TIME DEPENDENT INDENTATION TESTING AT NON-AMBIENT TEMPERATURES UTILIZING THE HIGH TEMPERATURE MECHANICAL PROPERTIES MICROPROBE

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ABSTRACT

Time dependent indentation data for pure indium from -100 °C to 75 °C is presented. The properties reported include hardness, indentation strain rate, stress exponent and apparent activation energy for creep. These properties were measured using a depth-sensing indentation system capable of performing experiments between -100 °C and 300 °C in ultra-high vacuum. In addition, by employing laser interferometric techniques, this system can obtain displacement data with time constants as low as 50 ns. This allows the investigation of the material response to very fast stress changes over an extremely wide range of strain rates. The adverse and beneficial dynamic effects of step-loading the indenter into the surface of the material will be discussed. Initial results obtained from this type of experiment show that it is possible to obtain energy dissipation or damping information from the material being studied.

INTRODUCTION

Depth-sensing indentation testing involves the forcing of an indenter of known geometry into a planar surface while continuously recording the load on the indenter, the displacement of the indenter into the surface, and the time of the experiment. The resulting load-displacement-time data can then be used to determine certain mechanical properties of the material. The most commonly measured properties are the Young's modulus,[1], the hardness[1,2], and certain time-dependent properties of the material such as the stress exponent for creep or the strain rate sensitivity[3-5]. The equipment used for this study is capable of data acquisition rates as high as $1.9 \times 10^5 \text{ s}^{-1}$ and test temperatures between -100 °C and 300 °C. This unique combination of capabilities allows further investigation into the materials dynamic deformation response and into the mechanisms actually controlling the rate of deformation.

It is well documented that hardness can be related to the uniaxial flow strength[5,6]. Recent work has shown that the indentation strain rate ($\dot{\epsilon} = \frac{1}{h} \frac{dh}{dt}$)[3] can be related to the uniaxial strain rate[7]. These relationships together with the ability to change the test temperature allows one to determine the activation energy for rate controlling mechanisms by tabulating indentation strain rate data as a function of test temperature at a constant hardness. It should be noted in this study that the hardness is defined as the load divided by $24.5h_t^2$ where h_t is the total depth of the indentation. The work presented here represents data acquired for pure indium during the initial testing and characterization of the new High Temperature Mechanical Properties Microprobe(HTMPM). Although the data presented here is from a bulk material, the results of this work show tremendous possibilities for future work on thin film materials.

EXPERIMENTAL EQUIPMENT AND TECHNIQUES

All of the experiments discussed in this work were performed with the new High Temperature Mechanical Properties Microprobe (HTMPM) at the Oak Ridge National Laboratory. The HTMPM is a low load, ultra-high vacuum (UHV), high temperature, continuous depth sensing indentation system. It combines much of the technology found in the current ambient temperature mechanical properties microprobes with the ability to perform

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similar experiments at temperatures ranging from $-100\text{ }^{\circ}\text{C}$ to approximately $300\text{ }^{\circ}\text{C}$ in a variety of atmospheres.

The physical structure of the indentation system is very similar to the Nanoindenter II®[1]. The resolution of the loading system is $1\text{ }\mu\text{N}$. One primary difference is that displacement measurements in the HTMPM are achieved through the use of an Axiom 2/20™ He-Ne laser interferometric displacement sensing system manufactured by the Zygo Corporation. The complete interferometric system is mounted outside of the vacuum chamber. The laser beams are passed into the vacuum chamber through a strain-free sapphire viewport. Two of the beams from the interferometer strike a mirror that is clamped behind the indenter while the other two beams strike a mirror that is rigidly coupled to the specimen. This configuration allows for a direct measure of the displacement of the indenter into the specimen with very little possibility for adverse contributions from dimensional instabilities in the load frame. The resolution of the displacement system is 0.125 nm .

The Axiom 2/20 system is capable of producing displacement measurements with a time constant as short as $0.05\text{ }\mu\text{s}$. By conducting dedicated reads from the laser system to the data acquisition system, current hardware allows short "bursts" of displacement data to be acquired at rates up to $190,000\text{ data points/s}$. These "bursts" of displacement readings can be performed at any time during the experiment, for example during a step-load, without interrupting normal experimental flow. The burst data is then retrieved on completion of the experiment. This very exciting feature of the measurement system gives the HTMPM unique capabilities not found in other mechanical properties microprobes.

Temperature monitoring in the HTMPM is achieved through the use of one thermocouple located directly on the indenter tip and another located just below the specimen block. Digital controllers independently monitor the temperature of the indenter and the specimen and control two independent power supplies for heating. The resolution of the temperature measurement is $0.2\text{ }^{\circ}\text{C}$. Heating is achieved through the use of tungsten and tantalum resistance heaters, one located just above the indenter, the other located in the specimen stage. Temperatures less than ambient are achieved by flowing liquid nitrogen through cooling channels in the copper shroud that contains the indentation head and the copper stage which supports the specimen block.

The results of two types of experiments will be reported in this work. The first involved ramping to some fraction of the maximum load immediately followed by a step to the maximum load. The step load required less than 10 ms to achieve full load. The second involved ramping the load at a constant loading rate, applying the full load in approximately 1 s . The remaining segments of the two experiments were similar in that each involved a hold segment at constant load to allow longer term displacement versus time or indentation creep data to be obtained. Indentation creep data in the $-100\text{ }^{\circ}\text{C}$ to $75\text{ }^{\circ}\text{C}$ range was obtained using the 1 s ramp loading scheme. All of the results reported here are for a high purity, large-grained, electro-polished indium. This material was chosen as it was possible to cover a large range of homologous temperature operating in the $-100\text{ }^{\circ}\text{C}$ to $75\text{ }^{\circ}\text{C}$ range.

RESULTS AND DISCUSSION

Step Loading

Fig. 1 is a semi-log plot of the displacement vs. time response of the indenter obtained for a variety of loading conditions all to a maximum load of 10 mN . The upper four curves show the response to an applied step load with varying amounts of the total load being applied during the ramp prior to the step to maximum load. The percentage of P_{max} stepped on at $t=0$ is indicated for each curve. There are several important features to note from these four curves. First, note the wide range of descent rates experienced by the indenter as indicated by the span of eight orders of magnitude on the time axis. This reveals one of the useful aspects of the interferometric technique being used for the displacement measurement. Secondly, the depth achieved immediately following the step load is largest for the largest percentage P_{max} stepped

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Fig. 1. Displacement vs. time response for different loading conditions

on. This is believed to be due to the large inertial or dynamic forces that are being experienced by the specimen as are evident from the oscillatory response of the indenter following the applied step as shown in "A" in Fig. 1 and enlarged in Fig. 2. Thirdly, immediately following the step load, an incubation period is observed where the displacement rate of the indenter is observed to be smallest for the largest step-fraction and the four curves are seen to reverse their order in terms of overall displacement. Since under normal data acquisition conditions the first data point on these curves is obtained at approximately 1 second, the region on the time axis where these four curves intersect is not normally observed. This initial reduced creep rate is believed to be due to 1.) the stronger structure resulting from the higher strain rate experienced by the material when a larger fraction of the maximum load is applied in a step fashion and 2.) the larger contact area and therefore the lower stress that results from the dynamic overload. Finally, after the initial incubation period, the displacement rates for the four curves become similar.

Fig. 2. Oscillatory response to 9 mN step load.

Fig. 2 is an enlargement of the area marked in "A" in Fig. 1. It is a plot of displacement vs. time showing the oscillatory response of the indenter as it comes to rest during the 80 ms immediately following an applied step load of 9 mN (90% of P_{max}). Inertia calculations using the maximum acceleration experienced by the indenter reveal that a dynamic overload of as much as 40% of the applied load can be experienced by the specimen. This phenomenon is seen to decrease as the step-fraction is reduced and is completely absent when the step-fraction is reduced below 30% as shown in Fig. 1. A number of interesting observations can be

made about the observed oscillation following the step-load. The natural frequency of the oscillation, which is determined predominantly by the stiffness of the contact and the mass of the indenter, is observed to be 975 cycles/s (6126 rad/s). This together with the known mass of the indenter yields a contact stiffness of 3.3×10^5 N/m. If the contact area of the indentation is calculated from the mean displacement of the oscillation, this along with the calculated

stiffness yields a modulus of 12.8 ± 0.2 GPa, very close to the literature value of 12.7 GPa. It is also possible from the exponential decay of the oscillation to calculate an effective damping coefficient of 7 N s /m. After careful characterization of the dynamics of the indentation system, it has been determined that this number represents the damping or energy absorbed by the sample material in the vicinity of the indentation and is not energy absorbed elsewhere in the system. More work to characterize this capability of the HTMPM is currently being conducted.

Ramp Loading

Also included in Fig. 1 is the displacement vs. time response obtained during a 1 s ramp to P_{max} . Note from the response that no overload effects were evident and that the displacement vs. time data obtained past 1 s is nearly identical to that obtained during the hold following the 30% step-fraction. This seems to indicate that dynamic loads are small and can be ignored. This means that the actual load experienced by the sample increases throughout the experiment. The remainder of the data discussed in this work was obtained using the load ramping scheme at temperatures ranging from -100 °C to 75 °C.

Fig. 3 is a plot of the displacement vs. time results obtained during the constant load hold segment immediately following the load application as a function of the test temperature. The data is terminated after 40s for the low temperatures due to longer term system stability problems at temperatures less than ambient. Some important features to note from these curves are 1.) the displacement achieved by the indenter is less as the temperature is reduced indicating the material is capable of supporting greater stresses and 2.) the sharpness of the break in the curves at lower temperatures suggests a very high stress exponent, i.e., the stress is becoming nearly constant very quickly. Fig. 4 shows the results of the hardness vs. test temperature calculated from the applied load and the displacement at 15 s. It is observed that over the homologous temperature range of 0.4 to 0.8 the hardness decreases by over an order of magnitude.

Fig. 3. Displacement vs. time as a function of test temperature.

Fig. 4. Hardness at 15 s as a function of homologous temperature.

By differentiating the displacement versus time data, the displacement rate of the indenter can be obtained continuously during the constant load hold segment. The indentation strain rate can then be obtained by dividing the instantaneous descent rate of the indenter by the displacement at that point in time. In this way, one can produce plots of indentation strain rate vs. hardness analogous to strain-rate vs. stress curves obtained using conventional bulk testing techniques. Fig. 5 is a log-log plot of indentation strain rate vs. hardness for experiments conducted at ambient conditions (28 °C), 50 °C and 75 °C. The lower temperature data is not included due to the previously mentioned experimental stability problems. All of the data exhibit similar trends with a slope of approximately five with the ambient temperature data

Fig. 5. Log indentation strain rate vs. Log hardness as a function of temperature.

Fig. 6. Natural log indentation strain rate vs. $1/T$.

beginning to exhibit what appears to be power-law breakdown behavior at the higher strain rates.

At temperatures above $0.5 T_m$, creep in pure metals is thought to be controlled by the climbing of dislocations out of their glide planes to overcome obstacles to motion. The climb velocities are limited by the rate at which vacancies can diffuse in the material. As such, creep at elevated temperatures is expected to follow an Arrhenius type of behavior of the form $\dot{\epsilon} = \dot{\epsilon}_0 \exp(-Q/RT)$ where Q is the activation energy, R is the gas constant and T is the absolute temperature. A semi-log plot of strain-rate vs. $1/T$ will then yield the activation energy for the rate controlling mechanism. Fig. 6 is a semi-log plot of indentation strain rate at constant hardness vs. $1/T$ for experiments conducted between 28 °C and 75 °C. The apparent activation energy for the deformation process obtained by a linear fit to the data is 77.9 kJ/mol. This is in excellent agreement with the activation energy for self-diffusion in pure indium which is reported to be in the range 75-78 kJ/mol[8,9] and in accord with the observation that the activation energy for creep in pure metals is nearly equal to the activation energy for self diffusion[10].

Shown in Fig. 7 is a log-log plot of temperature compensated indentation strain rate vs. hardness for the three sets of data shown in Fig. 5. The fact that all three sets of data, when compensated for the test temperature, can be fit by a single curve supports the premise that the deformation is being controlled by a single thermally active process. The slope of five agrees with observed values for stress exponents in pure metals obtained from conventional means where the rate controlling process is dislocation climb[11].

While a limited amount of indentation work at elevated temperature has shown that the activation energies for indentation creep are similar to those observed in bulk specimens[12-14], the majority of this work has been done using hot hardness tests [12,13] or by impression creep at large displacements[14]. Investigations into the time dependent properties of thin films using depth-sensing indentation techniques have been limited to the determination of stress exponents for creep and have not dealt with activation energies for rate controlling mechanisms[3-5]. The ability to obtain activation energies for creep processes from depth-sensing indentation creep techniques will now make it possible to begin to explore more fully the mechanisms responsible for time dependent deformation in thin film materials.

CONCLUSIONS

1.) Step-loading large fractions of the applied load results in significant dynamic loads. Valuable information about time dependent properties such as damping can be gained from the resulting overload.

Fig. 7. Temperature compensated indentation strain rate vs. hardness for ambient, 50 °C, and 75 °C data.

2.) The hardness as a function of test temperature of pure indium was found to range from 0.19 GPa at -100 °C to 0.012 GPa at 75 °C. The room temperature modulus of the material was determined to 12.8 GPa.

3.) The apparent activation energy for indentation creep at temperatures greater than ambient was determined to be 77.9 kJ/mol. This is nearly equal to the activation energy for self diffusion in the material. The stress exponent for creep was determined to be approximately five in accord with uniaxial data where dislocation climb is the rate controlling mechanism. This suggests that the mechanisms responsible for elevated temperature creep during indentation are the same as those observed during standard bulk creep experiments.

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