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The Elastic, Plastic, and Time Dependent Properties of Thin Films as Determined by Ultra Low Load Indentation

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ABSTRACT

Using a highly spatially resolved mechanical properties microprobe, the elastic, plastic and time dependent mechanical properties of sapphire and a 1.9 μm amorphous alumina film on a sapphire substrate have been studied. Young's modulus, hardness, and stress-exponent data are reported. The technique for characterizing time dependent properties via indentation (hardness versus displacement rate/displacement) are directly compared to standard uniaxial compressive techniques (stress vs strain rate) for a bulk Pb-In alloy to further quantify the relationships between the two techniques.

INTRODUCTION

Instrumented indentation techniques have long been used to measure the elastic and plastic properties of thin films[1-5]. Recently these techniques have been used to measure time dependent properties of small volumes of material[6,7,8]. Along with this new frontier of research comes a completely new set of problems, the answers to which can be masked by hardware characteristics, thermal anomalies, and new questions concerning "steady state" behavior. The problem of relating indentation properties, i.e., displacement, displacement rate, and hardness to conventional uniaxial testing parameters such as strain, strain rate, and flow stress must also be considered. The relationship between hardness and flow stress has been addressed by Tabor[9]. The results of his study showed that hardness is approximately 3.3 times the uniaxial stress at a characteristic strain of 8-10%. Mayo et al.[6] in an indentation study of superplasticity in Pb, Sn, and Sn-38 wt% Pb define an indentation strain rate as the descent rate of the indenter divided by its current depth, ($1/h \cdot dh/dt$), however the relationship between this indentation strain rate and uniaxial strain rate has not yet been clearly defined. This work presents the findings of a study of the elastic, plastic, and time dependent properties of single crystal Al_2O_3 (sapphire) and a 1.9 μm electron beam deposited amorphous Al_2O_3 film. The time dependent indentation properties of a bulk Pb-65 at% In alloy are also presented and compared directly to data obtained in conventional compression tests to try to further quantify the relationships between the two types of tests.

EXPERIMENTAL EQUIPMENT AND TECHNIQUE

All of the indentation experiments were performed in a commercially available mechanical properties microprobe*, the mechanics of which are described elsewhere[3]. A load-time history of a typical experiment utilized in this study is shown in Figure 1. This type of experiment was first utilized by Mayo et al. in their study of nanophase TiO_2[7]. However, in the experiment used in this study, the indenter was step loaded from the point of contact to the desired load after which the load was held constant and the displacement monitored as a function of time. This type of loading enabled very high displacement rates

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* The Nanoindenter®, Nano Instruments, Inc., Knoxville, TN.

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to be achieved during the first portion of the constant load segment. Data was initially acquired at a rate of 3 data points per second during the initial rapid descent of the indenter. The data acquisition rate was slowed to approximately one data point every five seconds after the first fifteen seconds of the experiment. The hold segment was continued until a specified minimum displacement rate was reached. The indenter was then step unloaded to a load of 20 µN, held at constant load, and then ramp reloaded and ramp unloaded to obtain the elastic and plastic properties of the material. This experiment was carried out on two materials: sapphire,and a 1.9 µm electron beam evaporated amorphous Al_2O_3 film on sapphire. The peak loads used were 82, 41, 20.5, 10.25, 5.14, and 2.57 mN. Hardness and modulus data were calculated using standard mechanical properties microprobe analysis techniques[3, 10].

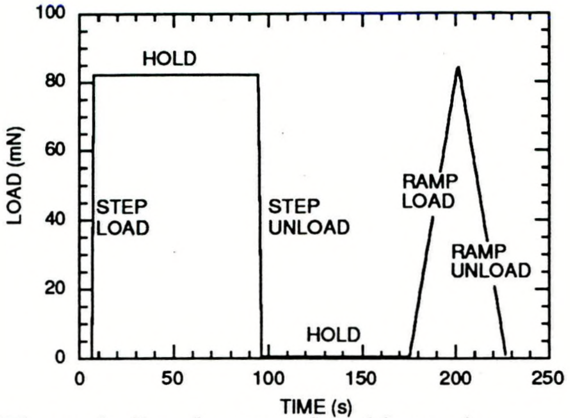

Figure 1. Load versus time history in strain rate sensitivity test.

In order to attempt to quantify the time dependent data obtained via indentation, a series of indentation creep tests and compression tests were performed on a bulk Pb-65 at% In alloy. This alloy was chosen for its low melting point (T_m=195° C) and well-characterized properties [11]. The Pb-65 at% In alloy used for both indentation and compression testing was prepared from commercially obtained Pb and In of greater than 99.999% purity. The indentation specimens were prepared by rolling the homogenized alloy to approximately 0.75 mm and then annealing for 1.5 hours at 170° C the resulting grain size was approximately 1 mm. The compression specimens were prepared by swaging an as-cast 3/8" rod to 1/4", homogenizing for 24 hours at 170° C, swaging to 1/8" and then machining to approximately a 3:2 height to diameter ratio. The machined specimens were then annealed 40 minutes at 100° C; the resulting average grain size was 50 µm.

RESULTS AND DISCUSSION

The hardness and modulus data as a function of depth for the amorphous Al_2O_3 film and the sapphire substrate are shown in Figure 2. The maximum plastic depth achieved for the amorphous Al_2O_3 was approximately four times that achieved at the same peak load for the sapphire, resulting in a factor of four difference in the hardness of the two materials. The moduli of the two materials showed similar trends. The important observations here are the large difference in the absolute values of the hardness and moduli of the two materials and the fact that the hardness and modulus of the amorphous film can be measured with little or no effect of the substrate. This is expected since the plastic depth of penetration is only roughly one-third of the film thickness. An interesting note here is that the values obtained for the hardness and Young's modulus of the amorphous Al_2O_3 film agree within in a few percent of the values obtained by Oliver et al.[4] in their 1987 study of an amorphous Al_2O_3 layer formed by ion implantation.

Figure 4 shows an example of the data obtained during a constant load hold segment on the sapphire and the amorphous film. Note the wide range of descent rates experienced by the indenter and the difference in the absolute magnitude of the displacements. This data was then differentiated to obtain a displacement rate (dh/dt) for each displacement during the hold segment. No elastic correction was made to the indenter penetration. Using the displacement rate and hardness calculated at a specific displacement (h), the stress exponent of the two materials was obtained from a log-log plot of the indentation strain rate (1/h•dh/dt) versus hardness. The results of this analysis appear in Figures 5 and 6.

The wide range of indentation strain rates shown in Figures 5 and 6 demonstrates the utility of the indentation experiment. The extremely high values of the stress exponent are indicative of high strain rate tests performed at small fractions of the melting temperature. The importance of obtaining all of the data for the stress exponent determination from one indentation to avoid indent-to-indent variations in the hardness of the material is evident, since variations in hardness have little effect on the slope of the curve, just its placement on the hardness axis.

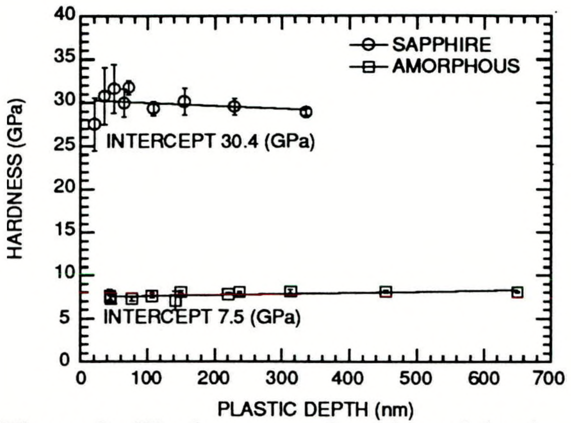

Figure 2. Hardness as a function of depth for Al_2O_3.

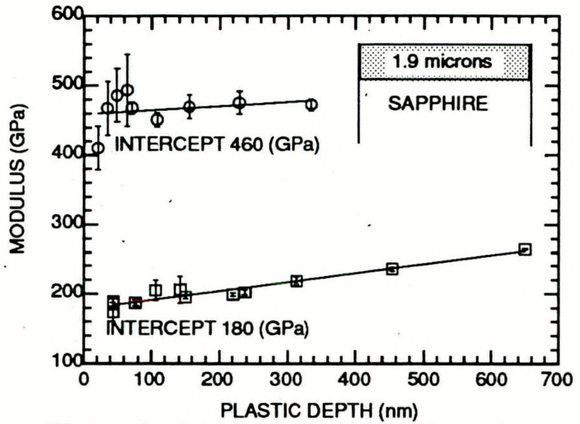

Figure 3. Modulus as a function of depth for Al_2O_3.

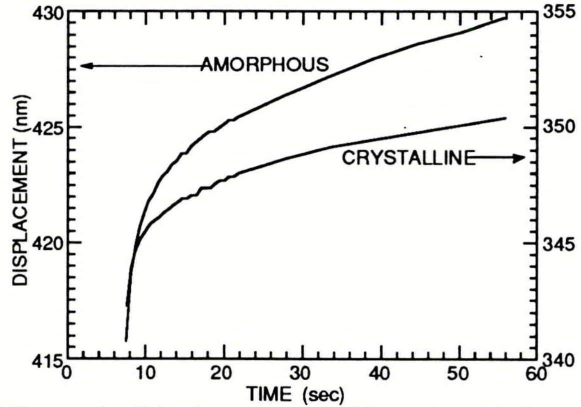

Figure 4. Displacement vs Time for Al_2O_3

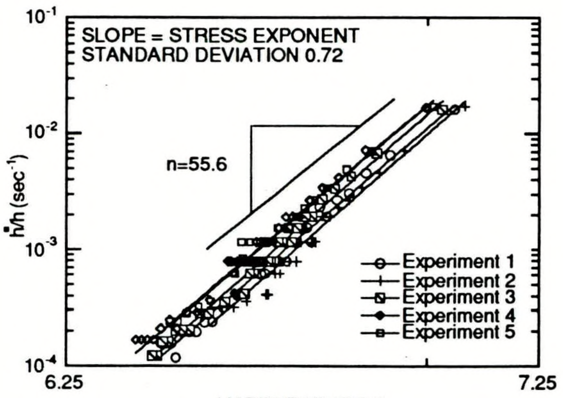

Figure 5. Log (1/h dh/dt) vs. Log H for the amorphous Al_2O_3 film.

Figure 7 shows the results of a similar analysis of the indentation creep properties of the Pb-In alloy. Again, the importance of obtaining all of the data from a single indentation is evident from the fact that indent-to-indent variations in the hardness of the material have little effect on the overall shape of the curve, just its placement on the hardness axis. A stress exponent of 90 was obtained at the high strain-rate portion of the curve with the slope approaching 6 as the strain rate decreases. The fact that the indentation stress versus strain rate curve can be described in terms of power law behavior suggests that some "steady state" is being reached and that a correlation could exist between the indentation data and the compression data at steady state, however the correlation of the indentation creep data with the compression data does not lend itself to such an easy

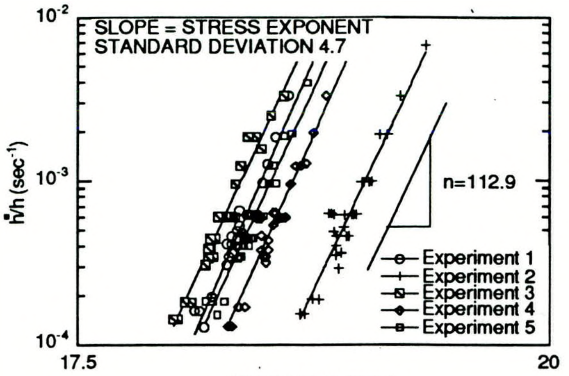

Figure 6. Log (1/h dh/dt) vs. Log H for single crystal Al_2O_3.

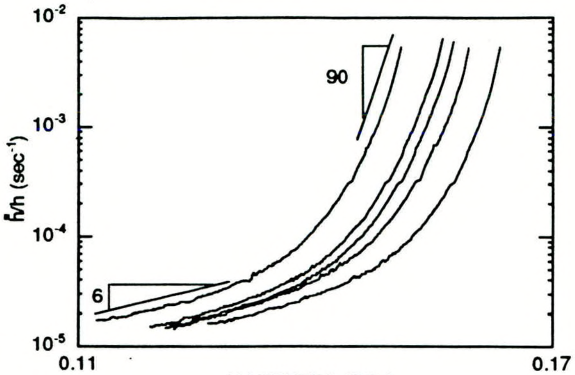

Figure 7. Log (1/h dh/dt) vs. Log H for Pb-65 at% In .

analysis. Figure 8 shows the stress-strain rate results of the compression study of the Pb-In alloy as a function of strain. Note that the stress exponent that one determines from the compression data is highly dependent on the percent strain at which the stress-strain rate pairs are tabulated.According to Tabor[8], the indentation hardness of a material should be approximately 3.3 times its strength at a characteristic strain of 8-10%. When the indentation hardness data are divided by 3.3 and plotted versus the indentation strain rate along with the compression stress-strain rate data at 10% strain, the plot shown in Figure 9 results. The striking similarity of these two curves implies that indentation creep is not a steady state process, but is being controlled by transient mechanisms, i.e. a volume of material that has undergone approximately 10% strain. This conclusion agrees with Atkins et al.[12] in that indentation creep behavior reflects the transient creep properties of the material. In their work, Atkins et al. find that it is possible to describe the indentation hardness properties of solids in terms of a transient-creep equation of state. The fact that the indentation stress-strain rate data obtained in this study very nearly approximate compressive stress-strain rate data at a strain of 10% lends validity to these conclusions.

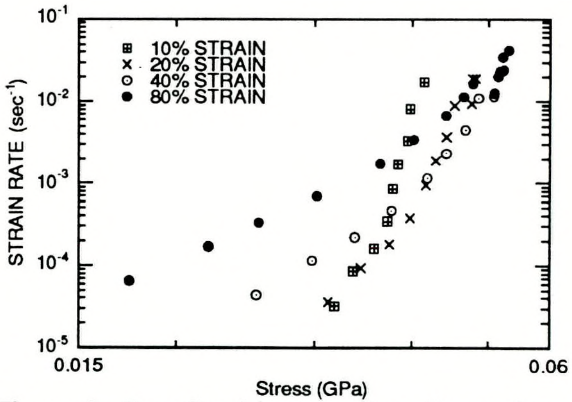

Figure 8. Log Strain rate vs Log Stress for for Pb-65 at% In as a function of strain in the material.

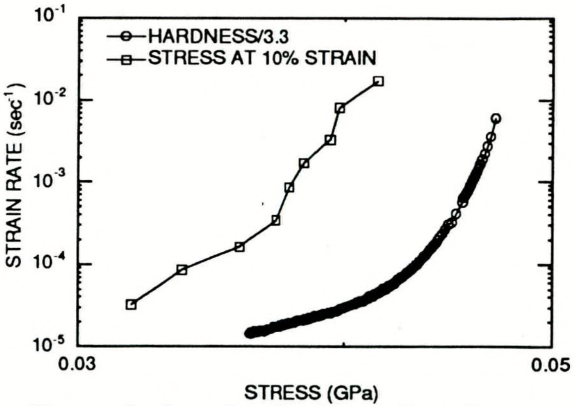

Figure 9. Log Strain rate vs Log Stress for Pb-65 at% In.

CONCLUSIONS

Indentation techniques can be successfully applied to creep testing of both bulk materials and thin films with very high stress exponents. The technique allows the determination of the stress exponent over three orders of magnitude of strain rate in a single experiment. By obtaining all of the data from a single experiment, error due to indent-to-indent variations in the hardness of the material can be avoided.

The work on Pb-In is but a small step toward fully understanding indentation creep and its correlation with conventional uniaxial mechanical properties testing. The indentation stress-strain rate data does approximate the compression stress-strain rate data at a strain of 10%, suggesting that indentation creep is controlled by transient mechanisms.

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