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IRRADIATED MATERIALS

M. L. Hamilton
F. A. Garner
A. Okada

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Pacific Northwest Laboratory
Richland, Washington 99352

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MICRO-BULGE TESTING APPLIED TO NEUTRON IRRADIATED MATERIALS - A. Okada (Hokkaido University),
M. L. Hamilton (Pacific Northwest Laboratory)^(a) and F. A. Garner (Pacific Northwest Laboratory)

OBJECTIVE

This work was performed in an effort to extend the applicability of micro-bulge tests to standard electron microscopy disks.

SUMMARY

Micro-bulge testing was conducted on several Fe-Ni-Cr alloys irradiated as 0.3 mm thick disks to 10 dpa at 603 and 773 K in the Oak Ridge Research Reactor. Miniature tensile tests were performed on specimens of the same alloys irradiated concurrently. Good correlation between the tensile yield strength and the bulge yield load was observed in unirradiated specimens, however, the correlation was not simple for irradiated specimens. Good correlation was also observed between the ultimate tensile strength and the maximum bulge load. While irradiation produced a significant reduction in total elongation in the tensile test, irradiation caused only a small decrease in the deflection corresponding to the maximum bulge load compared to that observed on thinner disks used in earlier experiments. The results suggest that the thinner disk is better suited for ductility evaluations than the thicker disk. The area bounded by the load-deflection traces of the bulge tests shows a systematic variation with both alloy composition and irradiation condition which is not observed in the tensile data. It is anticipated that this parameter may prove useful in the evaluation of material toughness.

PROGRESS AND STATUS

Introduction

Among the various types of miniaturized mechanical testing developed for reactor materials research,¹ test techniques using transmission electron microscopy (TEM) disks 3 mm in diameter have a great advantage in reducing the radioactivity of the irradiated materials and in the occupation of a smaller fraction of the limited irradiation volume available. TEM disks have been utilized in the micro-bulge,²⁻⁴ bend,⁵⁻⁷ shear punch⁸ and small punch⁹ tests. In the micro-bulge test, a spherical punch (usually a tiny steel ball) is pressed into a disk, generally ~0.1 mm in thickness, whose periphery is held by a pair of dies. The load for the bulge deformation is recorded against the deflection of the punch. One of the present authors published a correlation between micro-bulge and tensile data for several metals and alloys in both the irradiated and unirradiated conditions, as well as the dependence on die geometry.¹⁰ Very little data are available for materials irradiated to doses as high as 10 dpa or for specimens as thick as 0.3 mm. In the present work, micro-bulge tests were conducted on several Fe-base ternary austenitic alloys irradiated as 0.3 mm thick disks to 10 dpa. The validity of the micro-bulge test results is discussed vis-a-vis their correlation with the tensile data.

Experimental Procedures

TEM disks 0.3 mm in thickness and miniature tensile specimens of seven Fe-Ni-Cr ternary austenitic alloys were irradiated concurrently to 12-14 dpa at 603 and 773 K in the MFE-4 experiment in the Oak Ridge Research Reactor.¹¹ The compositions are shown in Table I. The miniature tensile specimens were 0.25 mm thick with a gauge length and width of 12.7 and 1.0 mm, respectively. A hardened steel ball 1 mm in diameter was employed for the tip of the punch. The diameter of the lower die hole was 1.7 mm (clearance between ball and die hole was 0.35 mm) to accommodate disks up to 0.3 mm in thickness, and its shoulder radius was 0.4 mm.

Figure 1 shows typical examples of micro-bulge test curves and the parameters extracted from them for the evaluation of irradiation hardening and ductility loss. The parameters are determined as follows: a) bulge yield load (L_t : the load at $t/10$ offset from the tangent of the initial slope of the curve, where t is the disk thickness), b) maximum bulge load (L_{max}), c) bulge depth (D_{max}) and d) bulge energy (E_b : the area bounded by the load-deflection curve). These parameters were normalized⁹ to account for the variation in disk thickness, ϵ , by dividing by t^2 , dt , d and d^2t/d^2t , respectively, where d is the ball diameter and t_0 is 0.3 mm, the nominal disk thickness. Normalizing parameters were chosen on the basis of earlier micro-bulge tests on other materials. The bulge energy is normalized to the equivalent value expected for disks 0.3 mm thick.

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Results and Discussion

The normalized results are given in Table 2 and summarized below according to various parameters of interest. Note that the normalized yield load is larger than the normalized maximum bulge load due to the choice of normalizing parameter (t^2 versus dt). The tensile data on the same materials were reported in Reference 11.

Comparison of Bulge and Tensile Tests. The tensile test traces for the same materials as in Figure 1 are shown in Figure 2.¹¹ The irradiated specimens show a very large ductility loss in the tensile curve while the bulge test curves still show a considerable amount of deflection. The ball displacement for both the maximum bulge load and the load drop at the point of disk fracture are not too different for the unirradiated and irradiated specimens. The difference between the irradiated and unirradiated conditions in the tensile and bulge tests is due to the difference in the deformation mode, viz., the uniaxial stress condition in the tensile test and the essentially biaxial condition in the bulge deformation. This demonstrates the potential for using the bulge test technique for material property evaluations by virtue of the larger deformations that are unobtainable in a uniaxial tensile test due to the necking that results from the larger instability of deformation.

Yield Load. The bulge yield load and yield strength are shown in Figure 3 as a function of the alloy composition. In the unirradiated specimens, both sets of data show a similar dependence on composition, while the irradiated specimens show an opposite dependence on nickel level at 15 Cr. The origin of this result may be described as follows. The shoulder of the bulge curve corresponds to a change in the deformation mode from indentation to plastic bending. This transition may be controlled by both the hardness and the rigidity of the specimen, particularly in the case of irradiation hardened, relatively thick disks such as were used in this experiment. If the hardness of the disk exceeds a certain level, the transition to plastic bending will take place earlier than in a less hardened material; therefore, the load L_y at the shoulder of the curve may be smaller. To avoid unnecessary complexity in analyzing test data, disks thin enough to avoid the situation discussed above may be better suited for the bulge test.

Maximum Bulge Load. A straight line through the origin can be drawn as shown in Figure 4 for the correlation between the maximum bulge load and the ultimate tensile strength regardless of composition. The slope of this line is different than that drawn for 0.1 mm thick disks. In the case of thinner disks, as thin as 0.1 mm, the correlations for the fully annealed and the fully hardened materials were separated into two groups independent of material type and different correlation lines could be drawn for each of them.⁴ The separation between the lines for annealed and cold worked materials will decrease with thickness. The ductility loss and the hardening induced by irradiation are clearly observed in such a plot. The less distinct results obtained in the present experiment on thicker disks suggest that the thinner disks are better suited for the evaluation of strength and ductility using the micro-bulge test.

The maximum bulge load depends on the bulge depth, corresponding to the load at the necking of the bulged disk. If the material is soft, the punch presses deeper into the disk and conversely presses less deeply if the material is hard, so the radius of the contacting circle between the specimen and the punch is larger in the former case and smaller in the latter. Therefore, ductility loss and an increase in material hardness tend to occur concurrently.

Bulge Depth. Irradiation caused only a small decrease in the deflection corresponding to the maximum bulge load (Figure 5), especially when compared to the decrease observed in thinner disks.⁴ The bulge depth of the specimens irradiated at 773 K was almost the same as that of specimens irradiated at 603 K. The uniform elongation of the irradiated specimens, however, was very small. It was almost zero for the 603 K irradiation, suggesting that localized deformation may control the tensile test. The tensile test may therefore more correctly provide only fracture-related information rather than true ductility data in the embrittled materials. The bulge test may provide truer ductility data on these specimens, although the test does not seem to be sensitive enough for the specimen thickness considered. The results suggest that the materials retained considerable ductility for biaxial deformation even after irradiation at 603 K.

Relationship Between Maximum Bulge Load and Bulge Depth. As mentioned above, the bulge depth must be taken into consideration for evaluating the maximum bulge load, because the load is a function of bulge deflection. The maximum bulge loads are plotted against the bulge depth in Figure 6 for the solution annealed specimens. Ductility loss and irradiation hardening are easily observed in the figure as an overall shift to the left and upward, respectively. Unirradiated cold worked specimens exhibit lower bulge depths and higher maximum bulge loads. After irradiation, they exhibit less hardening than the solution annealed specimens with no distinct change in the bulge depth.

Bulge Fracture Energy. The area bounded by the load-deflection curve gives the work done throughout the bulge deformation process and is defined as the bulge fracture energy. The bulge energy is plotted against alloy composition in Figure 7. A clear variation is evident in the bulge fracture energy as a function of nickel level, decreasing as Ni increased from 20 to 35% and increasing again at 45 Ni. Such a variation was not present in the tensile data. This should reflect the microstructural evolution of the alloy, and is

considered to correspond to the observation by Sekimura¹² that a larger variation in microstructure was seen at 45 Ni in this alloy system. The bulge energy may therefore be a useful parameter for mechanical property evaluation, regardless of disk thickness.

Scatter. The scatter in the present experiment was a little larger than in similar experiments performed on the thinner 0.1 mm disks, but it is still only on the order of 10%. Some of the scatter may be due to the difficulty of holding the thicker disk firmly against the die as well as the fact that the deformation mode was not constant.

CONCLUSIONS

It appears that the bulge test data may be used in two ways in the present experiment for the evaluation of mechanical properties. The tensile properties can be estimated from the bulge data by correlation of the bulge yield load, maximum bulge load and bulge depth with the yield strength, tensile strength and uniform elongation, respectively. Alternatively, the bulge fracture energy may be of potential use in the absence of tensile data.

FUTURE WORK

Micro-bulge tests will be performed on ferritic alloys irradiated in FFTF/MOTA, JOYO and JMTR for comparisons between different neutron spectra and irradiation temperatures.

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Table 1

Alloys Used in the MFE-4 Experiment^(a)

Alloy	Composition, wt%	TEM Disk	Tensile
E19	Fe-19.7Ni-14.7Cr	SA	--
E20	Fe-24.4Ni-14.9Cr	SA, CW	SA
E21	Fe-30.1Ni-15.1Cr	SA	--
E22	Fe-34.5Ni-15.1Cr	SA, CW	SA
E23	Fe-45.3Ni-15.0Cr	SA	SA
E37	Fe-35.5Ni-7.5Cr	SA	--
E38	Fe-35.1Ni-21.7Cr	SA	SA

(a) SA - solution annealed.
CW - 30% cold worked.

Table 2

Normalized Micro-Bulge Data on Fe-Ni-Cr Alloys

Alloy	Yield load/t ² (MPa)			Max. Bulge Load/dt (MPa)			Bulge Depth/d (unitless)			Average Normalized Bulge Fracture Energy (Nm)		
	Unirr.	603 K	773 K	Unirr.	603 K	773 K	Unirr.	603 K	773 K	Unirr.	603 K	773 K
E19-SA	368	1561	1356	1209	1423	903	0.952	0.775	0.419	0.228	0.281	0.065
	398	1398	1334	1095	1346	840	0.927	0.787	0.356			
E20-SA	345	1374	1286	1113	1470	1337	0.927	0.838	0.787	0.216	0.254	0.239
	315			1095			0.902					
E20-CW	1471	1601	1717	1430	1540	1383	0.762	0.762	0.813	0.256	0.261	0.244
	1430			1425			0.737					
E21-SA	420	1357	1334	1065	1319	1334	0.876	0.762	0.787	0.202	0.274	0.261
	445	1151		1187	1394		0.902	0.775				
E22-SA	420	1587	1470	1157	1329	1326	0.889	0.749	0.775	0.191	0.268	0.261
	435		1536	1084		1308	0.851		0.762			
E22-CW	1366	1465	1519	1278	1377	1360	0.724	0.762	0.801	0.259	0.265	0.266
	1639	1480		1366	1400		0.724	0.762				
E23-SA	524	1774	1465	1189	1405	1350	0.876	0.762	0.762	0.205	0.272	0.283
	517			1237			0.876					
E37-SA	403	1420	1334	1034	1278	1148	0.864	0.787	0.762	0.173	0.358	0.233
	420	1315	1351	1067	1238	1167	0.876	0.775	0.749			
E38-SA	467	1420	1379	1223	1465	1406	0.889	0.775	0.778	0.222	0.284	0.311
	459			1189			0.851

Figure 1. Examples of the bulge test curves and the parameters determined from bulge testing. Dashed line denotes the elastic deformation of the fixture, L_y is the bulge yield load, L_{max} is the maximum bulge load, D_{max} is the bulge depth, E_b is the bulge fracture energy, and t is the disk thickness. Curves shown for 25Ni-15Cr.

Figure 2. Tensile test curves for the same materials whose bulge test curves are shown in Figure 1.

39006035.3

Figure 3. The bulge yield load (a) and yield strength (b) plotted against alloy composition. The bulge yield load is normalized by t^2 , where t is the disk thickness. The solid lines denote Ni variations for a constant Cr level (15%), and the dotted lines denote Cr variations for a constant Ni level (35%).

39006035.4

Figure 4. Maximum bulge load versus ultimate tensile strength. The maximum bulge load is normalized by the product dt , where d is the ball diameter and t is the specimen thickness.

Figure 5. The bulge depth (a) and uniform elongation (b) plotted against alloy composition. The bulge depth is normalized by the ball diameter, d . The solid lines denote Ni variations for a constant Cr level (15%), and the dotted lines denote Cr variations for a constant Ni level (35%).

Figure 6. Shift in the maximum bulge load and in the bulge depth induced by irradiation in solution-annealed specimens.

Figure 7. Bulge fracture energy as a function of alloy composition. The solid lines denote Ni variations for a constant Cr level (15%), and the dotted lines denote Cr variations for a constant Ni level (35%).

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