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MICROHARDNESS TESTS FOR
HIGH-ENERGY NEUTRON-SOURCE EXPERIMENTS

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MICROHARDNESS TESTS FOR HIGH ENERGY NEUTRON SOURCE EXPERIMENTS

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In a development effort to extract mechanical property information from miniature specimens, standard diamond pyramid microhardness (DPH) tests have been conducted at Hanford Engineering Development Laboratory (HEDL) on specimens irradiated in RTNS-II; and techniques to extend the information available from microhardness tests have been developed at the University of California, Santa Barbara (UCSB). In tests at HEDL, radiation hardening has only been observed in relatively pure materials irradiated to neutron fluences less than 4×10^{17} n/cm². In copper, specifically, a proportional increase in the DPH with neutron fluence has been observed, and this microhardness increase has been correlated with an increase in the 0.2 percent offset yield strength. At UCSB it has been found that hardness and microhardness data obtained with spherical indenters can be used to determine the true stress-true plastic strain relationship of the test material; moreover, it has been found that features of the indentation lip geometry can be used to characterize localized flow phenomena like Lüders strain in steel. Consequently, microhardness test techniques appear attractive for small specimen test applications.

1. INTRODUCTION

A fundamental understanding of the effects of high energy neutron irradiation on the microstructure and mechanical properties of materials is requisite for the development of alloys suitable for use in fusion devices. To this end, the test environments of high energy neutron sources, such as the Rotating Target Neutron Source (RTNS-II), will be used over the near term to obtain the necessary data to develop this understanding. However, because of the volume constraints in such devices, a key element in the fusion materials program is the development of test techniques to extract mechanical property information from specimens which are small and have perhaps a multipurpose geometry. Transmission electron microscopy (TEM) discs are an example. Moreover, it is desirable to develop mechanical test techniques which are nondestructive or at least pseudo-nondestructive so that a single specimen can be used either for multiple testing or for periodic tests in an extended irradiation or post-irradiation conditioning schedule.

Potentially, microhardness testing is one such technique. Microhardness tests are applicable to most specimen geometries, they only damage a small fraction of the sample per indentation, and the data can be used to indicate relative hardening.

Consequently, standard diamond pyramid microhardness (DPH) tests have been conducted at Hanford Engineering Development Laboratory (HEDL) to investigate the utility of this test technique for characterizing radiation hardening in materials irradiated in RTNS-II. In addition, techniques to increase the information obtainable from a microhardness

test have been developed in a concurrent program at the University of California, Santa Barbara (UCSB); this work has concentrated on static indentation tests with a spherical indenter, i.e., ball-microhardness tests. The results obtained to date in both programs are presented below.

2. STANDARD DPH TESTS

As described in detail elsewhere [1], a large part of the HEDL program to utilize RTNS-II involves the irradiation of TEM disc specimens at different temperatures to different neutron fluences. To date, 26 metals in a total of 39 metallurgical conditions have been irradiated in 4 different capsules at ambient temperature to peak neutron fluences of 4×10^{17} n/cm². The capsules have been designed to hold a large number of samples; and because of the large flux gradients in the capsules, samples have received a range of doses up to this peak fluence.

Each type of metal irradiated in these capsules has been subjected to DPH testing. Both prior to and following irradiation, microhardness tests have been performed on selected TEM discs using, at minimum, a 50 gram load and 3 indentations to obtain an average microhardness number.

Only 3 of these materials, pure copper, pure nickel, and an Fe-Cr-Ni ternary, have exhibited significant hardening. For each of these materials, the DPH was observed to increase monotonically with increasing neutron fluence. For copper the DPH was approximately 50% greater after a neutron fluence of 4×10^{17} n/cm²; for Ni, 40% after 3×10^{17} n/cm²; and for the Fe-Cr-Ni ternary, 30% after 4×10^{17} n/cm².

In the case of copper, referred to here as DAFS copper, some attempt has been made to correlate the DPH data with the tensile data reported by Mitchell for Cominco and LLNL copper irradiated to similar neutron fluences at 25°C in RTNS-I [2]. Two approaches have been taken. In the first approach, the following relation between the yield strength of copper and DPH was assumed.

$$\sigma_y = K \text{ DPH } (B)^n \quad (1)$$

where σ_y is the 0.2 percent offset yield strength expressed in MPa, K is a numerical constant equal to 3.27, B is another constant taken as 0.1 and DPH is expressed in kg/mm² [3]. The parameter n is the strain hardening exponent of the material, and it was taken as 0.414 for unirradiated copper. It was found that while the change in the yield strength derived from the DPH data with Eqn. (1) agreed with that measured by Mitchell at low neutron fluences, at fluences greater than $\sim 5 \times 10^{16}$ n/cm² it was lower by a factor of 2.

This discrepancy does not appear to be a result of chemistry or damage differences among the materials. The Cominco, LLNL and DAFS copper were all irradiated in the recrystallized condition. The Cominco and LLNL copper had a solute content of 25 ppm while the DAFS copper has a solute content of 500 ppm; however, the microstructure of the irradiated metals showed no significant differences in the number or size of visible defects, and it appears that solute contents up to as much as 5 atomic percent do not affect the relative radiation hardening of copper binary alloys [4].

A more likely explanation of the discrepancy is that the value of n decreases with neutron fluence. Hence, using the relatively large unirradiated value of n to correlate irradiated material hardness data to tensile data could underpredict the yield strength change. Efforts are currently in progress to evaluate n in the unirradiated material from spherical indentation hardness tests by techniques similar to those developed at UCSB and described later.

In an alternate approach to correlating the DPH data to the tensile data for copper it was assumed that

$$\sigma \sim \text{DPH}/3 \quad (2)$$

where σ is the flow stress of the material at some effective plastic strain characteristic of the indenter (approximately 8% for a DPH-type indenter) [5]. If the flow stress at 8% plastic strain changes as the 0.2% yield strength, it follows that

$$\Delta \sigma_y \text{ (MPa)} = 3.27 \Delta \text{DPH (kg/mm}^2\text{)} \quad (3)$$

This DPH-derived change in the yield strength is compared to the results of Mitchell in Fig. 1. As can be seen, agreement is reasonable over the range of fluences investigated.

Hence, at low doses it appears that a relationship such as Eqn. (3) reasonably correlates microhardness data to tensile data for copper. Preliminary indications are that this may be true for other materials irradiated to low doses in RTNS-II as well. However, it is not expected that such a relationship will remain valid after high doses because of the considerable loss of strain hardenability.

PROPERTY - PROPERTY CORRELATION HARDNESS - YIELD STRENGTH

Fig. 1. Correlation between measured strengthening and calculated strengthening based on hardness data for irradiated copper.

3. BALL-MICROHARDNESS TEST TECHNIQUES

To extend the information which can be obtained from a single microhardness test and to eliminate some of the empiricism used to correlate microhardness data to other flow properties, further development of the microhardness test technique has been undertaken at UCSB. The major focus has been on performing microhardness tests with spherical indenters, and interpreting the information obtained from such tests. The objective has been to determine the true stress-true plastic strain relationship of the test sample from microhardness data obtained at a single penetration location.

The selection of a spherical penetrator for this application was based on considerations of indentation geometry. For indenters like cones or pyramids, indentations made at different loads are geometrically similar; that is the ratio of the depth to the breadth of the indentation is independent of the applied load. Consequently, the effective plastic strain associated with the indentation and the load divided by the projected area of the indentation are constants for a given material. As stated previously, for example, the effective plastic strain associated with a DPH indentation is approximately 8%, and the DPH value can be related to the flow stress at this strain.

Indentations made with a spherical indenter at different loads, on the other hand, are geometrically dissimilar. That is, the depth-to-breadth ratio of the indentation increases with increasing applied load and the load divided by the projected area also increases with increasing load. Consequently, the effective plastic strain and flow stress associated with a given indentation increase with the applied load to create the indentation. The potential thus exists to determine the effective true stress-true plastic strain relationship from a single penetration if the load can be ascertained as a function of the indentation geometry.

Based on this philosophy, a set of equations was derived from the literature [5,6] to relate load and indentation geometry from a ball indentation test to the effective true stress and true plastic strain of the sample material. The equations are given as follows

$$\sigma = P_m / \psi \quad (4a)$$

$$\epsilon_p = 0.2 d/D \quad (4b)$$

$$d = \left[\frac{K \frac{D}{2} (h_p^2 + (\frac{d}{2})^2)}{h_p^2 + (\frac{d}{2})^2 - h_p D} \right]^{1/3} \quad (4c)$$

$$K = 5.47 W (1/E_1 + 1/E_2) \quad (4d)$$

$$P_m = \frac{4W}{\pi d^2} \quad (4e)$$

$$\psi = \begin{cases} 1.07 & \phi \leq 0 \\ 1.07 + .53 \ln \phi & 0 < \phi \leq 27 \\ 1.07 + .53 \ln 27 & \phi > 27 \end{cases} \quad (4f)$$

$$\phi = \frac{\epsilon_p E_2}{.43 \sigma} \quad (4g)$$

where σ = true flow stress at a true plastic strain ϵ_p

W = applied load

E_1 = elastic modulus of the ball

E_2 = elastic modulus of the specimen

and the parameters d , h_p , and D are described schematically in Figure 2. These relations appear to be general for a wide range of metals; that is, the parameters are not material specific. The only caveat is that the σ - ϵ values calculated from hardness data are those which would be obtained in a tensile test performed at the same effective strain rate as the hardness tests. Hence, some difficulties can arise in correlating hardness data and tensile properties in strain rate sensitive material.

Two different approaches have been taken in obtaining the hardness data to use in these relations: an instrumented hardness approach and a ball-microhardness approach. As described in detail elsewhere [7], in the instrumented hardness approach, the spherical indenter is attached to the load cell in the cross head of an Instron mechanical testing machine and driven into a test sample held on a fixed stage. The displacement of the indenter relative to the specimen is measured with an attached LVDT. After achieving the desired load the indenter is withdrawn, and from the load-displacement curve h as a function of W can be determined. This procedure can be repeated in a cyclic fashion at increasing maximum loads to obtain a number of data at a single penetration location.

Fig. 2. Geometric parameters in ball indents.

This technique has been used on a variety of materials, including samples of 6061-T6 aluminum, SAE 1015 steel, yellow brass, AISI 304 stainless steel and A302B steel. Hardness tests were run at ambient temperatures with indenter diameters ranging from 1.59 mm to 12.7 mm. Uniaxial stress-strain curves were also generated for each of these materials, and the uniaxial data were compared with the hardness data. Representative results are shown in Figure 3. As can be seen, the hardness derived stress-strain data agree well with the tensile stress-strain curve.

Based on the good agreement between the hardness and tensile data, some attempt was subsequently made to scale the instrumented hardness test down to smaller sample sizes and indentation sizes, i.e., to a microhardness test regime. For the displacements encountered in the microhardness regime, however, the parameter h_p became too small to measure conveniently. Consequently, a Tukon microhardness tester was modified in such a way that spherical indenters of different diameters could be used to indent the test samples. These ball microhardness tests have been conducted by indenting the sample at a small load (e.g. 100 gms), measuring d of the resulting indentation with a filar eyepiece, and repeating the process at increasingly higher loads; typically a sequence of 100, 200, 400, 600, 800 and 1000 gms has been used. Hence, 6 sets of (W, d) pairs are obtained for a given penetration location, and these data can be converted to stress-strain data with Eqn. (4).

Fig. 3. Comparison of hardness-derived stress-strain data with uniaxial tensile data for 6061-T6 Al.

The indentations achievable with such a procedure and with small, TEM disc type samples are limited in size. The general constraints which have been found for obtaining valid data are penetration depths less than 1/10 the sample thickness, chordal diameters less than 1/2 the specimen thickness, and indentations which intersect at least 5 grains at the surface. Outside of these constraints the relations in Eqn. (4) may not accurately predict the tensile stress-strain relation.

Subject to these constraints, the best approach used to date has been to indent samples such as TEM discs with two different ball indenters, one .25 mm in diameter and one 1.59 mm in diameter. The use of two ball sizes extends the range of plastic strain for which hardness-derived stress-strain data can be determined. An example comparison of microhardness data with tensile data for an SAE 1015 steel is shown in Fig. 4.

The microhardness data fit the strain hardening portion of the tensile curve in Fig. 4 well. This has been a general observation. In addition, one other feature in Fig. 4 is worth noting. The tensile curve featured a Lüders plateau, whereas the microhardness data did not necessarily indicate such. Since Lüders strains may be an important flow parameter to monitor in irradiated material, some effort has been devoted to characterizing the magnitude of the Lüders strain exhibited by mild steels from geometric features of the indentation.

Fig. 4. Comparison of microhardness-derived stress-strain data with uniaxial tensile data of a 1015 steel aged 20 min at 204°C.

A set of 47 tensile samples of SAE 1015 steel were heat treated at different temperatures to exhibit different Lüders strains when tensile tested under the same conditions (Lüders strain, ϵ_L , ranging from 0.48% to 5.4%). Following tensile testing, the undeformed end tabs of the tensile specimens were subjected to ball microhardness tests, and the lip geometry of selected indentations was characterized by profilometric and optical interferometric techniques.

Of the various geometric features investigated, it was found that the volume of material piled up in the indentation lip was one of the best indicators of the magnitude of Lüders strain in the test material, and this was most easily determined with interferometric techniques. Three optical interferographs and the corresponding profilometric traces are presented in Fig. 5. These are representative of the range of indentation geometries observed for materials exhibiting different Lüders strains. These particular indentations were produced with a 1.59 mm diameter ball loaded at 9.07 kg.

Fig. 5. Profilometer traces and interferographs of indentations made at 9.07 kg on a 1015 steel with a $\epsilon_L = 0.88\%$, b) $\epsilon_L = 2.76\%$, c) $\epsilon_L = 4.86\%$.

The horizontal axis corresponds to the rolling direction of the sheet material along which the profilometer traces were obtained. Optical interferometry was performed with a xenon light source using a highly monochromatic cadmium filter. Each interference fringe corresponds to a 322 nm displacement out of the plane of the paper. For increasing Lüders strain exhibited by the material, the number of interference fringes around the corresponding indent increases. The increased number of interference fringes means an increase in the volume of material piled up around these indents. The relationship between this volume and ϵ_L was found to be nearly linear.

It should also be noted that the edge of the lip around each indent showed a crown-like appearance. That is, nested interference fringes appear at the indentation edge. This is apparently a feature of steels which exhibit Lüders bands [8]. Because such features may be useful indicators of other localized flow mechanisms which may arise in response to radiation damage, this phenomenon is currently under further investigation.

5. CONCLUSIONS

Microhardness tests appear to be an attractive technique for extracting mechanical properties from the small specimens being used in high energy neutron irradiation experiments. Standard DPH microhardness tests have already been used to indicate relative hardening in pure and simple ternary materials. Moreover, semi-empirical correlations between ΔDPH and $\Delta\sigma$ appear to be useful in tracking strength changes in copper. Such correlations are consequently being applied to other materials irradiated in RTNS-II.

The ball microhardness test also appears to be an attractive technique for extracting mechanical properties from small specimens. This test approach seems to have an advantage over other types of microhardness tests like the Knoop or DPH in that the stress-strain relationship rather than a single point on the stress-strain curve can be determined from the data. Moreover, characterization of the lip geometry around a spherical indentation promises to provide additional flow information, particularly with respect to localized plasticity phenomena.

Consequently, efforts are currently in progress to modify a Tukon microhardness tester to integrate the microhardness test with interferometric techniques and computerized data processing. Moreover, sample preparation techniques are being developed to permit rapid preparation and handling of TEM disc specimens for ball microhardness testing. This should provide a significant advancement in specimen testing capabilities for the fusion materials effort.

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