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Low-temperature mechanical properties of Fe–0.06C–18Cr–10Ni–0.4Ti austenitic steel determined using ring-pull tensile tests and microhardness measurements

V.S. Neustroev [a], E.V. Boev [a], F.A. Garner [b,*]

[a] *Research Institute of Atomic Reactors, Dimitrovgrad, Russia*
[b] *Pacific Northwest National Laboratory, P.O. Box 999, M/S P8-15, Richland, WA 99532, USA*

Abstract

Irradiated austenitic stainless steels removed from Russian water-cooled VVERs experience irradiation temperatures and He/dpa conditions that are very similar to steels to be used in ITER. Data are presented on the radiation hardening of the Russian analog of AISI 321 at 0.2–15 dpa in the range of 285–320 °C. The Russian variant of the ring-pull tensile test was used to obtain mechanical property data. Microhardness tests on the ring specimens provide useful information throughout the deformed regions, but at high hardening levels caution must be exercised before application of a widely accepted hardness-yield stress correlation to prediction of tensile properties. Low-nickel austenitic steels are very prone to form deformation martensite, a phase that increases strongly with the larger deformation levels characteristic of microhardness tests, especially when compared to the 0.2% deformation used to define yield stress.

1. Introduction

Light water-cooled power reactors and first generation fusion devices such as ITER share certain similarities with respect to the environmental conditions experienced by the major structural materials, which are austenitic stainless steels. These conditions are relatively low operating temperatures, water-cooling, relatively high helium/dpa ratios and relatively low dpa rates compared to those of typical fast reactors. In Russia and the USA attention is now directed toward low-temperature irradiation-hardening, low-temperature void swelling and void-induced embrittlement of PWR and VVER-1000 austenitic internals [1–4].

2. Experimental details

Test specimens were cut from thin-wall instrument guide tubes located in fuel assemblies of several VVER-1000 reactors after 3–4 years of irradiation to doses of 0.2–15 dpa in the range of 285–320 °C [5]. Dpa levels were calculated using the standard NRT formulism and temperatures were assumed to be that of the local water temperature, an assumption that is appropriate for thin-wall tubes with water on both sides. The temperature is known within ±2 °C.

* Corresponding author. Tel.: +1 509 376 4136; cell: +1 509 531 2112; fax: +1 509 376 0418.
E-mail address: Frank.garner@pnl.gov (F.A. Garner).

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The tubes were constructed from Fe–0.06C–18Cr–10Ni–0.4Ti steel (Russian analog of AISI 321) in the austenized state (1050 °C, 30 min, AC) and were 12.6 mm in diameter with wall thickness of 0.8 mm. The specimens were cut to produce rings of 3 mm length. The rings were deburred, cleaned and lightly polished prior to testing.

Note that the Russian variant of the ring-pull test involves no local reduction in the ring diameter to produce thin gauge sections as frequently employed in other studies [6–8]. This standardized test [9,10] is frequently employed in the states of the former Soviet Union and allows tests to be performed on irradiated tubes without expensive mechanical or electrical machining. Results of other studies using this test technique have recently been reported [11,12].

Mechanical properties on both irradiated and unirradiated specimens were determined via tensile tests and microhardness measurements conducted on the ring-shaped specimens. Tensile tests were conducted at temperatures between 20 and 800 °C, but microhardness measurements were conducted only at 20 °C. The tensile test proceeded with a deformation rate of ∼1 mm/min. Fig. 1 shows the geometry of this test configuration. The measurement error of mechanical properties via this test is estimated at ∼5%.

Microhardness was measured remotely using ring specimens mounted on a mandrel. A standard diamond pyramid indenter was forced into a specimen using 100 g load and the microhardness calculated from the size of the indentation. The measurement error of the microhardness is estimated at ∼2%.

3. Results

Fig. 2 presents the yield stress measured at 20 °C for the irradiation temperature range 285–320 °C as a function of dpa. The measured yield stress increase was approximated by a formula used by Kalinin and co-workers to process data for ITER application [13].

$$\Delta\sigma = A\sqrt{1 - e^{(-Kt/D_0)}}. \tag{1}$$

Factor A characterizes the saturation level of yield stress increase in MPa. The factor D_0 describes the duration of the transient regime and Kt is the damage dose, where the latter two parameters are both in dpa. In this data set D_0 at 20 °C is 4.7 dpa, independent of irradiation temperature over this limited range. The factor A reaches 600 MPa at 20 °C, 500 MPa at 300 °C and 240 MPa at 600 °C. D_0 was kept constant at 4.7 dpa for the higher test temperatures.

A similar dependence (Fig. 3) was obtained for microhardness. Factor A at 20 °C in this case is equal to 230 kg/mm^2 when factor D_0 was chosen to be 4.7 dpa to match that of the yield stress increase. The ratio between saturation levels of increases in yield stress and microhardness at damage doses of about 9–15 dpa was found to be 2.61.

The ring specimen has another positive feature from the viewpoint of microhardness examination. Since the specimen has two working ligaments, and rupture usually occurs only in one of them, there is another deformed but still intact ligament on the other side of the ring where the specimen progressively narrows toward the likely failure position. This 'narrowing' area allows microhardness change measurements in the longitudinal direction, i.e. at progressively changing deformation levels within the ligament.

In non-irradiated specimens, changes of microhardness with distance from the rupture are easily observed (Fig. 4). In this case a noticeable strain hardening is observed that is consistent with an increase of deformation-induced dislocation density. The local deformation was calculated by measuring the local thickness decrease of the non-irradiated specimen at a given distance from the thinnest section. In the irradiated specimen the strain hardening was much lower, as seen in the lack of variability in the hardness vs. distance curve, a behavior consistent with the hardening induced by high densities of small Frank loops during irradiation [14].

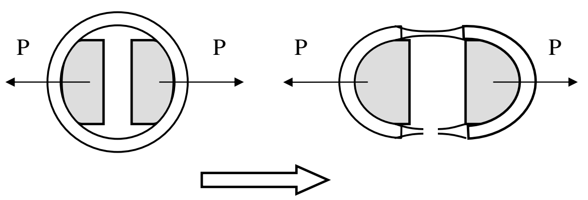

Fig. 1. Schematic diagram showing loading and testing of tube rings in a ring-pull test. Note that two regions on opposite sides of the tube are being deformed, but only one will fail and terminate the test. The non-failed ligament can be used to measure the approach to failure along the full length of the ligament. P indicates the force applied to separate the two mandrels inside the ring.

4. Discussion

Busby and his colleagues [15] analyzed and published an extensive correlation of published yield

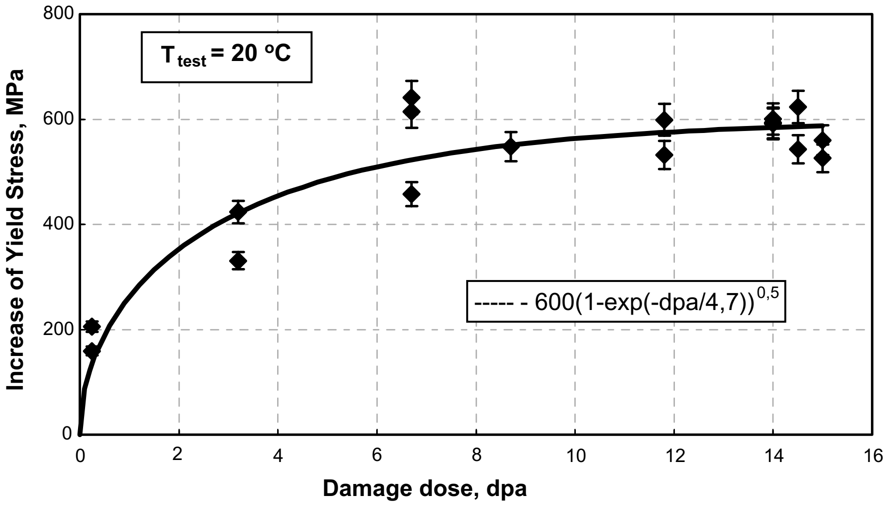

Fig. 2. Dose dependence of yield stress increase.

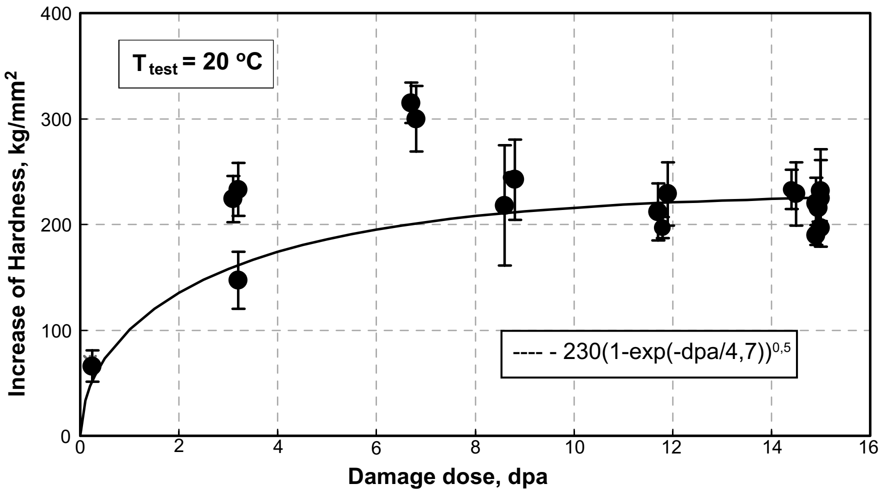

Fig. 3. Dose dependence of microhardness increase.

stress and microhardness data on austenitic steels (AISI 316, 347 and 304) irradiated in both fast reactors and thermal reactors. A ratio of $\Delta\sigma_{0.2}/\Delta H_{\mu} = 3.03$ was obtained when the changes in tensile and hardness properties, rather than their absolute values, were used. Busby noted that published data frequently involved complicating factors that might account for the scatter associated with these results. One of the most important sources of scatter was the tendency to conduct tensile tests at the irradiation temperature while the hardness tests were made at room temperature. Gusev and co-workers [16] recently conducted a similar study on Fe–0.06C–16Cr–11.4Ni–1.6Mn–1.8Mo steel irradiated in the BN-350 fast reactor, but noted that additional scatter might arise from specimens that were irradiated in flowing sodium. Such exposure changes both the composition of the near-surface region but also frequently produces an undetected ferrite layer on the surface.

Hardness measurements are more sensitive to such surface modifications than are tensile measurements which involve the full thickness of the specimen. In Gusev's study the surface layers were removed before testing and the correlation coefficient was found to be 2.96, essentially identical to the 3.03 of Busby.

In this paper, however, we observe for Fe–0.06C–18Cr–10Ni–0.4Ti a correlation coefficient of only 2.61 derived using all the data, but we cannot

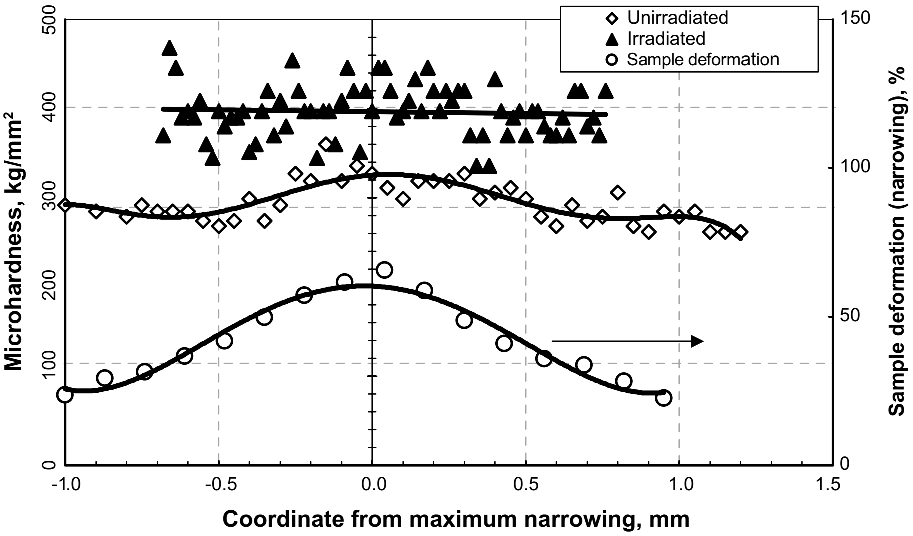

Fig. 4. Changes of microhardness and narrowing in unirradiated and irradiated (290 °C, 8.4 dpa) specimen of Fe–0.06C–18Cr–10Ni–0.4Ti austenitic stainless steel.

ascribe this lower value solely to surface modification. The compositional and phase changes observed during sodium exposure do not occur to any significant degree in the water coolant of PWRs and VVERs. Note that when we compare our data with that of Busby's compilation and also that of Gusev in Fig. 5, we see that our lower exposure data initially tend to follow a correlation coefficient of ∼3 but then plateau out at yield stresses of ∼600 MPa for hardness increases >200 kg/mm^2. This behavior was not observed by Gusev, however.

The difference between our results and those of Gusev lie primarily in the composition of the steels and not the radiation/coolant environment. The Fe–16Cr–11.4Ni–1.6Mn–1.8Mo steel of Gusev has higher nickel content than our steel, and is therefore, more resistant to formation of deformation martensite. Microhardness measurements involve much greater amounts of local deformation when compared to that associated with yielding.

In an earlier study Kadyrzhanov and Maksimkin clearly showed that in Fe–0.06C–18Cr–10Ni–0.4Ti

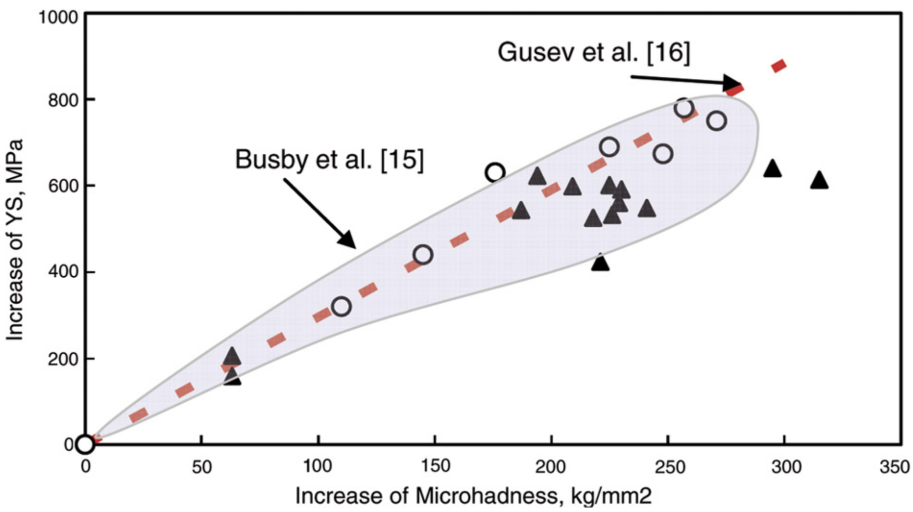

Fig. 5. Correlation between yield stress increase and microhardness increase of Fe–0.06C–18Cr–10Ni–0.4Ti austenitic stainless steel after low-temperature irradiation in VVER-1000 reactors. Also shown are the data band of Busby and co-workers [15] and the data (o) and trend line (- - -) of Gusev and co-workers on Fe–16Cr–11.4Ni–1.6Mn–1.8Mo irradiated in BN-350 [16].

steel that the microhardness behavior of this steel is strongly determined by martensite formation as compared with the behavior of yield stress and other characteristics of mechanical properties [17]. Martensite instability is enhanced by lower test temperatures and the strength and microchemical changes induced by irradiation, shifting the regime of martensite formation to higher temperatures. Experiments currently underway appear to confirm that martensite formation in our rings increases strongly with increasing deformation moving from the relatively undeformed to strongly deformed regions in the unbroken ligament.

5. Conclusions

Ring-pull tensile tests on tube sections can be used to measure radiation-induced hardening, and when coupled with microhardness measurements can be used to study and make predictions of anticipated mechanical behavior for both light water reactors and ITER.

The development of radiation-induced hardening in Fe–0.06C–18Cr–10Ni–0.4Ti austenitic steel irradiated in VVER-1000 reactors at $\sim$300 °C reaches a saturation level of 600 MPa at $\sim$5 dpa at $T_{test} = 20$ °C. The tensile hardening relationship can be described by

$$\Delta\sigma_{0,2} = A\sqrt{1 - e^{(-Kt/D_0)}},$$

where D_0 is 4.7 dpa, and the factor A [MPa] depends on test temperature.

A similar relationship can be used to describe the increase in microhardness with $D_0 = 4.7$ dpa and $A = 230$ kg/mm^2 for tests conducted at 20 °C.

At higher hardness levels reached during low-temperature irradiation the well-established correlation between changes in yield stress and microhardness breaks down for low-nickel steels when deformed at room temperature. The breakdown is suggested to arise from the onset of deformation martensite; a process that impacts microhardness measurements much more than it affects determination of yield stress.

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