CORRELATION OF YIELD STRESS AND MICROHARDNESS IN 08CR16NI11MO3 IRRADIATED TO HIGH DOSE IN THE BN-350 FAST REACTOR–O. P. Maksimkin, M. N. Gusev, O. V. Tivanova, N. S. Silnaygina (Institute of Nuclear Physics, National Nuclear Center, Almaty, Kazakhstan), and F. A. Garner (Pacific Northwest National Laboratory)*

OBJECTIVE

The objective of this effort is to develop methods of extracting data on radiation-induced changes in mechanical properties when the material of interest is highly irradiated, in an inconvenient location or configuration, or when significant gradients in mechanical properties are anticipated over small dimensions.

SUMMARY

The relationship between values of the microhardness H_μ and the engineering yield stress, σ_{02}, in steel 08Cr16Ni11Mo3 (Russian analog of AISI 316) heavily irradiated in the BN-350 reactor has been experimentally derived. It agrees very well with the previously published correlation developed by Toloczko for unirradiated 316 in a variety of cold-work conditions. Even more importantly, when the correlation is derived in the K_Δ format where the correlation involves changes in the two properties, we find excellent agreement with a universal K_Δ correlation developed by Busby and coworkers.

With this K_Δ correlation, one can predict the value of yield stress in irradiated material based on measured values of microhardness. The technique is particularly suitable when the material of interest is in an inconvenient location or configuration, or when significant gradients in mechanical properties are anticipated over small dimensions. This approach makes it possible to reduce the labor input and risk when conducting such work. It appears that the derived correlation is equally applicable to both Russian and Western austenitic steel, and also in both irradiated and unirradiated conditions.

Additionally, this report points out that microhardness measurements must take into account that high temperature sodium exposure alters the metal surface to produce ferrite, and therefore the altered layers should be removed prior to testing.

PROGRESS AND STATUS

Introduction

The yield strength $\sigma_{0.2}$ is a basic parameter used in engineering calculations and its proper determination is an important task for design of fission or fusion reactors. For fusion applications, however, data generated in the appropriate spectra are not available, so data from surrogate spectra are required to validate the measurement technique and to allow extrapolation to the target spectra.

Even in surrogate spectra it is not always possible, however, to determine $\sigma_{0.2}$ on highly irradiated material using direct techniques such as uniaxial tensile tests, particularly when there are large levels of induced radioactivity. It can also be difficult when the material volume is either too small to produce a tensile or other mechanical property specimen, when the material of interest is in an inconvenient location or configuration, or when significant gradients in mechanical properties are anticipated over small dimensions. The latter might arise where there are strong local gradients in temperature or neutron flux across the material of interest.

One approach to surmount such difficulties is to establish property-property correlations using appropriate correlation relations with other quantities, such as the critical transverse strain measured using the shear punch test [1] or the microhardness H_μ [2]. While both of these techniques are useful for measurement using small specimens, the latter is particularly suited to measurements made in very small dimensions with potentially steep environmental gradients. Both of these techniques are very convenient for working with irradiated material.

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A review summary of microhardness-tensile correlations has recently been published by Busby, Hash, and Was [3]. They demonstrated that two types of correlation have been published based on either the direct measurements ($H_\mu \sim k_1 \cdot \sigma_{02} + \sigma_1$) or the change in measurements $H_\mu \sim k_\Delta \cdot \sigma_{02}$. As noted by Busby et al., the successful use of the first correlation requires that both properties be measured at the same temperature.

It is important to note that σ_{02} is a bulk-averaged property while H_μ reflects primarily the surface properties, so care should be taken to ensure that no significant surface modification has occurred in the surrogate environment. If the potential for such modification is present, then the surface layers should be removed.

In the present work, the quantities $\sigma_{0.2}$ and H_μ are measured and compared for Russian austenitic stainless steel designated 08Cr16Ni11Mo3 (Russian analog of AISI 316, chemical composition – Cr: 16%, Ni: 11.4%, Mn: 1.6%, Mo: 1.8%) which was irradiated in flowing sodium to doses as large as 15.6 dpa in the BN-350 fast reactor in Aktau, Kazakhstan. A limited range of specimen temperatures was chosen to minimize the influence of variables other than dpa level.

The objective of this effort is to provide yield strength predictions for Russian steels for fusion reactor conditions employing fast reactor data as surrogates. A secondary objective is to show that Russian and Western steels all respond in a similar manner to radiation exposure.

Experimental Procedure

Specimens with different damage doses and irradiation temperatures (see Table 1) were cut from the protective hexagonal shroud of spent assembles H-214(II), H-110, B-337, and B-300, with all assemblies located at different distances from the reactor core center line.

Two types of specimens were selected (see Fig. 1). The first type was sliced from the central part of the wrapper faces. The second type was sliced from the corners of the hexagonal assembly. The use of the two types of specimens reflects the fact that the technique used for manufacturing the wrappers may have induced structural differences between the faces and corners of the wrapper.

Both types of specimens were removed by slicing, followed by mechanical and electrolytic polishing of the specimens to remove the influence of the specimen surface, which had been in long contact with sodium at elevated temperatures and therefore likely to be unrepresentative of bulk composition and properties [4-7]. In general, such exposure leads to the removal of nickel, chromium, and other elements near the surface, often producing a ferrite layer on the surface as a consequence.

Table 1. Irradiation conditions

Distance from reactor core, mm	Damage dose, dpa	Dose rate, dpa/s	Irradiation temperature, °C
-1200 (H-214)	0.25	$8 \cdot 10^{-10}$	280
-900 (H-214)	1.27	$4 \cdot 10^{-9}$	281
+500 (H-214)	6.03	$2.2 \cdot 10^{-8}$	365
-500 (B-300)	11	$4.9 \cdot 10^{-8}$	302
-500 (B-337)	12	$2.6 \cdot 10^{-7}$	309
0 (H-110)	13	$4.2 \cdot 10^{-7}$	311
0 (H-214)	15.6	$4.8 \cdot 10^{-7}$	337

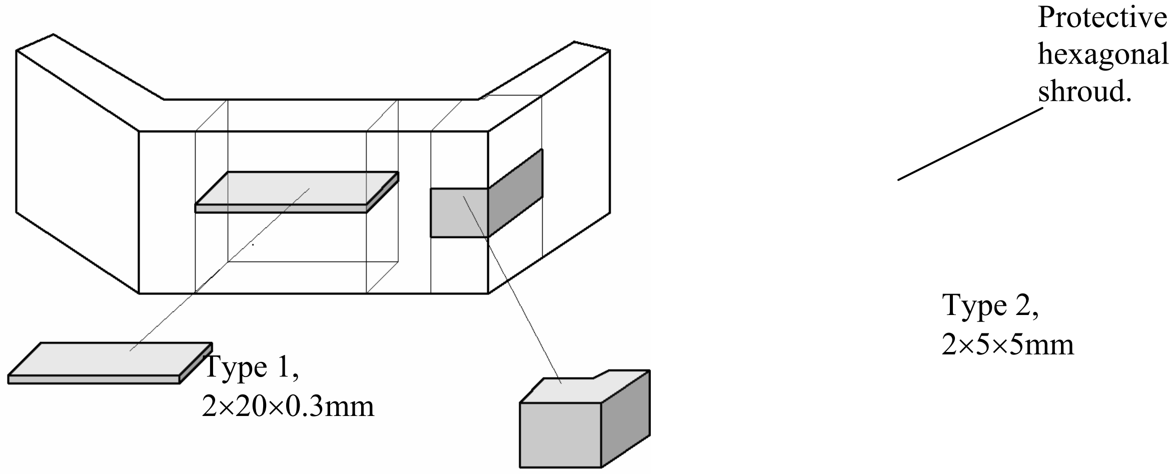

Fig. 1. Specimen slicing scheme.

Flat tensile specimens of Type 1 with dimensions 10 × 2 × 0.3 mm (see Fig. 1) were also subject to mechanical grinding and electrical polishing, in order to achieve the desired thickness and surface quality. Pneumatic grips were used for holding of the specimen.

Tensile tests of both unirradiated and irradiated Type 1 face specimens were performed with an Instron-1195 test machine at 20°C with a strain rate of 8.3 x 10^{-4} s^{-1}. The Vickers microhardness was determined on both specimen types using the PMT-3 device, employing a diamond pyramid with vertex angle of 136°C. The load on the indenter was 100 g. The microhardness of each sample was measured 30–40 times. Both types of tests were conducted at room temperature. The estimated inaccuracies in determination of $\sigma_{0.2}$ are ≤ 5%, and for the microhardness were estimated to be 3 to 4%.

Results

Results of mechanical tests of irradiated specimens of steel 08Cr16Ni11Mo3 are presented in Table 2. One can observe that the values of $\sigma_{0.2}$ and H_μ initially increase as the damage dose increases. The values of $\sigma_{0.2}$ and H_μ peak at ~ 12 and ~ 11 dpa, respectively, falling with increasing dose, but most likely reflecting the stronger influence of increasing temperature rather than reflecting a late-term softening with dose.

Table 2. Microhardness and yield stress of 08Cr16Ni11Mo3 irradiated in the BN-350 reactor

Dose, dpa	microhardness $H\mu$, kg/mm^2			Yield stress $\sigma_{0.2}$, MPa
	Face	Corner	Mean value	
unirradiated	--	--	150	230
0.25	260	260	260	550
1.27	297	293	295	670
6.03	350	303	326	860
11	407	435	421	980
12	404	412	407	1010
13	391	407	398	904
15.6	370	380	375	920

Small differences in microhardness between the face and the corner of specimens at the same nominal dose level were observed (see Table 2). Up to ~ 6 dpa, the microhardness of the face is slightly higher than the microhardness of the corner. Above ~ 6 dpa, the corner has higher microhardness. There is the possibility that the faces and corners have slightly different dpa levels and temperatures, however, which might account for the observed small variations. A decision was therefore made to average the values for comparison with the tensile data.

The correlation between σ_{02} and H_μ for the current steel can be described by the following relation:

$$H_\mu \sim k_1 \cdot \sigma_{02} + \sigma_1 \quad (1)$$

where k_1 =2.85 and σ_1 = -177, with units of MPa and kg/mm^2, respectively.

The correlation between changes of σ_{02} and H_μ for the current steel (see Fig. 2) can be described by the following relation:

$$\Delta H_\mu \sim k_\Delta \cdot \Delta\sigma_{02} \quad (2)$$

where k_Δ =2.96.

The utility of the microhardness measurement is best illustrated when there are no tensile data for comparison. In addition to the specimens listed in Table 1, another fragment of a similar wrapper was available (10 dpa at 365°C), but attempts to measure its yield strength have not been successful as a result of unexpectedly high brittleness. These specimens broke during tensile testing, either in the course of putting them in the pneumatic grippers or at the very beginning of straining. Our derived K_Δ correlation was used to estimate the yield strength of this material after successful measurement by microhardness. At the same time, some specially prepared specimens (diameter 3 mm and thickness 0.3 mm) were used to perform shear–punch tests on the same material [1,8,9]. As one can see from Table 3, the yield strength values predicted from the microhardness measurements and the shear-punch tests are rather close at 670 ± 30 MPa.

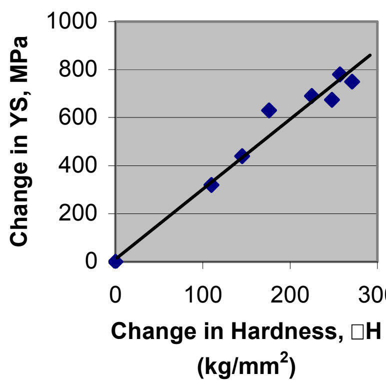

Fig. 2. Correlation between changes of microhardness and stress yield of steel 08Cr16Ni11Mo3 irradiated in the BN-350 reactor.

Table 3. Yield strength predictions for 08Cr16Ni11Mo3 steel in brittle condition obtained by the use of two different property-property correlation techniques

Correlation technique used	Predicted value $\sigma_{0.2}$, MPa
Microhardness measurement (H_μ = 296 kg/mm^2, ΔH_μ = 146 kg/mm^2)	700
Shear-Punch test [1,8,9]	640

Discussion

The derived relationship, H_μ =2.85 σ_{02} -177, is very similar to the one derived by Toloczko et al. [8] for unirradiated 316 cold-worked to various levels, where H_μ =2.7 $\sigma_{0.2}$ -125. As reviewed by Busby et al. other correlations of this form were developed for both unirradiated and irradiated stainless steels but suffer from the complication that the tensile tests were conducted at elevated temperatures (~ 300°C) while the hardness values were conducted at room temperature [10-11]. Busby shows that such mixed data correlations require the K_Δ equation form where the relative changes in property have been shown to be temperature-independent, with K_Δ = 3.03 for all steels.

The change in property relationship derived in this study, ΔH_μ =2.96·$\Delta\sigma_{02}$, is very similar to the one derived by Busby. This study has the benefit that both types of tests were conducted at room temperature, and any sodium-modified surface layers were removed prior to microhardness testing.

Conclusions

The relationship between values of the microhardness H_μ and the engineering yield stress, σ_{02}, in heavily irradiated steel 08Cr16Ni11Mo3 (analog of AISI 316) has been experimentally derived.

Our results appear to agree very well with the correlation developed for unirradiated 316 in a variety of cold-work conditions. Even more importantly, when the correlation is derived in the K_Δ format, we find excellent agreement with the universal correlation developed by Busby and coworkers. With this universal correlation, one can predict the value of yield stress in irradiated material based on measured values of microhardness. This approach makes it possible to reduce the labor input and risk when making such work.

It appears that the derived correlation is equally applicable to both Russian and Western austenitic steel, and also in both irradiated and unirradiated conditions.

References

[1] M. B. Toloczko, K. Abe, M. L. Hamilton, F. A. Garner, and R. J. Kurtz, The Effect of Test Machine Compliance on the Measured Shear Punch Yield Stress as Predicted Using Finite Element Analysis, Small Specimen Test Techniques: Fourth Volume, ASTM STP 1418 (2002) 339–349.
[2] J. R. Cahoon, W. H. Broughton, and A. R. Kutzak, The determination of yield strength from hardness measurements, Metall. Trans., Vol. 2 (1971) 1979–1983.
[3] J. T. Busby, M. C. Hash, and G. S. Was, The relationship between hardness and yield stress in irradiated austenitic and ferritic steels, J. Nucl. Mater. 336 (2005) 267–278.
[4] W. F. Brehm, Interaction of Sodium with Breeder Reactor Materials, in R. P. Agarwala (ed.), Diffusion Processes in Nuclear Materials (Elsevier Science Publishers B. V., 1992) 323–352.
[5] P. T. Nettley, I. P. Bell, K. Q. Bagely, D. R. Harries, A. W. Thorley, and C. Tyzack, Problems in the Selection and Utilization of Materials in Sodium Cooled Fast Reactors, BNES, Fast Breeder Reactors, Pergamon, Oxford (1967) 825–849.
[6] W. Charnock, C. P. Haigh, C. A. P. Horten, and P. Marshall, CEBG Research (November 1979) 3.
[7] I. I. Balachov, F. A. Garner, Y. Isobe, M. Sagisaka, and H. T. Tang, NDE Measurements of Irradiation-induced Void Swelling, Eleventh International Conference on Environmental Degradation of Materials in Nuclear Systems – Water Reactors (2003) 640–645.
[8] M. B. Toloczko, G. E. Lucas, G. R. Odette, R. E. Stoller, and M. L. Hamilton, An Investigation of Microstructures and Yield Strengths in Irradiated Austenitic Stainless Steels Using Small Specimen Techniques, 17th International Symposium on Effects of Radiation on Materials, ASTM STP 1270, (1996) 902.
[9] O. P. Maksimkin, L. G. Turubarova, N. V. Sherbinina, P. V. Chakrov, F. A. Garner, and A. B. Johnson, Microstructural and Mechanical Studies of the Stainless/Aluminum Alloy Control Rod of the WWR-K Research Reactor, Tenth International Conference on Environmental Degradation of Materials in Nuclear Systems – Water Reactors (2001) CD format.
[10] H. R. Higgy and F. H. Hammad, Effect of fast neutron irradiation on mechanical properties of stainless steels: AISI types 304, 316, and 347, J. Nucl. Mater. 55 (1975) 177–186.

[11] M. Kodama, S. Suzuki, K. Nakata, S. Nishimura, K. Fukuya, T. Kato, Y. Tanaka, and S. Shima, Mechanical Properties of Various Kinds of Irradiated Austenitic Stainless Steels, 8th International Symposium on Environmental Degradation-Water Reactors (August 1997) 833.