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Techniques for In Situ HVEM Mechanical Deformation of Nanostructured Materials

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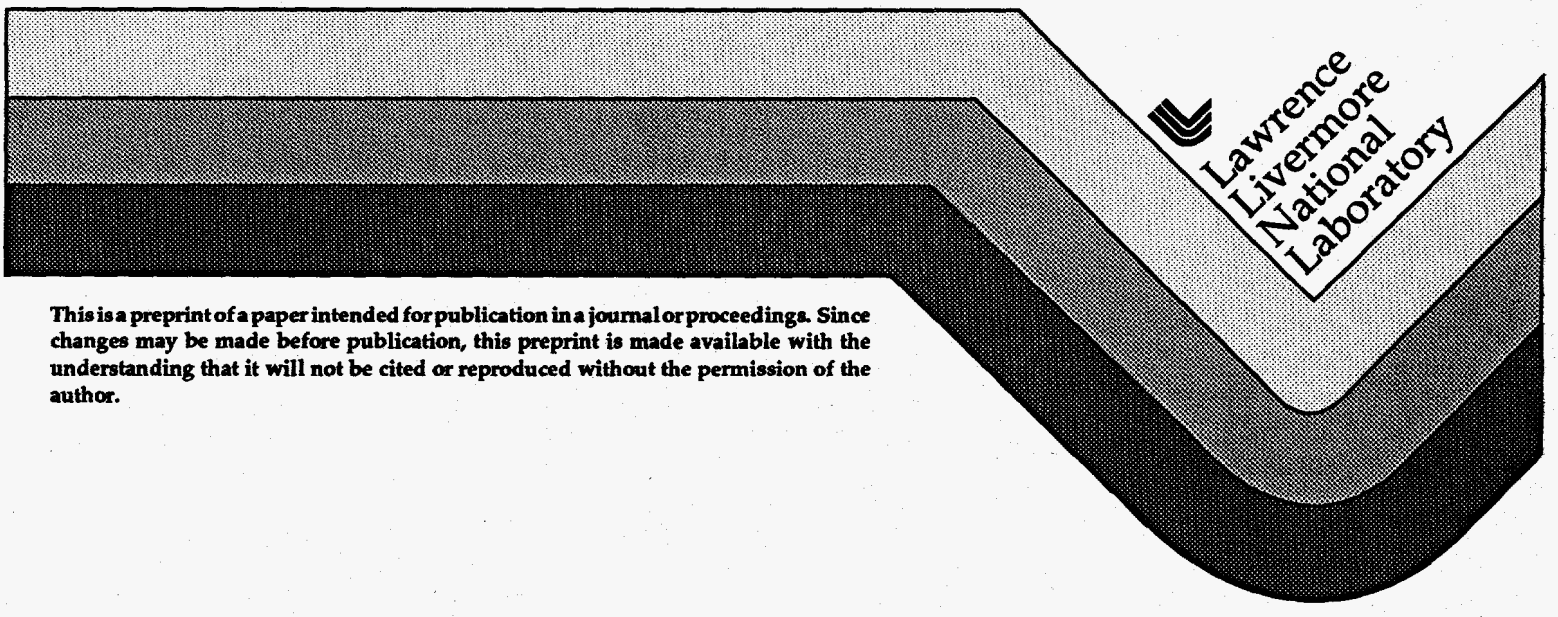

DISCLAIMER

TECHNIQUES FOR IN SITU HVEM MECHANICAL DEFORMATION OF NANOSTRUCTURED MATERIALS

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We have developed two in situ HVEM experimental techniques which allow us to begin fundamental investigations into the mechanisms of deformation and fracture in nanostructured materials. First, a procedure for the observation of tensile deformation and failure of multilayer (ML) materials in cross-section is detailed. Second, the development of an in situ HVEM nanoindentor of surfaces and films on surfaces in cross-section is presented.

Nanostructred ML materials often exhibit enhanced physical properties [1] such as increased hardness and strength. There have been few (if any) direct observations of the mechanisms by which nano-scale structures affect deformation thereby enhancing mechanical performance. Nanoindenting [2] has become a primary technique for measuring the mechanical properties of small volume materials. The mechanical properties of small volumes can vary greatly from bulk values. The elastic and plastic response and microstructure evolution in these small volumes under the indentor tip has never been dynamically observed before. Observation of the behavior of material under these conditions would no doubt further our understanding of the mechanical behavior of nanostructured materials.

Our development of in situ tensile deformation technique of multilayers in cross-section is focused upon ML sample design and specimen preparation. Sample design consisted of the synthesis of a ML sample having materials with different mechanical properties, a small period (<100nm) yet large enough to view at moderate magnification and a large total thickness for handling during specimen preparation. A Cu/Zr ML with a period of 90nm (Cu 80nm/Zr 10nm) and a total thickness of 120 μm was synthesized by magnetron sputter deposition. Specimens were prepared by a modification to a technique detailed by Wall,1994 [3]. Free standing ML foils were electroplated to bulk dimensions, sliced, lapped, dimpled and low angle ion milled. The final step was to the use a focused ion beam to mill a micro gauge section. This determines the location at which deformation and fracture will be first observed at high magnification. Figure 1 is SEM micrograph of the micro gauge section in the tensile specimen. The as deposited structure in the micro gauge section is shown in figure 2a, point P. After straining and observing dislocation motion within the Cu layers a series of Zr layers failed and Cu layers deformed to form a row of voids and ligaments, (figure 2b). The voids and ligaments form a crack which under continued loading grows by necking of the Cu layers. Crack growth continues by void formation in front of the crack tip and Cu ligament elongation, leading to failure.

In situ nanoindenting of surfaces in cross-section required a new specimen holder design that had to be built and tested. The holder features 3 axis mechanical and piezo position control and a replaceable indentor tip. Cleaved or cross-sectioned samples are mounted on a removable specimen platform. The results of initial testing are shown in figure 3. The sapphire indentor is positioned on the surface of a cleaved Si wedge in cross-section, (fig. 3a). The estimated specimen thickness is 3 μm. When the indentor is depressed into the surface the elastic response of the Si is seen by the formation of strain contours radiating out from the indentor tip, (fig.3b). Further indenting plastically deforms the Si, (fig. 3c).

Correspondence of the microstructural scale and the imaging capabilities of the HVEM make it productive to apply in situ characterization techniques to nanostructured materials. These two novel techniques provide two new tools which will generate new insight into the micromechanisims of deformation and failure in nanostructured materials.

All in situ experiments were performed on a Kratos 1.5Mev HVEM at Lawrence Berkeley Laboratory.

References

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4. The authors wish to thank Tim Weihs and Richard Gross of Lawrence Livermore National Laboratory and Doug Owen of Lawrence Berkeley Laboratory for their scientific and technical support. This work was performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under contract W-7405-Eng-48.

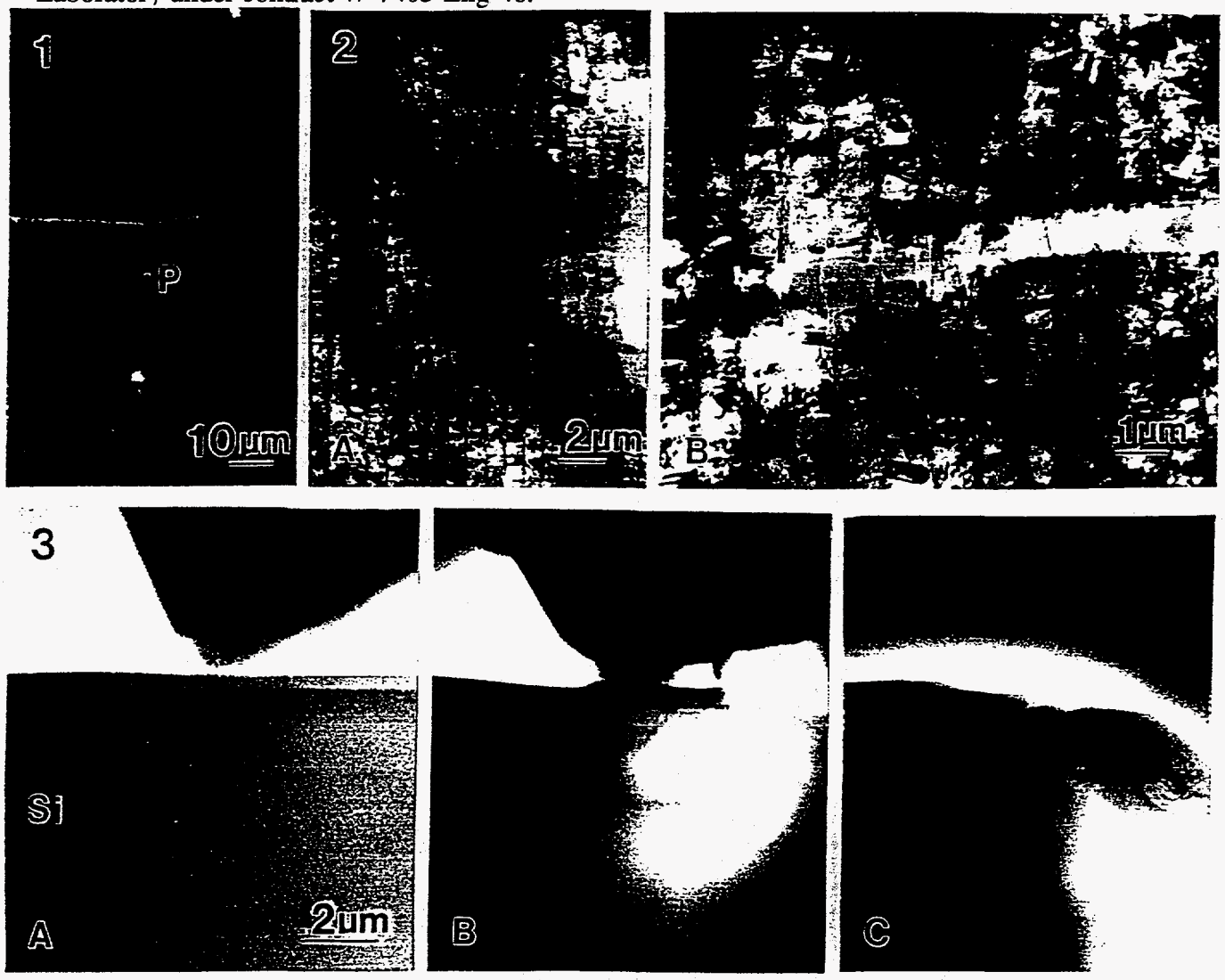

FIG. 1.-SEM image of the micro gauge section in a multilayer cross-section tensile specimen. Point P is the electron transparent viewing region during the tensile deformation experiment.
FIG. 2.-(a) The as deposited Cu/Zr multilayer structure viewed in cross-section at point P in figure 1; (b) Crack formation consisting of voids originating at the Zr layers and elongated Cu ligaments.
FIG. 3.-(a) A sapphire indentor tip on the surface of a cleaved chip of Si oriented in cross-section; (b) Indentor tip elastically deforming bulk Si; (c) Indent and plastic deformation of bulk Si.