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Correlation of yield stress and microhardness in 08Cr16Ni11Mo3 stainless steel irradiated to high dose in the BN-350 fast reactor

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Abstract

The relationship between the microhardness and the engineering yield stress in 08Cr16Ni11Mo3 steel after irradiation in the BN-350 reactor has been experimentally derived and agrees with a 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, excellent agreement is found with a universal K_Δ correlation developed by Busby and coworkers. Additionally, this report points out that microhardness measurements must take into account that sodium exposure at high temperature and neutron fluence alters the metal surface to produce ferrite, and therefore the altered layers should be removed prior to testing.

1. Introduction

The yield stress $\sigma_{0.2}$ 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 specimen 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.

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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.

A review summary of microhardness–tensile correlations has recently been published by Busby, Hash and Was covering three austenitic steels (AISI 304, 316, 347) irradiated in both fast and thermal reactors [3]. They demonstrated that two types of correlation have been published based on either the direct measurements ($\sigma_{0.2} \sim k_1 H_\mu + \sigma_1$) or the change in measurements $\Delta\sigma_{0.2} \sim K_\Delta \cdot \Delta H_\mu$. 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 $\sigma_{0.2}$ is a bulk-averaged property while H_μ reflects primarily the near-surface properties, especially at low indenter loads, so care should be taken to insure that no significant surface or near-surface modification has occurred in the surrogate environment. If the potential for such modification is present then the surface layers should be removed. As the indenter load increases the impact of surface modification is expected to decease.

In the present work the quantities $\sigma_{0.2}$ and H_μ are measured and compared for Russian austenitic stainless steel designated 08Cr16Ni11Mo3 (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 irradiation temperatures was chosen to minimize the influence of variables other than dpa level.

The objective of this effort is to provide yield stress 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 respond in a similar manner to radiation exposure.

2. Materials and experimental techniques

Specimens with different damage doses and irradiation temperatures (see Table 1) were cut from the protective hexagonal shroud of spent fuel assembles H-214(II), H-110, B-337 and B-300, with all assemblies located at different distances from the reactor core center line. The ducts have flat to flat size of 96 mm and are 2 mm thick.

Table 1
Irradiation conditions

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

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 [4–8]. In general such exposure, especially during extended neutron irradiation, leads to the removal of nickel, chromium and other elements near the surface, often producing a surface ferrite layer as a consequence. Frequently this ferrite layer is not detected during post-irradiation testing.

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

Tensile tests on two specimens of both unirradiated and irradiated type 1 face specimens were performed with an Instron-1195 test machine at 20 °C at a strain rate of $8.3 \times 10^{-4}\ \mathrm{s}^{-1}$. 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

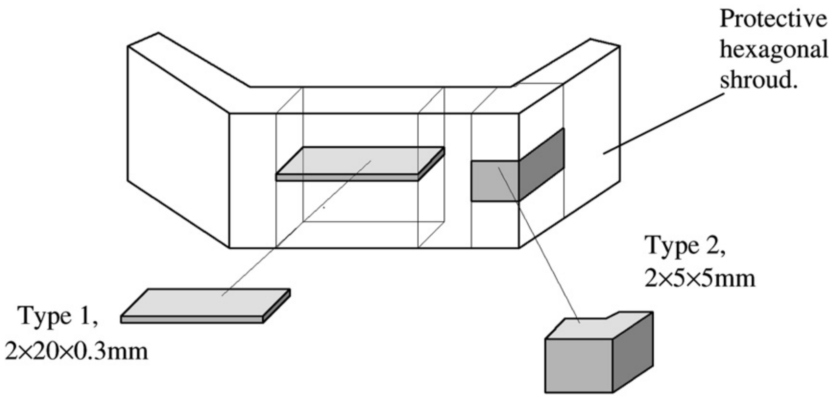

Fig. 1. Specimen slicing scheme.

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 $\leqslant 5\%$, and for the microhardness were estimated to be 3–4%.

3. 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.

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 of the face and the corner for comparison with the tensile data.

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

Dose (dpa)	Microhardness H_μ (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

The correlation between $\sigma_{0.2}$ and H_μ for the current steel can be described by the following relation:

$$\sigma_{0.2}\ [\mathrm{MPa}] \sim k_1 H_\mu\ [\mathrm{kg/mm^2}] + \sigma_1\ [\mathrm{MPa}], \tag{1}$$

where $k_1 = 2.85$ and $\sigma_1 = -177$.

The correlation between changes of $\sigma_{0.2}$ and H_μ for the current steel (see Fig. 2) can be described by the following relation:

$$\Delta\sigma_{0.2} \sim k_\Delta \cdot \Delta H_\mu, \tag{2}$$

where $K_\Delta = 2.96$.

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 stress 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 of the yield stress 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 [1,9,10]. As one can see from

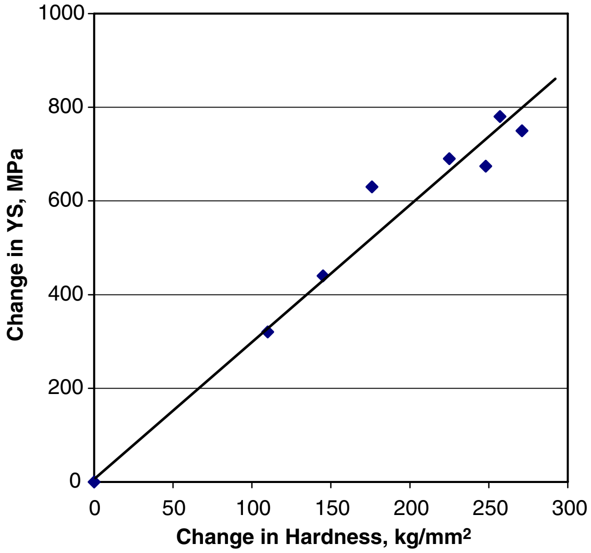

Fig. 2. Correlation between changes of microhardness and yield stress of steel 08Cr16Ni11Mo3 irradiated in the BN-350 reactor.

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 ($H_\mu = 296$ kg/mm^2, $\Delta H_\mu = 146$ kg/mm^2)	700
Shear–punch test [1,9,10]	640

Table 3, the yield strength values predicted from the microhardness measurements and the shear–punch tests are rather close at 670 ± 30 MPa.

4. Discussion

The derived relationship, $\sigma_{0.2} = 2.85 \cdot H_\mu - 177$, is very similar to the one derived by Toloczko et al. [9] for unirradiated 316 cold-worked to various levels, where $\sigma_{0.2} = 2.7 \cdot H_\mu - 125$. 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 often conducted at elevated temperatures (~300 °C) while the hardness values were usually conducted at room temperature [11,12]. Busby shows that such correlations require the change in property correlation form since the changes in property have been shown not to be temperature dependent, with $K_\Delta = 3.03$ for all steels.

The change in property relationship derived in this study, $\Delta\sigma_{0.2} = 2.96 \cdot \Delta H_\mu$, is very similar to the one derived by Busby. The cumulative data scatter in the various studies collected by Busby was rather large compared to the smaller data set with more limited scatter of the current study, but previous studies utilizing fast reactor irradiation did not consider the possibility of compositional change and surface ferrite layer formation. The current study has the benefit that both types of test were conducted at room temperature, a larger number of indents were made on a single specimen, and the removal of potentially sodium-modified surface layers prior to microhardness testing.

An additional consideration concerning the general applicability of the tensile–hardness correlation was recently identified by Neustroev and coworkers [13]. They noted that the tensile–hardness correlation breaks down in 12Cr18Ni10Ti stainless steel at high hardening levels when tested at room temperature. They attribute the breakdown to formation of deformation martensite during microhardness measurements of this low-nickel steel, a process that does not occur as strongly in deformation occurring before the tensile strength is reached. One of the current authors (Maksimkin) has previously shown [14] this steel to be very prone to martensite formation during plastic deformation, and irradiation can strongly increase intensity of this process for low nickel steels. In the higher nickel steel used in the current study, 08Cr16Ni11Mo3, this phenomenon does not appear to impact the correlation significantly.

5. Conclusions

The relationship between values of the microhardness H_μ and the engineering yield stress, $\sigma_{0.2}$, in heavily irradiated steel 08Cr16Ni11Mo3 (analog of AISI 316) has been experimentally derived. The derived correlation appears 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 radiation risk when conducting such work.

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

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