a spring in series whose stiffness includes the deformability of the piezoelectric element and the support stiffness. The support stiffness of our system is greater than 1700 N m^{-1} and it has been seen that the stiffness of the tungsten probe is the deciding factor in

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the overall stiffness of the probe. The effect of displacement-controlled nanoindentation experiments and the influence of the feedback system on material response has been discussed in detail by other workers [9–11].

A schematic of the probe and sample in relation to the electron beam direction is shown in the inset of Figure 1. The tungsten probe is moved along the x-axis, perpendicular to the electron beam and the specimen edge, by applying uniformly decreasing voltages to the four outer quadrants of the piezoelectric tube. Small misalignment in the axis of movement of the piezoelectric tube is adjusted by superimposing corrective voltages to two adjacent quadrants. The movement of the tip is confined to the focal plane which is verified with the help of Fresnel fringes at higher magnifications.

An electron-transparent sample of dimension 4 mm × 3 mm was prepared from the recrystallized 1 mm thick sheet of commercial grade AA5754 Al–Mg alloy. The highly deformed electron-transparent edges were obtained by holding the sample at a small angle (~2°) while polishing in a precision polishing machine (Buehler Minimet 1000). Mechanical polishing with 5 μm grit diamond abrasive simulates the mechanical wear process resulting in a tribolayer.

A tungsten probe was fabricated by electrochemical etching using the "drop off" method [12]. Etching results in a sharp tip of radius less than 100 nm and it has been found that indenting with this tip results in a highly localized deformation [5]. To simulate a more realistic asperity we bend the probe into a hook (as shown in Fig. 1, inset) inside the TEM by moving the sharp tip against a ledge in the sample in a controlled manner. This resulted in a probe that has two principal radii of curvature of 500 nm about the z-axis and 2 μm about the y-axis at the point of contact with the sample.

The probe is moved towards the sample at a constant rate of 33 nm s^{-1} by applying a constant rate of voltage to the piezoelectric tube for 4 s. The voltage is held constant for 20 s and the probe is moved back by reversing the voltage rate. The average voltage applied as a function of time to the inner electrode of the piezoelectric tube relative to the outer electrodes is shown as the solid line in Figure 1. The whole process was captured digitally using a side-entry CCD camera (Gatan Erlangshen ES500W Model 782).

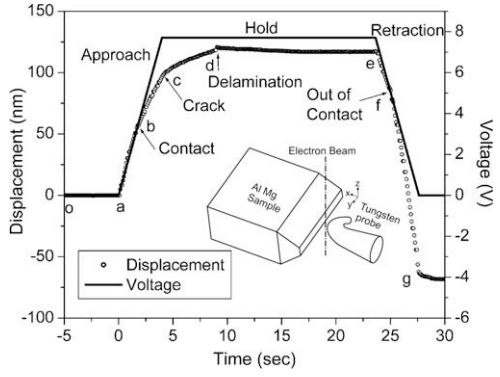

Figure 1. Variation of the displacement of the probe with time (open circles). Distinct changes in the slope of the displacement curve are observed, corresponding to contact of the probe with sample (b), crack nucleation (c), detachment of a layer of the tribolayer (d) and out of contact (f). The voltage applied to the piezoelectric tube is shown as a continuous line. A schematic of the wedge-shaped sample and tungsten probe is shown in the inset.

The actual movement of the probe is measured from the individual frames of the video. The position of a part of the probe in the consecutive frames is tracked with a cross-correlation based image-registration technique [5]. Sub-pixel accuracy is obtained by bicubic interpolation of the location of the correlation peak. The measured displacement of the tip relative to the sample is plotted in Figure 1. The size of the pixel was determined through calibration using standardized grids.

It can be seen from Figure 1 that when the probe is not in contact, if the voltage on the piezoelectric tube is held constant, the position of the holder (oa) remains constant with no appreciable drift. The measured drift rate was less than 0.2 nm s^{-1} over a period of 5 s. The probe starts moving as soon as the applied voltage starts changing. Until the probe comes in contact with the sample, the displacement is proportional to the voltage applied (ab). The probe continues to move even after the voltage is held at the constant value of 7.7 V. This could be due to relaxation of the sample as well as the piezoelectric material. It should be noted that the relaxation of the piezoelectric material is small as can be seen from the relaxation observed after the unloading at 'g' in Figure 1.

During the hold portion (ce), the probe continued to show displacement even though the piezoceramic was not moved. The displacement curve in Figure 1 shows a distinct change in slope at $t = 8.92$ s. At that instance, the corresponding image data shows that a small section of the material gets detached suddenly. This detached material then gets attached to the probe (Fig. 2b) and is found to slowly rotate out of transparency (Fig. 2c) in about 0.12 s. After this delamination-like material failure, the movement of the probe drops and the probe moves by less than 4 nm in 15 s (compared to over 25 nm in 5 s prior to the delamination event). As the probe is moved out, it loses contact with the sample at the point 'f ' in Figure 1. There is a distinct change in the displacement curve at that point.

Figure 3a–d shows close-ups of a portion (enclosed by a rectangle in Fig. 2a) in order to bring out the contrast changes in the sample close to the contact point corresponding to times a–d in Figure 1. Figure 3a shows the tungsten probe in close proximity to the electron-transparent region of the sample just before contact. The tungsten probe is opaque to the electrons at 200 keV and is seen with a dark contrast. The edge of the sample is electron transparent, with the transparency decreasing into the bulk. The mottled contrast of the sample is verified by tilting experiments to be from 10 to 30 nm size subgrains produced by severe mechanical deformation during mechanical thinning.

As the probe comes in contact with the sample (Fig. 3b), the slope of the displacement curve in Figure 1 changes and the subgrain contrast in the sample also changes. The contrast changes are either due to new subgrain formation (e.g. subgrain 1 in Fig. 3b) resulting from dislocation activity or due to localized rotation of the existing subgrains subjected to high local strain.

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