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Tensile properties of the NLF reduced activation ferritic/martensitic steels after irradiation in a fast reactor spectrum to a maximum dose of 67 dpa

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Abstract

The NLF series of steels are reduced activation ferritic–martensitic (RAFM) steels that are a part of the Japanese program to produce a suitable reduced activation ferritic–martensitic steel for the ITER project. Published reports on the NLF steels after about 35 dpa at 400 °C by Kurishita et al., indicate that these steels have similar strength and better ductility than other RAFM steels such as the JLF steels and F82H irradiated at 400 °C to similar doses. The tensile properties of NLF steels irradiated at ~400 °C to doses as high as 67 dpa are presented here. Tensile tests were conducted at a strain rate of $5 \times 10^{-4}\ s^{-1}$ at 25, 400 °C, and 500 °C. Variations in irradiation temperature in the range of 390–430 °C had a relatively small, but definite effect on the tensile properties for tests conducted at 25, 400, and 500 °C. The strongest hardening is observed for specimens irradiated at 390 °C, and very little hardening is observed for specimens irradiated at 430 °C. Strain rate jump tests were performed on NLF-0 and NLF-1 at 400 °C after irradiation to 52 dpa. The rate sensitivity, *m*, is quite low, 0.003–0.005 and does not appear to be affected by irradiation at 52 dpa for an irradiation temperature of 430 °C.

1. Background

The Advanced Fuel Cycle Initiative is investigating different options for transmutation of high level nuclear waste. In a proposed proliferation-resistant fuel cycle, a Generation IV fast reactor is used to transmute transuranic isotopes in spent nuclear fuel [1]. The structural materials used to contain this target will experience extensive radiation damage possibly up to 200 dpa at operating temperatures of 400–600 °C depending on the fast reactor concept chosen. To aid in quantifying the effect that this environment has on the mechanical properties of candidate alloys, a series of ferritic–martensitic steels irradiated in the fast flux test facility (FFTF) at ~400 °C to doses up to 67 dpa were tested in tension after irradiation.

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The specimens selected for testing after irradiation in the FFTF reactor are reduced activation ferritic–martensitic steels called NLF alloys. These alloys were engineered to reduce activation after irradiation by replacing the molybdenum typically used in Mod 9Cr–1Mo with tungsten. Also, additions of boron, titanium, aluminum and yttrium were added to specific alloys. Boron (0.003 wt%) was added to NLF 1, 2, 3, and 4 to produce helium during irradiation. Titanium (0.002 wt%) was added to all NLF alloys to suppress void swelling. Aluminum (0.03 wt% in NLF 2 and 3 and 0.01 wt% in NLF-4) was added to suppress hydrogen embrittlement but led to degradation in fracture toughness and an increase in the ductile-to-brittle transition temperature (DBTT). Yttrium (0.01 wt%) was added to NLF-3 and 4 to improve high temperature strength and minimize microstructural changes caused by irradiation [2]. One additional reason for adding yttrium and aluminum was to stabilize the dislocation structure in the martensitic phase.

Some tensile tests were performed previously on these alloys by Kurishita et al. [3]. The NLF-0 and 1 alloys were tested by Kurishita after irradiation to 28 dpa at 390 °C and after 33–35 dpa at irradiation temperatures of 430 °C, 520 °C, and 600 °C. NLF 2, 3, and 4 were also tested after irradiation to 28 dpa at 390 °C but for irradiation to 33 dpa, testing was only performed on specimens irradiated at 430 °C. Tensile test temperatures were room temperature, 200 °C, 400 °C, and 600 °C. In general, Kurishita's results showed the tensile properties of these alloys to have excellent irradiation resistance [3].

To further understand the effects of irradiation on the tensile properties of reduced activation ferritic–martensitic steels, the present work extends the previous testing performed on NLF-0, 1, 2, 3, and 4. Testing was performed at 25 °C, 400 °C, and 500 °C after irradiation up to 67 dpa at ~400 °C in the FFTF reactor. These results are compared with each other as well as to results from previous testing of these alloys. In addition, strain rate jump tests were performed on the NLF-0 and NLF-1 alloys before and after irradiation at 52 dpa.

2. Experimental

NLF alloys were melted in an induction furnace (50 kg), forged into plates of 15 mm thickness and hot rolled to 3 mm thickness at Material Research Lab., Kobe Steel Ltd. [4]. Chemical compositions of the alloys are given in Table 1. Heat treatment consisted of normalizing at 1323 K for 30 min and tempering for 1 h at 1013 K. Tensile specimens were punched from sheets machined to 0.25 mm thick. The specimens had the dimensions of an S-1 tensile specimen which is a flat dogbone specimen 16 mm long, 4 mm wide, 0.25 mm thick with gauge dimensions of 1.2 mm × 5 mm. The alloys were irradiated in the FFTF reactor under conditions listed in Table 2. Specimens were shipped from Pacific Northwest National Laboratory to Los Alamos National Laboratory using a Type 7A certified shipping container. Tensile testing was performed using an Instron test machine located in a hot cell. The test machine is equipped with a high temperature furnace capable of testing up to 700 °C in argon.

Testing was performed following ASTM E-8M-93. The initial strain rate was 5×10^{-4}/s which is similar to the rate used by others who have tested the NLF steels. Specimens tested at elevated temperatures were held for 1 h to equilibrate before testing. The tensile test matrix is shown in Table 3. Two specimens were tested in all conditions except for testing at 22 dpa as only one specimen was available. To investigate rate sensitivity in the NLF-0 and NLF-1 alloys, strain rate jump tests were performed at 400 °C on specimens after irradiation at 52 dpa and on control specimens. For these tests, the test was started at a strain rate of 10^{-4}/s and jumped to 10^{-3}/s after a uniform plastic flow regime was observed. Strain rate jumps were continued until the ultimate tensile stress was obtained.

Engineering stress vs. engineering strain was calculated from the original gauge thickness, length and width (measured before testing) after subtracting the machine and fixture compliance from the load/displacement curves. These curves were used to determine 0.2% offset yield stress, ultimate tensile strength, uniform elongation and total elongation.

Table 1
Alloy designation and composition of NLF alloys

Alloy	Composition (wt%)											
	C	Si	Mn	P	S	Cr	W	V	Ta	Ti	B	Y	Al
NLF-0	0.10	0.056	0.50	0.002	0.0017	8.65	1.92	0.25	0.044	0.020	–	–	–
NLF-1	0.10	0.042	0.53	0.002	0.0014	9.03	2.06	0.26	0.051	0.021	0.0032	–	–
NLF-2	0.10	0.050	0.53	0.002	0.0020	8.98	2.01	0.25	0.059	0.018	0.0029	–	0.028
NLF-3	0.10	0.040	0.52	0.002	0.0010	8.92	2.01	0.24	0.058	0.017	0.0027	0.009	0.029
NLF-4	0.10	0.030	0.52	0.002	0.0020	9.01	2.03	0.25	0.060	0.017	0.0027	0.010	0.009

Table 2
Irradiation conditions for NLF alloys after irradiation in FFTF

Material	MOTA/location	Irr. temp. (°C)	Incremental dose (dpa)	Total dose (dpa)
NLF-0	2A/1A3	390	22.2	22.2
NLF-1	2A/1A3	390	22.2	22.2
NLF-0	2A/2E3	407	36.4	36.4
NLF-1	2A/2E3	407	36.4	36.4
NLF-1	2A/2E3	407	36.4	36.4
NLF-0	2A/3D3	427	44.0	44.0
NLF-1	2A/3D3	427	44.0	44.0
NLF-0	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-0	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-1	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-1	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-1	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-2	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-2	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-2	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-3	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-3	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-3	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-3	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-4	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-4	2A/4D1, 2B/4D4	430, 430	29.2, 22.6	51.8
NLF-0	2A/3D3, 2B/2E2	427, 408	44.0, 23.5	67.5
NLF-1	2A/3D3, 2B/2E2	427, 408	44.0, 23.5	67.5

Table 3
Tensile test matrix for NLF alloys after irradiation in FFTF

Material	Irr. temp. (°C)	Dose (dpa)	Test temp. (°C)
NLF-0	390–430	22 (400 °C only), 36, 44, 52, and 67	20, 400, 500
NLF-1	390–430	22, 36, 44, 52, and 67	20, 400
NLF-2	430	52	20, 400
NLF-3	430	52	20, 400
NLF-4	430	52	20, 400

3. Results and discussion

Tensile tests were performed on NLF-0, 1, 2, 3, and 4 before and after irradiation to 52 dpa. Representative stress/strain curves for these alloys are shown in Fig. 1(A) and (B) before and after irradiation respectively. The stress/strain response for these alloys is quite similar. NLF-0 has the lowest yield stress before irradiation. After irradiation to 52 dpa, NLF-0 still has the lowest yield stress, while the lowest elongation is observed for NLF-2.

To investigate in detail the effect of irradiation dose on the tensile properties of the NLF alloys, NLF-0 and NLF-1 were tested at 25 and 400 °C after irradiation to 22, 36, 44, 52, and 67 dpa. The results show apparent but relatively small changes in tensile properties with dose. Representative stress/strain curves for NLF-0 specimens tested at 400 °C are shown in Fig. 2. The changes in yield stress, ultimate stress, and uniform elongation vs. dose are summarized in Figs. 3 and 4 for tests at room temperature and 400 °C, respectively. In general, maximum hardening and minimum uniform elongation are observed for testing after irradiation to 22 dpa at 390 °C, and little change is observed for testing after irradiation to higher doses. Some testing has been performed on these NLF alloys by Kurishita et al. after irradiation to 28 and 33–35 dpa [3]. These results are plotted with our results in Figs. 3 and 4 for NLF-0 and NLF-1. The previous results agree well with our results.

The yield stress (YS) and ultimate tensile stress (UTS) values vary non-monotonically with dose. This non-monotonic behavior has been observed in the RAFM JLF steels irradiated by other researchers in the same MOTA cycles that were used for the NLF irradiations, and no explanation of the dose dependence of

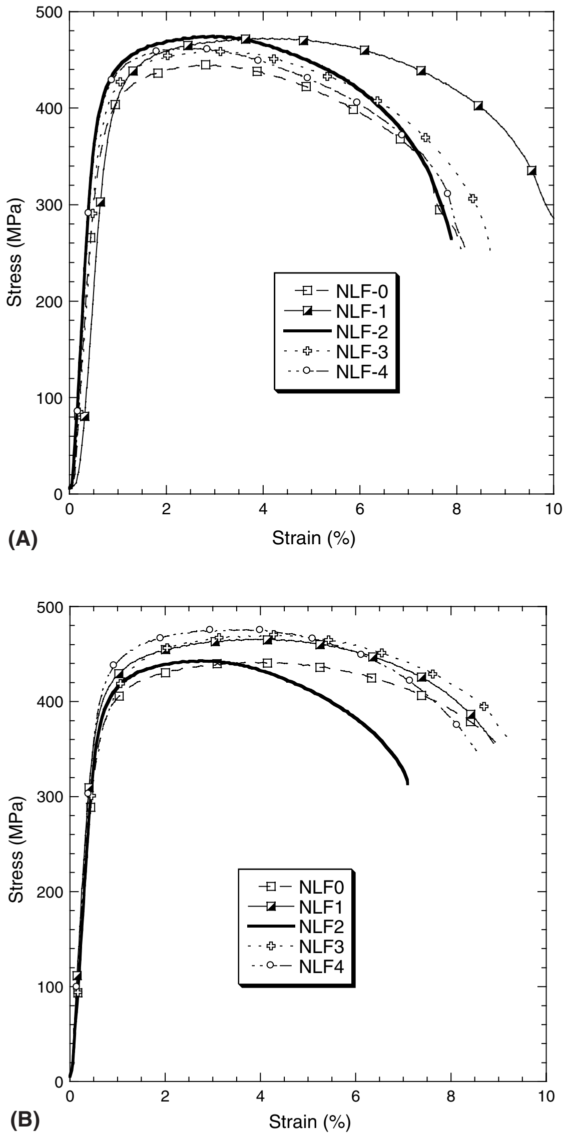

Fig. 1. Stress/strain curves measured on tensile specimens of NLF alloys at 400 °C before (A) and after (B) irradiation to 52 dpa at 430 °C in the FFTF reactor.

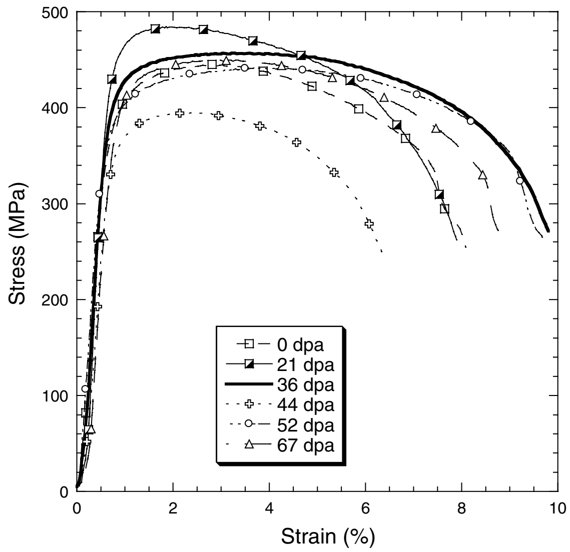

Fig. 2. Stress/strain curves measured on NLF-0 at 400 °C after irradiation in the FFTF reactor.

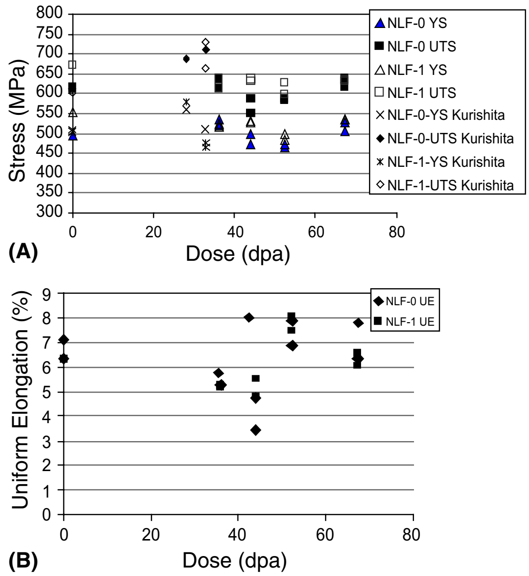

Fig. 3. Graphs showing the variation in yield stress and ultimate stress (A) and uniform elongation (B) with dose for NLF-0 and 1 measured at 25 °C after irradiation in the FFTF reactor.

the tensile properties was offered [5,6]. There are a limited number of open literature papers that present the dose dependence of the tensile properties of ferritic–martensitic steels [7–12]. All of these papers [5–12] all show that ferritic–martensitic steels irradiated at ∼400 °C harden within the first 10 dpa. Beyond about 15 dpa, there is some ambiguity as to whether the materials maintain their hardness or begin to soften with further dose. What does stand out in many of these papers [8,10] and another paper [13] though is the very strong irradiation temperature dependence of the tensile properties of ferritic–martensitic steels in the irradiation temperature range from 350 °C to 450 °C.

In considering the irradiation conditions for the NLF specimens and the tensile behavior reported in the literature, there appears to be an explanation for the apparent non-monotonic dose dependence of the NLF steels. In comparing the tensile properties and the exact irradiation

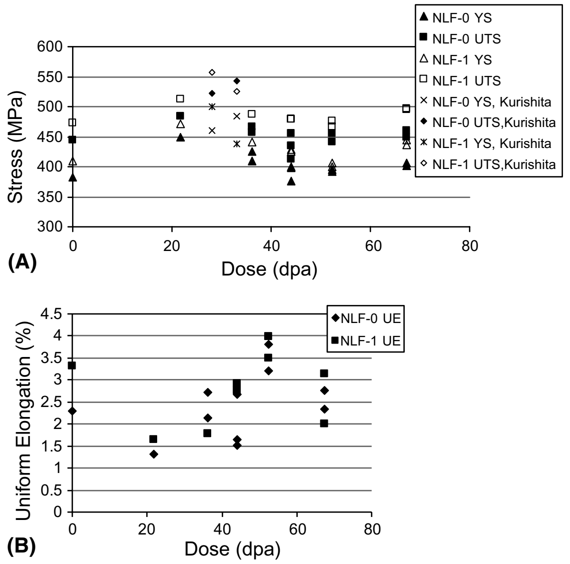

Fig. 4. Graphs showing the variation in yield stress and ultimate stress (A) and uniform elongation (B) with dose for NLF-0 and 1 measured at 400 °C after irradiation in the FFTF reactor.

temperature, there is a strong relationship between the magnitude of the tensile properties and the irradiation temperature in the temperature range from 390 °C to 430 °C. Fig. 5 plots the change in yield stress with irradiation temperature. Because the 67 dpa specimens finished with an irradiation at 407 °C, they are plotted with an irradiation temperature of 407 °C. The data shows clearly that the amount of hardening decreases significantly from irradiation temperatures of 390 °C to 407 °C to 430 °C. Very little hardening is observed for irradiations performed at 430 °C. There is one point at 430 °C (Fig. 5(A)) from previous testing by Kurishita et al. [3] that does show a noticeable hardening. This is puzzling but possibly the specimen was bent before testing as all other data show little to no hardening at 430 °C. This strong dependence of YS of the NLF alloys on irradiation temperature can be seen in the results of Kurishita et al. [3] and Kimura et al. [13], where the NLF alloys were tensile tested after 28–35 dpa at irradiation temperatures ranging from 390 °C to 600 °C. They show that NLF-0 irradiated to 33 dpa at 430 °C has a 50 MPa lower yield strength than the same material irradiated to 28 dpa at 390 °C. For NLF-1 at the same irradiation conditions, the difference in yield strength is 100 MPa.

The NLF-0 alloy was also tested at 500 °C before and after irradiation at temperatures from 407 to 430 °C. Stress/strain plots are shown in Fig. 6, and tensile properties vs. dose are shown in Fig. 7. In general, the 500 °C tensile test results display the same trends vs. dose as

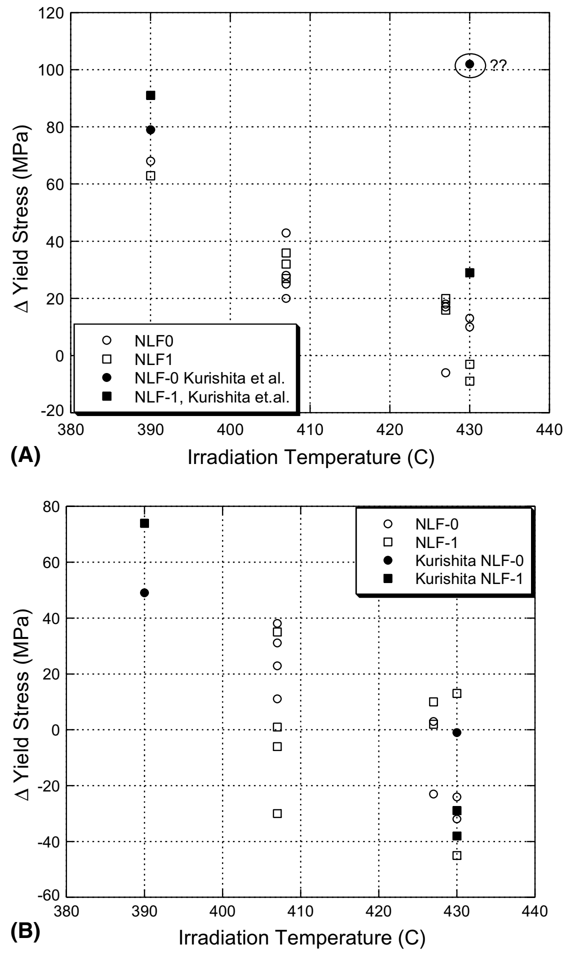

Fig. 5. Graph showing the variation in the change in yield stress with irradiation temperature for NLF-1 and NLF-0 for testing at (A) 25 °C and (B) 400 °C after irradiation in the FFTF reactor.

seen at 25 °C and 400 °C, but the strength values are about 100 MPa lower than observed after testing at 400 °C, and there is a decrease in the uniform elongation relative to the 400 °C tests. The effect of the small variation in irradiation temperature on the tensile properties can be seen even when the material is tested at 500 °C. As with the tensile tests conducted at 25 °C and 400 °C, the irradiated material is only equal in strength or just very slightly stronger than the unirradiated control material.

Strain rate sensitivity was investigated in NLF-0 and NLF-1 alloys before and after irradiation to 52 dpa at an irradiation temperature of 430 °C and a test temperature of 400 °C. Strain rate jump tests were performed by jumping the strain rate from 10^{-4}/s to 10^{-3}/s during testing. A typical rate jump test is shown in Fig. 8 for NLF-0 before (B) and after irradiation (A). Rate

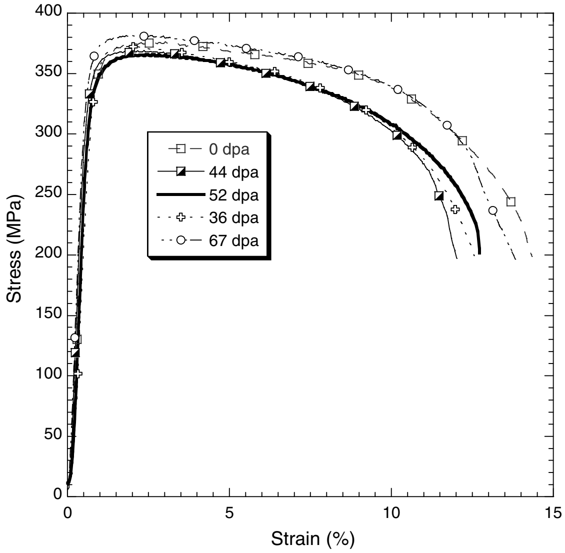

Fig. 6. Stress/strain curves measured on NLF-0 at 500 °C after irradiation in the FFTF reactor.

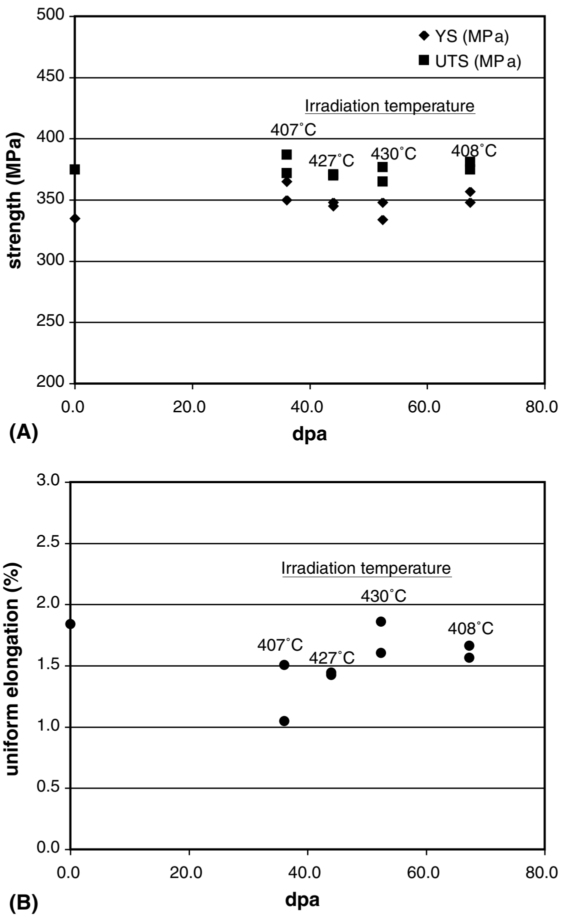

Fig. 7. (A) YS and UTS for NLF-0 irradiated at ~400 °C and tested at 500 °C, and (B) engineering uniform elongation.

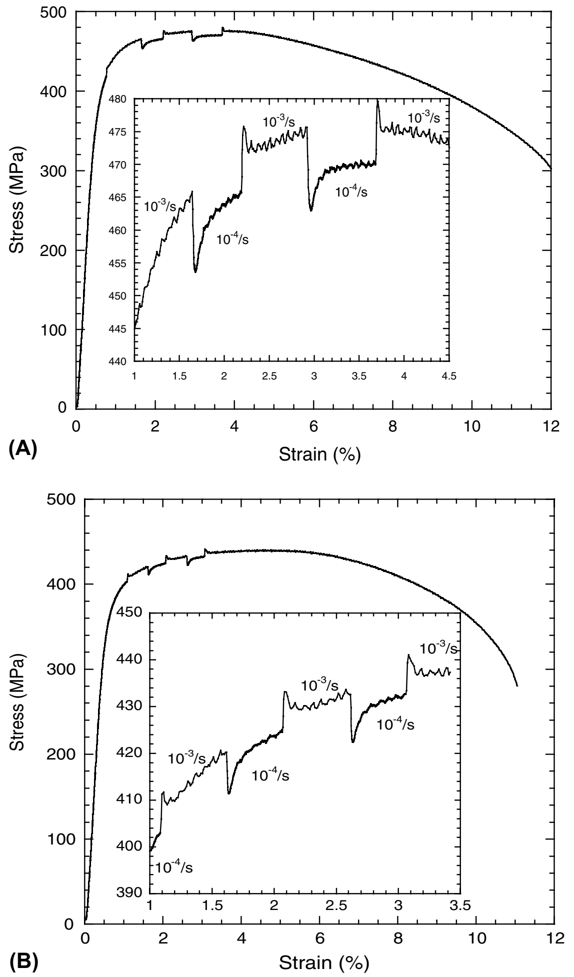

Fig. 8. Stress/strain curve for a rate jump test performed on NLF-0 at 400 °C (A) before irradiation and (B) after irradiation to 52 dpa in the FFTF reactor.

sensitivity, m, was calculated from these tests using the following formula:

$$m = \frac{\log(\sigma_2/\sigma_1)}{\log(\dot{\varepsilon}_2/\dot{\varepsilon}_1)},$$

where σ_1 and σ_2 are the stresses and $\dot{\varepsilon}_1$ and $\dot{\varepsilon}_2$ are the strain rates before and after the rate jump. The results of these rate sensitivity tests are a value of 0.0056–0.0062 before irradiation and 0.0020–0.0050 after irradiation. These results show that NLF-0 and NLF-1 alloys are very insensitive to strain rate changes before and after irradiation at 52 dpa at an irradiation temperature of 430 °C and a test temperature of 400 °C.

4. Summary and conclusions

Tensile testing has been performed at 25, 400, and 500 °C on FFTF-irradiated reduced activation ferritic–martensitic steels (NLF-0, 1, 2, 3, and 4) after irradiation to up to 67 dpa at ~400 °C. Variations in irradiation temperature in the range of 390–430 °C have a relatively small, but definite effect on the tensile properties for tests conducted at 25, 400, and 500 °C. The strongest hardening is observed for specimens irradiated at 390 °C, and very little hardening is observed for specimens irradiated at 430 °C. When these data are plotted vs. dose, these variations in tensile properties caused by irradiation temperature give the appearance that the alloys first harden with dose, and then later soften with further dose.[1] Where little irradiation-induced hardening is observed, the uniform elongation is about equal to the unirradiated value. In consideration of the irradiation temperature variations, it appears that any effect of irradiation dose on tensile properties occurs relatively early.

Strain rate jump tests were performed on NLF-0 and NLF-1 at 400 °C before and after irradiation to 52 dpa. The rate sensitivity, m, is quite low, 0.003–0.005 and does not appear to be affected by irradiation at 52 dpa for an irradiation temperature of 430 °C.

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