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The effects of fast reactor irradiation conditions on the tensile properties of two ferritic/martensitic steels

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Abstract

Tensile testing has been performed at 25 and at ~400 °C on two ferritic/martensitic steels (JFMS and HT-9) after irradiation in FFTF to up to ~70 dpa at 373–433 °C. As observed in previous studies, this range of irradiation temperatures has a significant effect on hardening. The percent increase in yield stress decreases with increasing irradiation temperature from 373 to 433 °C. The JFMS alloy, which has 0.7 wt% silicon, exhibits approximately a factor of two increase in yield strength between tests at 427 and at 373 °C, and shows an increase in hardening with increasing dose. A comparison of the JFMS tensile properties to the properties of other ferritic/martensitic steels suggests that this hardening is due to precipitation of a Si-rich laves phase in this alloy. The HT-9 alloy, which contains more chromium and more carbon but less silicon (0.2 wt%), less molybdenum and less nickel, hardens during irradiation at 373 °C, but shows less hardening for irradiations performed at 427 °C and no increase in yield stress with increasing dose beyond 10 dpa.
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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 and may be in contact with lead or a lead/bismuth eutectic 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 ~70 dpa were tested in tension after irradiation. In addition, the results of previous tensile testing at PNNL are also reported.

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Table 1
Composition of HT-9 and JFMS alloys

Alloy	Composition (wt%)									
	C	Si	Mn	P	S	Cr	Mo	Ni	V	Nb
JFMS	0.05	0.67	0.58	0.009	0.006	9.58	2.31	0.94	0.12	0.06
HT-9	0.2	0.2	0.6			11.95	1.0	0.6	0.3	

The specimens selected for testing after irradiation in the FFTF reactor are tensile specimens of the HT-9 and JFMS ferritic/martensitic steels. HT-9 was selected by the US breeder reactor program in the 1980s for its low swelling, high strength, and good sodium resistance [2,3]. JFMS is a dual-phase ferritic/martensitic steel that was one of the reference materials developed under the Japanese fusion program [4]. The compositions of these steels are shown in Table 1. A few specific differences can be noted when comparing the composition of the JFMS steel to that of the HT-9 alloy. The JFMS steel has much less carbon which allows the formation of a dual-phase ferritic/martensitic alloy upon initial quench from the austenization temperature, the JFMS steel has less chromium, and the JFMS steel has more silicon and molybdenum added to it. The silicon addition is of specific interest because alloys developed for improved corrosion resistance in lead–bismuth fast reactors have added silicon up to 1% but such large amounts of silicon have led to embrittlement in EP-823 for irradiations at temperatures below 460 °C [5].

There have been numerous studies on the effects of irradiation on the JFMS alloy. Many studies were performed to specifically look at tensile specimen size effects using this as a reference alloy. These were performed after irradiation in the Japanese Material Test Reactor (JMTR) and FFTF at temperatures from 160 to 520 °C [6,7] and doses up to 70 dpa and in the Rotating Target Neutron Source-II (RTNS-II) (14 MeV neutron irradiation) at 20–290 °C [8,9]. In addition, microstructural analyses and mechanical tests have been performed after 14 MeV neutron (RTNS-II) [10–12], helium [13,14] and aluminum [15] ion, and neutron irradiation in FFTF, JOYO and the Experimental Breeder Reactor-II (EBR-II) [4,16–21] at temperatures from 20 to 650 °C.

To further understand the effects of irradiation on the tensile properties of HT-9 and JFMS, the present work extends the previous testing performed to investigate the effects of irradiation and test temperature on tensile properties in these alloys. Testing was performed at 20, 250, 350 and 400 °C after irradiation up to 67 dpa at 373–427 °C in the FFTF reactor. Tensile data previously obtained on the JFMS alloy at PNNL are also reported. Most of this data have not been previously published, but some of this data have been published by Kohno et al. [6]. These JFMS specimens were also irradiated in FFTF under almost identical conditions to the other JFMS specimens. These results are compared with each other as well as to results from previous testing of these alloys.

2. Experimental

JFMS and HT-9 alloys were irradiated with the chemical compositions shown in Table 1. JFMS is a dual-phase ferritic/martensitic steel. Data on two different series of tensile studies on JFMS are presented. One set of specimens was tested at LANL while the other set was tested at PNNL as part of an earlier study [6]. The pre-irradiation heat treatment of the specimens tested at LANL was 1050 °C/30 min/AC followed by 775 °C/6 h/AC. The specimens had the dimensions of an S-1 tensile specimen which is a flat dogbone specimen 16 mm long, 4 mm wide, 0.15–0.25 mm thick with gauge dimensions of 1.2 mm × 5 mm. The JFMS specimens from the previous testing at PNNL were also S-1 type specimens, but with a thickness ranging from 0.10 to 0.50 mm thick. These specimens were part of a tensile specimen size effects study [6]. Several different heat treatments were used to obtain varying ratios of ferrite-to-martensite volume. The heat treatment schedules of these JFMS specimens could not be determined. Depending on which heat treatment was applied, the ratio of ferrite volume to martensite volume following normalization ranged from 0.32 to 0.37. The HT-9 specimens tested for this study were also in the S-1 geometry with a thickness of about 0.25 mm. The details on the heat treatment schedule of these specimens also could not be determined. The alloys were irradiated in the FFTF reactor under conditions listed in Table 2. Selected JFMS and HT-9 specimens were shipped from

Table 2
Irradiation and test conditions for HT-9 and JFMS alloys irradiated in FFTF

Alloy	MOTA 2A location	Irrad. temp. (°C)	Incremental dose (dpa)	MOTA 2B location	Irrad. temp. (°C)	Incremental dose (dpa)	Total dose (dpa)	Test temperatures (°C)
HT-9	BCE1	373	9.8	NP[a]	–	–	9.8	25, 400
	BCE4	373	5.8	BCE1	373	6.99	12.8	400
	1A3	390	22.2	NP	–	–	22.2	25, 400
	1A3	390	22.2	1A4	387	13.1	35.3	25, 400
	3D3	427	44.0	NP	–	–	44.0	25, 400
	3D3	427	44.0	2E2	408	23.5	67.5	25, 400
JFMS	BCE1	373	9.8	NP	–	–	9.8	25, 250, 350, 400
	1A3	390	22.2	NP	–	–	22.2	25, 400
	1A3	390	22.2	1A4	387	13.1	35.3	25, 400
	3D3	427	44.0	NP	–	–	44.0	25, 400
JFMS-Kohno	3C1	423	43.6	NP	–	–	43.6	25, 420
	3C1	423	43.6	3A4	433	25.8	69.7	25, 420

[a] NP: not present in this irradiation cycle.

Pacific Northwest National Laboratory to Los Alamos National Laboratory using a Type 7A certified shipping container. At both LANL and PNNL, tensile testing was performed using an Instron screw-driven test machine located in a hot cell. Both test machines are 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. Specimens tested at elevated temperatures were held for 1 h to equilibrate before testing. The tensile test matrix is shown in Table 2. One or two specimens were tested in all conditions.

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.

3. Results and discussion

Tensile tests were performed on JFMS and HT-9 steels before and after irradiation to doses up to ~70 dpa at room temperature and 400 or 420 °C. Representative stress/strain curves for these alloys are shown in Figs. 1 and 2 before and after irradiation for testing at 20 and 400 °C. Although both alloys show the expected increase in strength and decrease in ductility with increasing dose, there are some differences between the stress/strain response observed for these two alloys. First, the JFMS alloy has a lower initial strength and a higher ductility than HT-9. Second, the unirradiated JFMS alloy exhibits serrated yielding (dynamic strain aging) when tested at around 400 °C. Third, hardening continues to increase with dose in the JFMS steel at 390 °C but saturates at low doses in the HT-9 alloy at the same irradiation temperature. This is seen for the specimens irradiated at 390 °C showing that the yield stress continues to increase in the JFMS alloy as the dose is increased from 22 to 35 dpa.

To investigate the effect of test temperature on the mechanical properties of the JFMS alloy, additional testing was performed at 250 and 350 °C for specimens irradiated to 9.8 dpa at a temperature of 373 °C. The stress/strain curves for the specimens tested at these temperatures are shown in Fig. 3(A). As test temperature is increased from 20 to 400 °C, a decrease in yield stress and ductility are both observed. In addition, serrated yielding is once again observed when testing was performed at 350 °C as shown in Fig. 3(B). The strong decrease in ductility with increasing test temperature is not a large surprise. Previous work [22] has shown that this effect is related to the dynamic strain aging as it is clearly shown in the unirradiated specimens as well when comparing the stress/strain curves for the tests performed at 20 °C to the tests performed at 400 °C.

The yield stress and percent change in yield stress of JFMS and HT-9 are shown in Fig. 4(A) and (B),

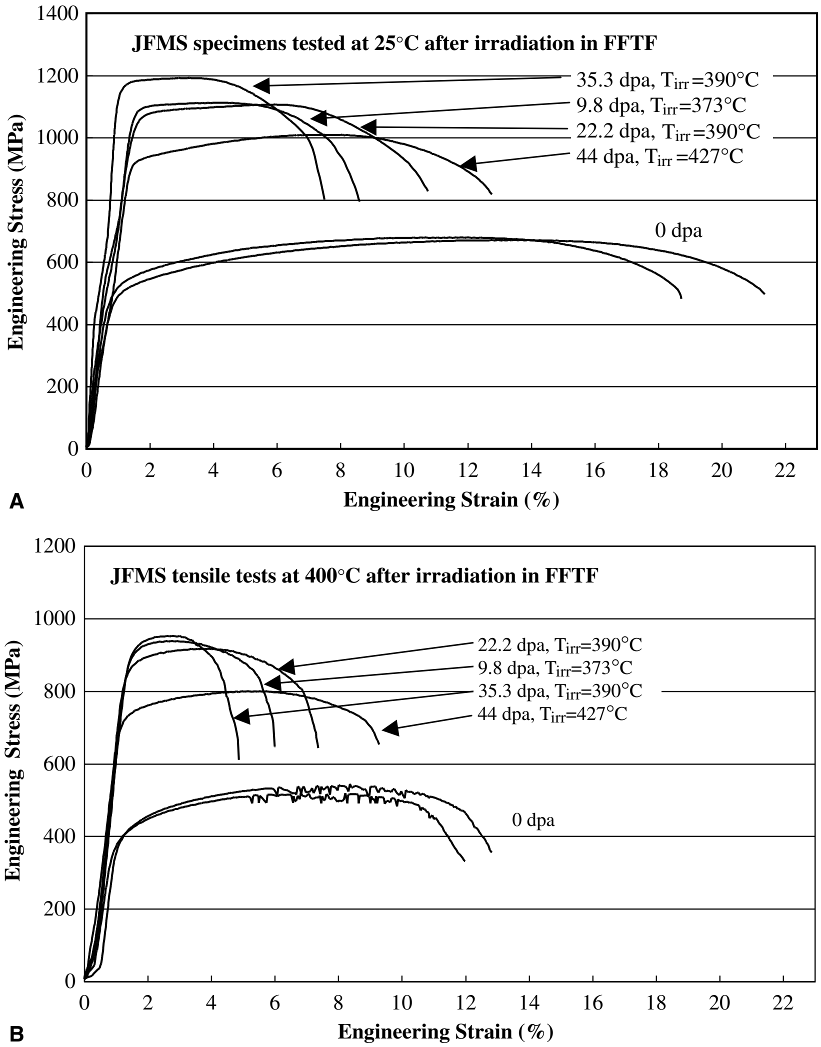


Fig. 1. Stress/strain curves measured on tensile specimens of the JFMS alloy at (A) 25 °C and (B) 400 °C after irradiation to up to 44 dpa at 373–427 °C in the FFTF.

respectively. Both alloys show an effect of irradiation temperature on hardening. Irradiation at 373 and 390 °C causes a much stronger increase in yield stress than irradiation at temperatures between 423 and 433 °C. The changes in ultimate tensile strength of the two steels mirror the changes in yield strength. The uniform elongation of JFMS and HT-9 are shown in Fig. 5. The pre-irradiation uniform elongation of JFMS is much higher than HT-9, but after irradiation, both materials have approximately the same uniform elongation.

For irradiations between 373 and 390 °C, HT-9 hardens by about 50%. This is in contrast to the developmental reduced activation ferritic/martensitic (RAFM) 9Cr steels such as the NLF steel. These steels harden by about 20% in this same temperature range [23]. The hardening in the HT-9 is thought to be due to the irradiation-induced formation of precipitates that block dislocation motion. There have been several microstructural examinations of HT-9 after irradiation at temperatures from 400 to 450 °C [24–28], but there are no reported microstructural observations in the literature after irradiation at temperatures from 350 to 400 °C. The TEM observations from 400 to 450 °C irradiations have shown that, in general, irradiation at ~400 °C causes the formation of a distribution of small loops and different types of precipitates less than 10 nm in size. The precipitates have been identified as a Si-rich G-phase [24–26], a Cr-rich α′ phase

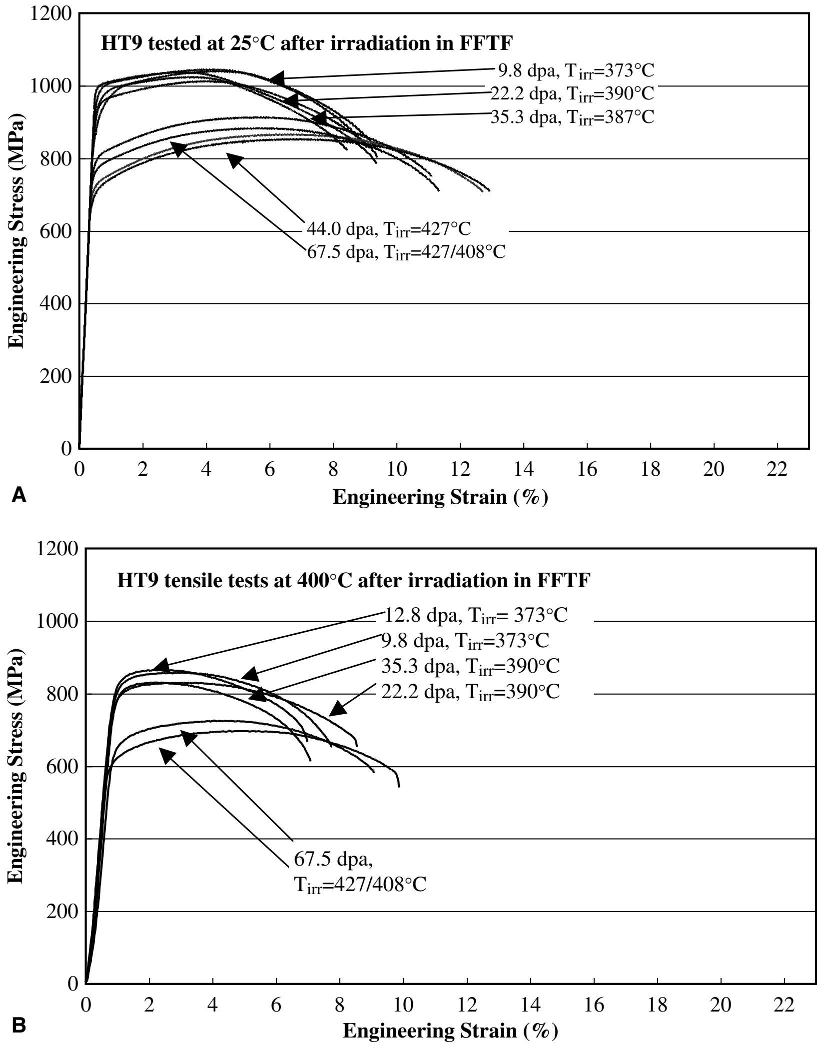
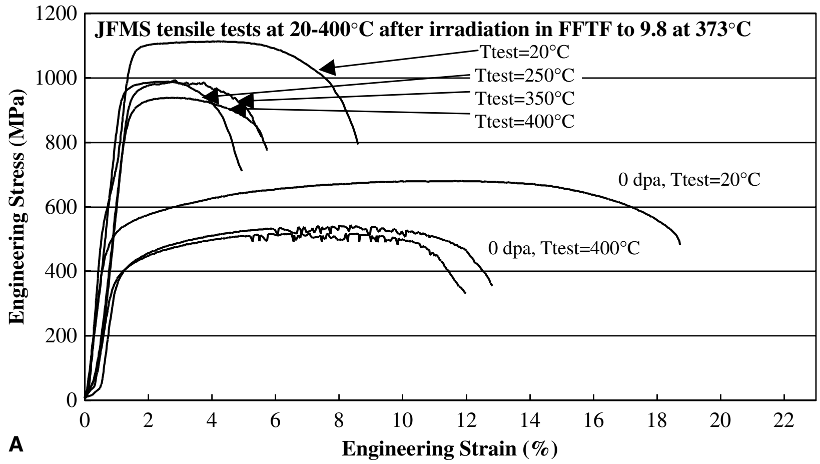


Fig. 2. Stress/strain curves measured on tensile specimens of the HT-9 alloy at (A) 25 °C and (B) 400 °C after irradiation to up to 67 dpa at 373–427 °C in the FFTF.

[24,25,27], and M_6C [28]. While the HT-9 does harden significantly, it maintains reasonable uniform elongation values which are equal to, or slightly better than that of the RAFM NLF steels [23]. It is expected that as the irradiation temperature is reduced somewhat below 400 °C, these precipitates will become smaller in size with a higher density. Some point defect clusters, which are perhaps just extremely small precipitates, would not be unexpected at 370 °C. An increasing density of small hard precipitates at lower irradiation temperatures would explain the increase in strength of the alloys at lower irradiation temperatures.

There are some marked differences between the JFMS and HT-9 alloys. First, a much larger percent change in yield stress in observed in the JFMS alloy at all doses and irradiation temperatures. Second, very little increase in yield stress is observed in the HT-9 alloy when irradiated at 427 °C while almost an 85% increase is observed in the JFMS alloy. This may be a result of the increased silicon content in the JFMS alloy. Aging studies performed on the JFMS alloy at temperatures from 500 to 600 °C show that Si has a significant effect on toughness caused by precipitation of laves phases containing Si and P [29,30]. Such precipitation has also been observed after irradiation at 400 °C [20,21]. Such strong increases in yield strength have also been seen in other high silicon ferritic/martensitic steels. EP-823, a ferritic/martensitic steel containing 1.1 wt% silicon,



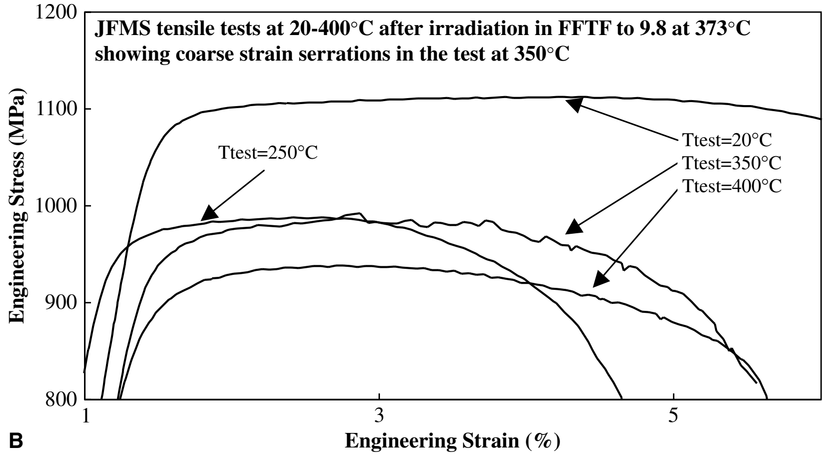


Fig. 3. (A) Overall stress/strain curves measured on tensile specimens of the JFMS alloy at various test temperatures from 20 to 400 °C after irradiation in the FFTF reactor to 9.8 dpa at a temperature of 373 °C, and (B) close-up of the plastic deformation showing strain serrations in the test performed at 350 °C.

was recently irradiated at 140–340 °C in STIP-II along with HT-9 specimens [31]. The relatively low irradiation temperature produced strong hardening in both materials. When they were tested at room temperature, the EP-823 fractured in the elastic regime, and when tested at 400 °C, the EP-823 had a consistently greater yield strength than the HT-9.

The pre-irradiation uniform elongation of JFMS is almost twice as great as HT-9 which shows that JFMS has good initial work hardening ability. After irradiation, the uniform elongation of the two steels is similar. The good initial work hardening ability of JFMS is offset by a large radiation-induced loss in work hardening ability, probably due to the formation of the Si-rich laves phase which substantially raises the yield strength of the material.

Although there have been many studies on the effects of irradiation on the JFMS alloy, there are few published studies at the equivalent irradiation temperature of 373–433 °C and at the high doses from irradiation in FFTF. Some data points were obtained from an irradiation in EBR-II [20]. The EBR-II irradiation was quoted as a total dose of 3×10^{26} n/m^2 which was (not knowing the exact position in reactor) translates to ~15 dpa using the correlation described by Garner [32]. The yield strength of JFMS from the EBR-II irradiation is in agreement with the yield stress from the FFTF irradiations.

4. Summary and conclusions

Tensile testing has been performed at 25 and 400 °C on FFTF-irradiated ferritic/martensitic steels (JFMS and HT-9) after irradiation to up to ~70 dpa at ~400 °C. As observed in previous

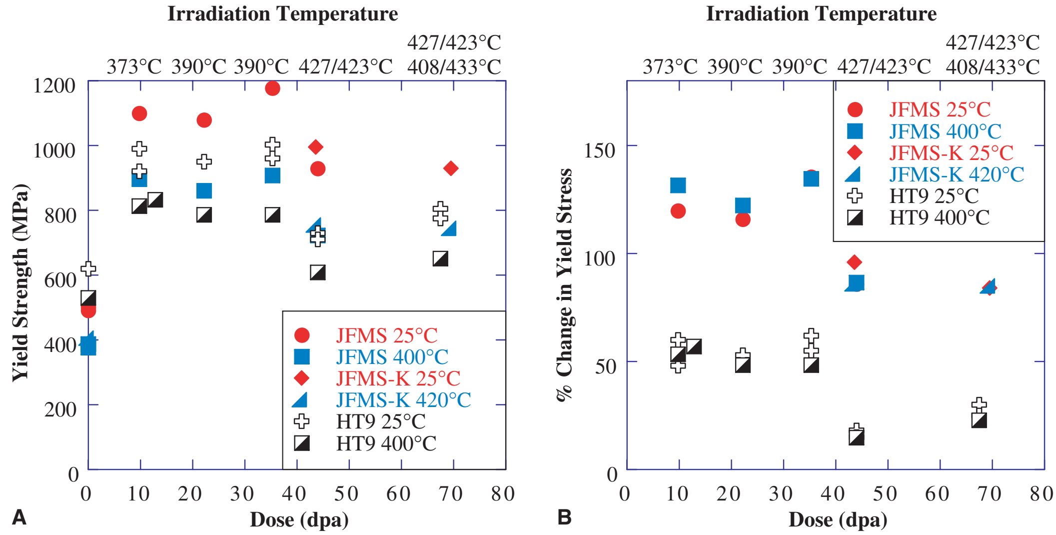


Fig. 4. Graphs showing the variation in (A) yield stress and, (B) the percent change in yield stress with dose for JMFS and HT-9 steels measured at 20 and 400 °C after irradiation in the FFTF reactor at ~400 °C. 'JFMS-K' specimens are specimens that were tested as part of Kohno's tensile specimen size effects study [6].

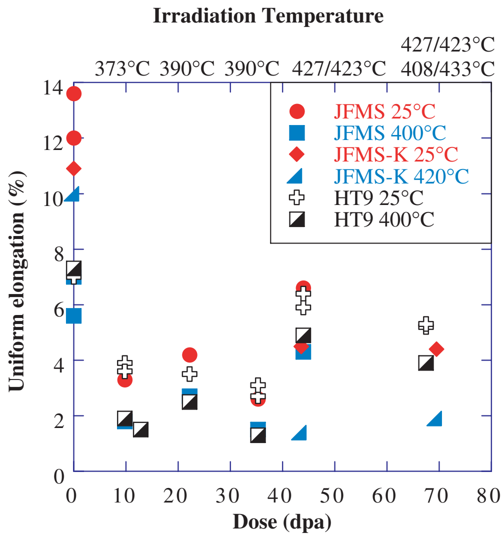


Fig. 5. Uniform elongation of JFMS and HT-9 after irradiation at ~400 °C in FFTF. 'JFMS-K' specimens are specimens that were tested as part of Kohno's tensile specimen size effects study [6].

studies, irradiation temperature has a significant effect on hardening, especially in the temperature range studied here. The percent increase in yield stress decreases with increasing irradiation temperature from 373 to 423 °C. The hardening of the HT-9 is likely due to the radiation-induced formation of a variety of precipitates including a Si-rich G phase and a Cr-rich α′ phase. For the JFMS alloy, these test results and test data on another high silicon ferritic/martensitic steel published in the literature suggest that silicon has a strong effect on hardening and embrittlement. The JFMS alloy, which has 0.7 wt% silicon, exhibits strong hardening at all irradiation temperatures, and at 390 °C irradiation temperature, JFMS shows an increase in hardening with increasing dose, at least to 35 dpa. Previous work has related this hardening to precipitation of the laves phase in this silicon containing alloy. The HT-9 alloy contains much less silicon (0.2 wt%) and shows less hardening than JFMS for irradiations performed at 427 °C and very little increase in yield stress with increasing dose.

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