DEFORMATION AND FRACTURE OF NANOCRYSTALLINE PLATINUM MICROPILLARS

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Summary The deformation and fracture behaviour of electroplated platinum micron-sized pillars with an average grain size of several nm is examined through uniaxial tension tests. Tests are performed on pillars with and without pre-cracks of different sizes and crack tip radii, and the resulting deformation and crack propagation behaviour is related to microstructure-driven deformation processes and to existing linearly elastic and elastic-plastic models for fracture.

INTRODUCTION

The effect of sample size on the mechanical properties of small-scale metals has now been firmly established[1]. An intriguing research question that remains is whether this size effect also manifests itself in the fracture behaviour of metals with extrinsic (sample size) and intrinsic (microstructural) dimensions reduced to the micro and nanoscale. Exceptional fracture toughness in biological nanocomposites has been attributed to flaw tolerance at the nanoscale[2]. Exploiting such nanoscale fracture phenomenon could aid in the design of strong and tough engineering materials. Conducting systematic fracture tests on nanocrystalline platinum micropillars containing pre-cracks allows the effect of both intrinsic and extrinsic length scales to be examined. Fracture testing at cryogenic temperatures is performed in order to understand thermally activated aspects of fracture at the small-scale. Platinum is an ideal model fcc metal for this study because it does not form a surface oxide, and is additionally a technologically relevant material because it is commonly used in MEMS devices. Mechanical studies on nanocrystalline platinum will contribute to understanding deformation mechanisms and fracture behaviour in nanocrystalline materials[3, 4]. A parallel computational effort will also be conducted using large-scale molecular dynamics simulations that will help reveal deformation and failure mechanisms, and increase understanding of size effect and flaw insensitivity.

EXPERIMENTAL METHODS

Platinum micropillars are formed by electrodeposition from an aqueous chloroplatinic acid bath into an electron-beam lithography patterned poly(methyl methacrylate) template created according to a previously described method[5]. A potential-controlled pulsed electroplating scheme is developed to avoid the formation of porous pillars caused by hydrogen evolution during electroplating. Pillars are purposely overplated such that the platinum extends above the top of the poly(methyl methacrylate) template in order to fit into a tension grip (Figure 1). Pillar microstructure before and after testing is characterized using dark field and bright field transmission electron microscopy (TEM) and scanning electron microscopy (SEM). Symmetrical notches are milled along the free surfaces of the pillar with a focused ion beam such that the pillar undergoes mode I crack propagation in tension testing. This sample geometry is analogous to that of an ASME standard[6].

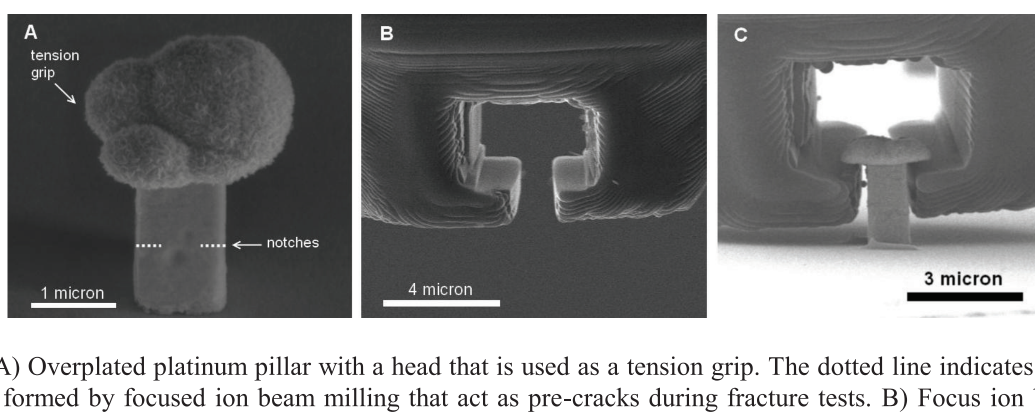

Figure 1. A) Overplated platinum pillar with a head that is used as a tension grip. The dotted line indicates the location of notches formed by focused ion beam milling that act as pre-cracks during fracture tests. B) Focus ion beam milled diamond tension grips that fits around the overplated head of the platinum pillar. The tension grips are connected to a nanoindentation module in the SEMentor which enables tension or compression testing. C) In-situ SEM image of in progress tension test.

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Compression testing of pillars without overplated heads is conducted using a commercial nanoindenter to determine the yield strength and understand the mechanical deformation of the nanocrystalline platinum micropillars. Tension testing is performed in SEMentor, a custom-built nanomechanical instrument comprised of a scanning electron microscope and nanomechanical module similar to nanoindenter. SEMentor is also equipped with a mechanical testing capability at cryogenic temperatures.

MD simulations are performed on Pt nanopillars with length L = 206 nm and diameter d = 43 nm. Two different microstructures are studied: 1. Single crystalline (SC) nanopillars having their long axis oriented in the [111] direction, and 2. nanocrystalline (NC) pillars constructed using a Voronoi procedure and having an average grain size of ~ 15 nm. In order to examine the notch sensitivity, U-shaped notches are introduced in the SC and NC samples. The ratio of notch depth to pillar width is kept equal to that of the experimental samples. The MD simulations were performed using the Large-scale Atomic/Molecular Massively Parallel Simulator (LAMMPS)[7], where interactions between Pt atoms were described using the Embedded Atom Method (EAM)[8] potential parametrized by Sheng *et al.*[9]. Periodic boundary conditions were imposed along the axial direction. To minimize spurious vibrations that arise from the rapid thermal expansion during heating, the nanopillars were carefully equilibrated at 300 K: we first increased the temperature from 0 K to 300 K over 100 ps using Langevin dynamics, then annealed at 300 K for 50 ps using molecular dynamics under the NPT ensemble, where constant temperature and zero normal stress in the y direction were maintained using a Nose-Hoover temperature thermostat and pressure barostat[10]. After the nanopillars were equilibrated, we applied uniaxial tensile loading by stretching the nanopillars in the axial direction at a constant true strain rate of 0.1 per ns. Deformation behavior obtained with MD simulations is compared to experimentally observed behavior to gain better understanding of deformation mechanisms.

PRELIMINARY RESULTS

TEM analysis reveals that the average grain size within the platinum pillars is 12 ± 5 nm. 74 of the 75 grains examined were between 5 nm and 25 nm in size, indicating that the pillars can be classified as nanocrystalline because all grains are below 100 nm in size. Although twin formation is known to occur in platinum (consistent with its low stacking fault energy), no twins were observed within the nanoscale grains.

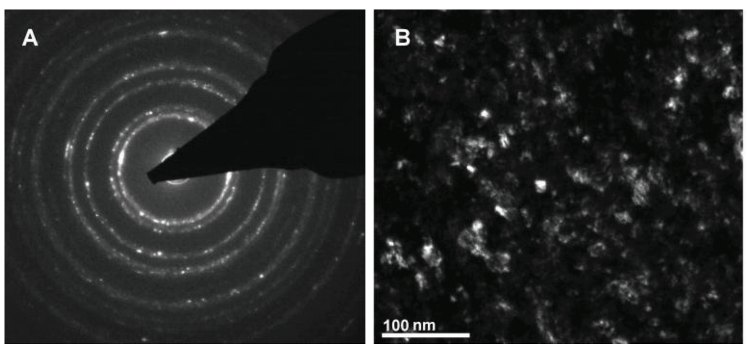

Figure 2. TEM was performed to characterize the pillar microstructure. A) Electron diffraction pattern showing nanocrystalline nature of pillar. B) Dark field images were taken to determine grain size and pillar microstructure.

Tensile tests on un-notched pillars and notched pillars are currently underway. Atomistic modeling is being performed simultaneously. This combination of experimental and computational methods should shortly yield new insights into small-scale deformation and fracture.

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