LA-UR- 00 - 320

Approved for public release;
distribution is unlimited.

Title: NANOINDENTATION STUDY OF AMORPHOUS METAL
MULTILAYERED THIN FILMS

Author(s): J.B. Vella, Johns Hopkins Univ
R.C. Cammarata, Johns Hopkins Univ
T.P. Weihs, Johns Hopkins Univ
C.L. Chien, Johns Hopkins Univ
A.B. Mann, Johns Hopkins Univ
H. Kung, MST-8

Submitted to: Materials Research Society Meeting
Symposium: Thin Film Mechanical Properties & Stresses
November 28-December 2, 1999
Boston, MA

Los Alamos

NATIONAL LABORATORY

Los Alamos National Laboratory, an affirmative action/equal opportunity employer, is operated by the University of California for the U.S. Department of Energy under contract W-7405-ENG-36. By acceptance of this article, the publisher recognizes that the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or to allow others to do so, for U.S. Government purposes. Los Alamos National Laboratory requests that the publisher identify this article as work performed under the auspices of the U.S. Department of Energy. Los Alamos National Laboratory strongly supports academic freedom and a researcher's right to publish; as an institution, however, the Laboratory does not endorse the viewpoint of a publication or guarantee its technical correctness.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, make any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

NANOINDENTATION STUDY OF AMORPHOUS METAL MULTILAYERED THIN FILMS

J. B. Vella*, R. C. Cammarata*, T. P. Weihs*, C. L. Chien**, A. B. Mann*, and H. Kung***,

*Department of Materials Science and Engineering, Johns Hopkins University, Baltimore, MD 21218. USA.

**Department of Physics and Astronomy, Johns Hopkins University, Baltimore, MD 21218. USA.

***Materials Science and Technology Department, Los Alamos National Lab, Los Alamos, NM 87545. USA.

ABSTRACT

Nanoindentation studies were performed on amorphous metal, multilayered thin films containing alternating layers of $\text{Fe}_{50}\text{Ti}_{50}$ and $\text{Cu}_{35}\text{Nb}_{65}$ in order to investigate the mechanism for plastic deformation in metallic glass. Films with a total thickness of $1\mu\text{m}$ and bilayer repeat lengths ranging from 2 to 50 nm were magnetron sputter-deposited onto sapphire substrates. In contrast to many crystalline multilayered systems, where large hardness enhancements have been observed when the bilayer repeat length is reduced below about 10 nm, no significant hardness enhancement as a function of bilayer repeat length was observed in the $\text{Fe}_{50}\text{Ti}_{50}/\text{Cu}_{35}\text{Nb}_{65}$ amorphous metal system. This result suggests that a dislocation-like mechanism for plastic deformation may not be appropriate for these amorphous metals.

INTRODUCTION

Metallic glasses remain a class of materials whose extraordinary properties have in large part yet to be fully employed in technological applications. Although some glassy alloys have been shown to far exceed the tensile strengths and hardnesses of their crystalline counterparts, their stringent processing requirements have severely limited their application as a structural material.¹ Progress is being made in metallic glass fabrication technology as alloying combinations and ratios continue to be refined to increase the glass transition temperature. It is therefore important to gain an understanding of the microscopic processes involved in the mechanical behavior of these materials as they begin to see broader application.

In attempt to understand the plastic deformation mechanism of metallic glass, two broad schools of thought have been developed. Davis and Gilman originally proposed a dislocation-like defect to be present in amorphous materials that governs plastic deformation, arguing that in its most general sense, a dislocation can be defined as the boundary between slipped and unslipped material.² They offer as partial evidence the fact that linearly propagating slip planes at zones of maximum shear have been observed in plastically deformed metallic glass, analogous to slip observed in crystalline materials. However, Spaepen and Turnbull have proposed instead that inhomogeneous flow in metallic glasses is driven by free volume fluctuations.³ This fluid-like flow mechanism is

RECEIVED

OCT 04 2000

OSTI

compelling because it utilizes the liquid-like amorphous atomic structure to focus on viscosity differences in a stressed amorphous specimen. For example, local dilations can be visualized in zones of maximum shear giving rise to higher concentrations of free volume and a decrease in material viscosity in this area. This decrease in viscosity can introduce an almost fluid-like layer on which material can flow, thus generating a shear band. This theory explains quite well why fluid-like vein patterns appear on metallic glass crack surfaces.⁴

In order to investigate the mechanism of plastic flow in amorphous metals, the mechanical behavior of $\text{Fe}_{50}\text{Ti}_{50}/\text{Cu}_{35}\text{Nb}_{65}$ multilayered, metallic glass thin films was investigated by nanoindentation. Several crystalline, multilayered metallic thin film systems have displayed large enhancements when the bilayer repeat length was reduced below about 10 nm^{5,6}. The large hardness enhancements are believed to result from dislocation interactions with the large density of interfaces that retard dislocation multiplication and motion. Possible origins for these retarding forces are image forces resulting from a modulus mismatch between the layers and the difficulty associated with Orowan bowing of a dislocation in a thin layer of material.⁵ If plastic deformation in metallic glass is governed by a dislocation mechanism, it might be expected that multilayered amorphous metal thin films would also display significant hardness enhancements at small bilayer repeat lengths. On the other hand, the free volume model would not predict any significant hardness enhancement when the bilayer repeat length is made small.

EXPERIMENTAL METHOD

Multilayered amorphous metal thin films were DC magnetron sputtered from sputter guns containing powder pressed $\text{Fe}_{50}\text{Ti}_{50}$ and $\text{Cu}_{35}\text{Nb}_{65}$ targets. Argon pressures were maintained at 10mT throughout sputtering at a base pressure of 10^{-7} Torr. The layering was obtained by dithering the substrate alternately over two guns. Equal layer thickness films with bilayer repeat lengths ranging from 2-50nm were deposited with a total thickness of 1 μm .

Figure 1. Sputtering Chamber. A rotating stage moved sapphire substrates over two DC powered guns. Targets were powder pressed $\text{Fe}_{50}\text{Ti}_{50}$ and $\text{Cu}_{35}\text{Nb}_{65}$. Mixing during deposition was minimized by cylindrical chimneys.

The microstructure of all films were investigated using a θ - 2θ x-ray diffractometer. High angle x-ray diffraction was used to demonstrate that the films were amorphous, and low angle x-ray diffraction was used to determine the bilayer repeat

length. This was done by obtaining low angle satellite peaks off of the (000) main beam and using a standard analysis to calculate the bilayer thickness⁷. On certain films, cross-sectional transmission electron microscopy (TEM) and electron diffraction were also performed to confirm the results obtained by x-ray diffractometry. The composition of the deposited films was determined by Auger electron spectroscopy.

The hardness and modulus of each film was measured using a Nanoinstruments Nanoindenter XP. These were obtained from the average of ten or more indents that were made to a depth of 200nm.

RESULTS AND CONCLUSIONS

Figure 2 gives x-ray diffraction (XRD) patterns obtained from monolithic $\text{Fe}_{50}\text{Ti}_{50}$ and $\text{Cu}_{35}\text{Nb}_{65}$ films. The lack of Bragg peaks indicates that these films were amorphous. Figure 3 shows an electron diffraction pattern obtained from a 20nm bilayer repeat length $\text{Fe}_{50}\text{Ti}_{50}/\text{Cu}_{35}\text{Nb}_{65}$ multilayered film. Two sets of amorphous ring patterns are evident in the figure.

Figure 2. XRD pattern of amorphous $\text{Fe}_{50}\text{Ti}_{50}$ and $\text{Cu}_{35}\text{Nb}_{65}$.

Figure 3. E-diffraction of $\text{Fe}_{50}\text{Ti}_{50}/\text{Cu}_{35}\text{Nb}_{65}$ multilayer (20nm bilayer repeat length).

A cross-sectional TEM bright field image obtained from a 20 nm bilayer repeat length film is given in Figure 4. As seen from the figure, the film had well defined, flat layers of approximately equal thickness.

Figure 4. Cross Sectional bright field TEM image of $\text{Fe}_{50}\text{Ti}_{50}/\text{Cu}_{35}\text{Nb}_{65}$ Multilayer (20nm bilayer repeat length)

Auger electron spectroscopy results indicated that the compositions of the two layer materials as deposited were approximately $\text{Fe}_{50}\text{Ti}_{50}$ and $\text{Cu}_{35}\text{Nb}_{65}$. The hardness as a function of bilayer repeat length of the multilayered films, as well as the hardness of monolithic films composed of the layer materials are given Figure 5. It is seen that no significant hardness enhancement was observed. This result is consistent with the free-volume model of plastic flow, and suggests that dislocation-mediated flow does not occur in these systems.

Figure 5. Hardnesses determined via Nanoindentation shows no significant dependence on bilayer repeat length. Hardnesses of $\text{Fe}_{50}\text{Ti}_{50}$ and $\text{Cu}_{35}\text{Nb}_{65}$ monolithic films are shown in grey.

ACKNOWLEDGEMENTS

The authors thank Mr. Anthony Wagner for his help in using the Auger spectrometer. This work was supported by the Materials Research Science and Engineering Center at Johns Hopkins, administered by that National Science Foundation.

REFERENCES

1. D. E. Polk, B. C. Gleissen, and F. S. Gardner, *Mat. Sci. Eng.*, **23**, 309 (1976).
 2. L.A. Davis, in *Metallic Glasses* (American Society for Metals, Metals Park, OH, 1976) p. 190.
 3. F. Spaepen and D. Turnbull, *Scripta Metall.*, **8**, 563 (1974).
 4. C. A. Pampillo, *J. Mater. Sci.*, **10**, 1194 (1975).
 5. G. S. Was and T. Foecke, *Thin Solid Films*, **286**, 1 (1996).
 6. R.C. Cammarata, *Thin Solid Films*, **240**, 82 (1994).
 7. R.C. Cammarata, *Nanomaterials: Synthesis, Properties, and Applications*, ed. A. S. Edelstein and R.C. Cammarata, (Institute of Physics Publishing, Philadelphia, 1996) pp. 117.
-