CONF-860421--25

POSTIRRADIATION TENSILE BEHAVIOR OF NICKEL-DOPED FERRITIC STEELS*

R. L. Klueh, P. J. Maziasz, and J. M. Vitek

Metals and Ceramics Division, Oak Ridge National Laboratory
Oak Ridge, TN 37831

CONF-860421--25

DE86 009502

Abstract

Tensile specimens of normalized-and-tempered 9Cr-1MoVNb, 9Cr-1MoVNb-2Ni, 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni were irradiated in the Experimental Breeder Reactor at 390, 450, 500, and 550°C to displacement-damage levels of approximately 16 dpa. The only difference in the effect of irradiation on the tensile behavior of the nickel-doped and undoped steels was attributed to the difference in tempering treatments the two types of steels received. The nickel-doped steels were stronger prior to irradiation due to a lower tempering temperature. After irradiation, the properties of the steels with and without nickel were similar, indicating that the presence of nickel did not affect the behavior of the steels during irradiation. Nickel was added to the steels to study the effect of helium on the properties of these steels. Helium can be formed in an alloy containing nickel by irradiating in a mixed-spectrum reactor. To help determine the effect of helium on properties, these steels are also being irradiated in fast reactors, where little helium is formed. The present fast-reactor results indicate that it is feasible to use the nickel-doped ferritic steels to study helium effects.

*Research sponsored by the Office of Fusion Energy, U.S. Department of Energy, under Contract No. DE-AC05-84OR21400 with the Martin Marietta Energy Systems, Inc.

DISCLAIMER

1. Introduction

To study alloys for fusion reactor first wall applications, it is necessary to have a technique for studying the effect of irradiation-produced displacement damage and transmutation helium simultaneously produced by irradiation. To do this in the 9% Cr and 12% Cr Cr-Mo steels, nickel is added to the steels, and they are then irradiated in a mixed-spectrum reactor such as the High Flux Isotope Reactor (HFIR) [1,2]. Displacement damage is produced by the fast neutrons in the spectrum, and helium is produced by a transmutation reaction of ^{58}Ni with the thermal neutrons in the spectrum. Such a study is in progress on 9Cr-1MoVNb and 12Cr-1MoVW steels to which up to 2% Ni has been added. The steels are being irradiated in HFIR to produce helium and displacement damage. They have also been irradiated in the Experimental Breeder Reactor (EBR-II), a fast reactor where displacement damage occurs but essentially no helium is produced.

This report presents tensile results from specimens irradiated in the AD-2 experiment conducted by the Hanford Engineering Development Laboratory in EBR-II. When the tests on the HFIR-irradiated specimens are completed, the effect of helium on the properties will be determined by comparing the results from the two experiments.

2. Experimental Procedure

Electroslag-remelted heats of standard 12Cr-1MoVW (0.5% Ni) and 9Cr-1MoVNb (0.1% Ni) steels were prepared by Combustion Engineering, Inc., Chattanooga, Tennessee. These compositions with 1 and 2% Ni, designated 9Cr-1MoVNb-2Ni, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni, were also prepared. Sheet tensile specimens were machined from 0.76-mm-thick cold rolled sheet. Gage lengths were machined parallel to the rolling direction; the reduced section was 20.3 mm long by 1.52 mm wide by 0.76 mm thick. The specimens were irradiated in the normalized and tempered condition. The normalizing treatment for the 9 Cr steels w 0.5 h at 1040°C and for the 12 Cr steels 0.5 h at 1050°C, after which they were cooled in flowing helium. The 9Cr-1MoVNb was tempered 1 h at 760°C; the 12Cr-1MoVW and 12Cr-1MoVW-1Ni were tempered 2.5 h at 780°C. The 9Cr-1MoVNb-2Ni and 12Cr-1MoVW-2Ni steels were tempered 5 h at 700°C. Tempered martensite microstructures were obtained by these heat treatments. Details on chemical composition, heat treatment, and microstructure have been published [1—3].

Specimens were irradiated in capsules designed to maintain temperatures of 390, 450, 500, and 550°C. Irradiation was in row 4 of EBR-II, and the specimens were exposed to fluences of ~3.2 to 3.4 × 10^{26} neutrons/m^2 (E > 0.1 MeV), depending on the axial position from the reactor midplane. This fluence produced displacement damage of about 15.4 to 16.2 dpa. The uncertainty in fluence has been estimated as ±10% and the temperature uncertainties are 390 ± 10°C, 450 ± 15°C, 500 ± 20°C, and 550 ± 30°C.

After irradiation, tensile tests were conducted at the irradiation temperature and, where specimens were available, at room temperature. As-heat-treated as well as thermally aged control samples were also tested to separate the effect of irradiation from thermal-aging effects. Thermal aging was at the irradiation temperatures for 5000 h — the approximate time of the irradiation. The tensile tests were made in a vacuum chamber on a 44-kN-capacity Instron universal testing machine at a crosshead speed of 8.5 μm/s, which results in a nominal strain rate of 4.2×10^{-4}/s.

3. Results

In the normalized and tempered condition, the steels with 2% Ni were considerably stronger than the standard materials and the 12Cr-1MoVW-1Ni steel [1,2]. This difference was probably caused by the different tempering treatments used for the steels with 2% Ni. These latter steels were tempered at 700°C, where tempering was slower than for the higher temperatures used for the standard steels and the one with 1% Ni. It was not possible to temper the 2%-Ni steels at the same temperatures because those temperatures are above the A_{C_1} temperature [2].

Thermal aging for 5000 h (the approximate time in the reactor) in the range 400 to 550°C had little effect on the strength properties of the 9Cr-1MoVNb, 12Cr-1MoVW, and 12Cr-1MoVW-1Ni. There was also no effect of thermal aging on the 9Cr-1MoVNb-2Ni and 12Cr-1MoVW-2Ni steels at 400 and 450°C; however, these steels showed a large decrease in strength after aging at 550°C, where the strength of the 2%-Ni steels approached that of the steels without nickel. There was little effect of aging on the ductility of any of the steels. Because of space limitations and because the aged specimens are the most appropriate as controls, the unaged data will not be presented.

The irradiated specimens were compared with the aged specimens for tensile tests at the irradiation (aging) temperatures (Figs. 1 to 6) and for tests at room temperature. (Details on the room-temperature results will not be presented here). Strengthening, as determined by an increased yield stress and ultimate tensile strength, occurred for the 9Cr-1MoVNb, 12Cr-1MoVW, and 12Cr-1MoVW-1Ni steels irradiated at 390°C. Little change in strength occurred for the steels with 2% Ni. There was considerably less difference in the strengths of the steels irradiated at 390°C with different nickel contents than there was in the unirradiated condition.

After irradiation at 450°C, the 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels had similar strengths (Figs. 1 and 2). The strength of the irradiated 9Cr-1MoVNb steel was also similar to the strength before irradiation and after aging. However, the strength of the 9Cr-1MoVNb-2Ni steel showed a large decrease over the unirradiated strength. The strength of the 12 Cr steels showed similar effects (Figs. 4 and 5), with the irradiated strengths of the 12Cr-1MoVW and 12Cr-1MoVW-1Ni steels being similar to the aged strengths, and the steel with 2% Ni showing a significant decrease from the aged values.

Irradiated and aged steels were also compared at 550°C. Irradiation softened the 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels more than aging did (Figs. 1 and 2), and there was little difference between the strength of the 9-Cr steel with no nickel added and with 2% Ni. In general, the strengths of the 12-Cr steels with and without nickel showed similar trends, although there was somewhat more scatter in the data (Figs. 4 and 5).

Irradiation appeared to have only a minor effect on ductility (Figs. 3 and 6). The major effect was at 400°C, where the irradiation-induced strengthening led to a decrease in uniform and total elongation. The tensile behavior measured by the room-temperature tests was similar to that observed in the elevated-temperature tests.

4. Discussion

The unirradiated 9-Cr and 12-Cr steels with and without 2% Ni have been examined by TEM [3]. Both contained a high density of $M_{23}C_6$ precipitates and some fine MC particles. In Fig. 7, micrographs of extraction replicas and foil specimens for the 12Cr-1MoVW and 12Cr-1MoVW-2Ni steels are shown. The 9-Cr steels with and without nickel were similar. However, they contained more MC particles than were present in the 12-Cr steels, which is due to the niobium in these steels [3]. The primary difference between the microstructures of the standard steels and the steels with 2% Ni was that the 2%-Ni steel had a finer average precipitate size, a finer cell size, and a higher dislocation density. The differences in microstructure can be attributed to the different tempering treatments (1 h at 760°C and 2.5 h at 780°C for the standard 9 Cr and 12 Cr steels, respectively, compared to 5 h at 700°C for the two steels with 2% Ni). Furthermore, the higher strength for the unirradiated and unaged 2%-Ni steels over the standard steels can also be attributed to this difference in microstructure.

The relative effects of aging on mechanical properties also seem to be due to the difference in the tempering treatment. The loss of strength of the 2%-Ni steels when aged at 550°C results from additional tempering of the unstable microstructure of this steel, whereas the highly tempered standard steels and the 12Cr-1MoVW-1Ni steel are relatively stable for the given aging conditions. This would be consistent with the thermal aging bringing the strength of the 2%-Ni steels in line with the other steels. Such an explanation is reasonable, because nickel is not expected to have a significant effect on the strength [4].

The different relative changes in strength among the various irradiated steels can also be attributed to the microstructures. The large increase in strength of the standard 9-Cr and 12-Cr steels and the 12Cr-1MoVW-1Ni steel when irradiated at 390°C is similar to the change previously observed for other heats of 9Cr-1MoVNb and 12Cr-1MoVW steels irradiated to similar fluences in EBR-II [5,6]. The fact that the 2%-Ni steels showed little strength change during irradiation reflects the tempering differences of these steels relative to the standard steels. Strengthening by irradiation at 390°C has been attributed to the formation of irradiation-induced dislocation loops and precipitates [5–7]. It seems reasonable that similar strengthening effects would occur for the standard steels and the steel with 1% Ni. The lack of a significant strength change for the steels with 2% Ni may mean that, in these steels, strengthening caused by the formation of an irradiation-induced structure is offset by an irradiation-aided tempering, which advances the process started at 700°C.

These same heats of steel were previously irradiated in HFIR at 50°C, and irradiation caused an increase in strength for all of the steels. The 2%-Ni steels were strongest before irradiation and were also strongest after irradiation. For those experiments, hardening was concluded to be caused by irradiation-induced dislocation-loop formation. At the lower temperature, no precipitation was expected, and no irradiation-aided tempering was possible.

When the irradiated and aged steels were compared at 450°C, there was little effect of irradiation on the standard steels and the 12Cr-1MoVW-1Ni steel, but the irradiated steels with 2% Ni showed a large strength decrease compared to the aged specimens. The strength of the irradiated steels with 2% Ni had strengths comparable to the other steels after irradiation at 450°C. Irradiation at 550°C also resulted in similar strengths for all steels, regardless of the nickel content. The strength of the irradiated steels in this case fell slightly below the strengths of the thermally aged standard steels. (The 2%-Ni steels had a higher strength than the other steels after thermal aging.) These differences in the behavior of the 2%-Ni steels can also be attributed to the different tempering treatments before irradiation and the subsequent irradiation-aided tempering of the nickel-doped steels.

In the future, we will examine TEM disks that were irradiated under the conditions of the present experiment. However, even without the information derived from such studies, several interesting observations can be made.

Previous irradiation studies of 12Cr-1MoVW steel indicated that nickel-rich G-phase forms during irradiation in EBR-II at 390°C, and it was concluded that much of the strengthening observed was caused by this precipitate [7]. It was also concluded that the hardening caused by G-phase could lead to an irradiation-induced increase in the ductile-brittle transition temperature (DBTT). If this were true, then the 2%-Ni steels should show a much larger irradiation-hardening effect than the standard steels. Neither an enhanced strengthening as measured by the tensile properties nor a larger increase in the DBTT shift was observed in the steels with 2% Ni [8]. Future TEM studies will provide insight to the obeserved properties behavior.

As stated in the Introduction, the primary purpose for investigating the nickel-doped steels is to irradiate the steels in a mixed-spectrum reactor to produce both displacement damage and transmutation helium. However, to use this technique, it is necessary to show that nickel does not cause any effects that are not present in the standard alloys. The present results indicate that the nickel has not produced microstructural changes that affect the postirradiation tensile behavior of the 9-Cr and 12-Cr steels prior to irradiation or thermal aging. The original higher strength for the 2%-Ni steels is an effect of the different tempering treatments. When aged or irradiated, the steels with and without nickel approached similar strengths; this is consistent with the fact that nickel generally adds little solid-solution strengthening and apparently does not cause the formation of additional precipitate phases after thermal aging [4]. Although TEM on these steels is required, the tensile and impact results indicate that the use of the nickel-doped steels to simulate the simultaneous production of helium and displacement damage appears valid.

5. Summary

The standard 9Cr-1MoVNb and 12C-1MoVW ferritic (martensitic) steels with up to 2% Ni were irradiated in EBR-II to 15 to 16 dpa at 390, 450, 500, and 550°C. After irradiation, the strength and ductility did not depend on the nickel content. All indications are that the addition of nickel to these steels does not adversely affect the unirradiated or irradiated behavior. Therefore, it should be possible to use the nickel-doped steels to study helium effects on the properties of the irradiated steels.

References

[1] R. L. Klueh, J. M. Vitek, and M. L. Grossbeck, J. Nucl. Mater. 103&104 (1981) 887–92.

[2] R. L. Klueh, J. M. Vitek, and M. L. Grossbeck, in: Effects of Radiation on Materials: Eleventh Conference, Eds., H. R. Brager and J. S. Perrin, ASTM STP 762, (American Society for Testing and Materials, Philadelphia, 1982), p. 648.

[3] P. J. Maziasz, R. L. Klueh, and J. M. Vitek, J. Nucl. Mater., this volume.

[4] F. B. Pickering, Physical Metallurgy and the Design of Steels, (Applied Science Publishers, Ltd., London, 1978), p. 10.

[5] R. L. Klueh and J. M. Vitek, J. Nucl. Mater. 132 (1985) 27.

[6] R. L. Klueh and J. M. Vitek, J. Nucl. Mater. 137 (1985) 44.

[7] D. S. Gelles and L. E. Thomas, in: Ferritic Alloys for Use In Nuclear Energy Technologies, Eds. J. W. Davis and D. J. Michel (The Metallurgical Society of AIME, Warrendale, Pennsylvania, 1984), p. 559.

[8] W. R. Corwin, J. M. Vitek, and R. L. Klueh, in: ADIP Semiannu. Prog. Rep. Sept. 30, 1984, DOE/ER-0045/13 (U.S. DOE, Office of Fusion Energy, Washington, DC), p. 120.

List of Figures

Fig. 1. The 0.2% yield stress as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels. Tests were at the aging and irradiation temperatures.

Fig. 2. The ultimate tensile strength as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels. Tests were at the aging and irradiation temperatures.

Fig. 3. The uniform and total elongation as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels. Tests were at the aging and irradiation temperatures.

Fig. 4. The 0.2% yield stress as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels. Tests were at the aging and irradiation temperatures.

Fig. 5. The ultimate tensile strength as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels. Tests were at the aging and irradiation temperatures.

Fig. 6. The uniform and total elongation as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels. Tests were at the aging and irradiation temperatures.

Fig. 7. Transmission electron micrographs of normalized-and-tempered 12Cr-1MoVW steel (left) and 12Cr-1MoVW-2Ni steel (right).

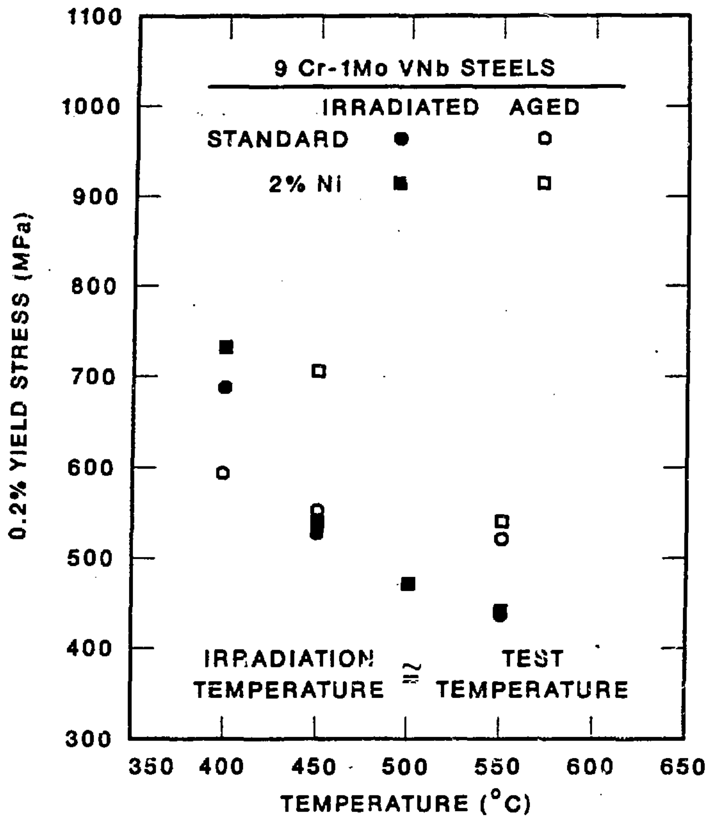

Fig. 1. The 0.2% yield stress as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels. Tests were at the aging and irradiation temperatures.

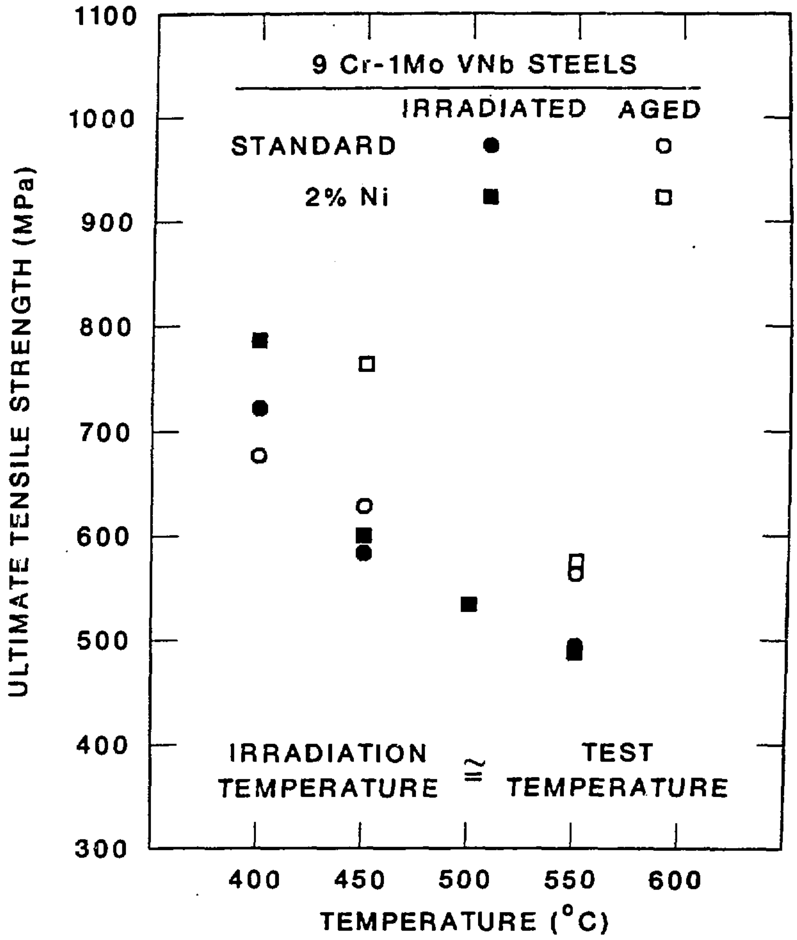

Fig. 2. The ultimate tensile strength as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels. Tests were at the aging and irradiation temperatures.

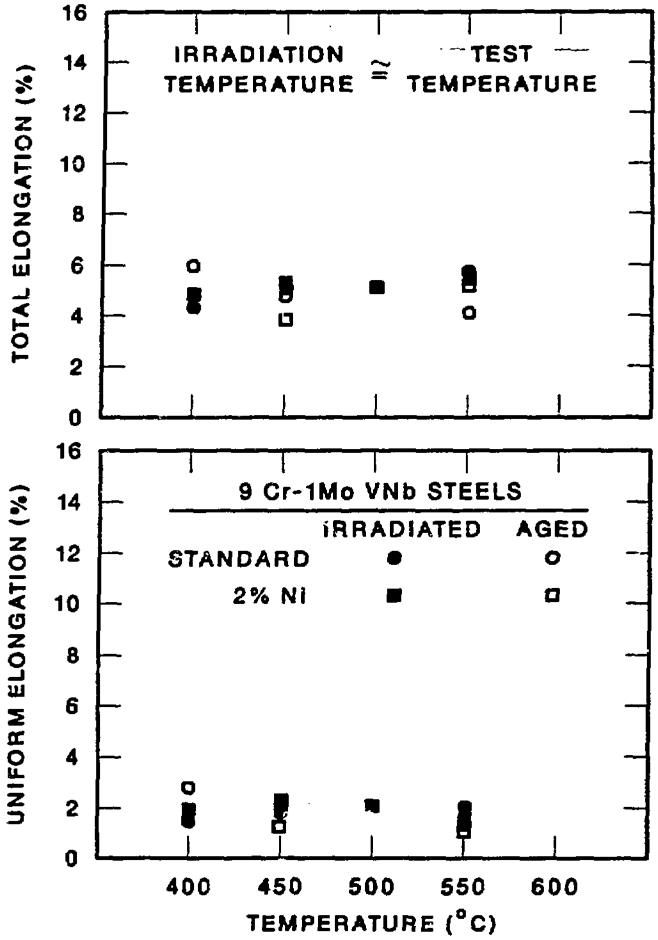

Fig. 3. The uniform and total elongation as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels. Tests were at the aging and irradiation temperatures.

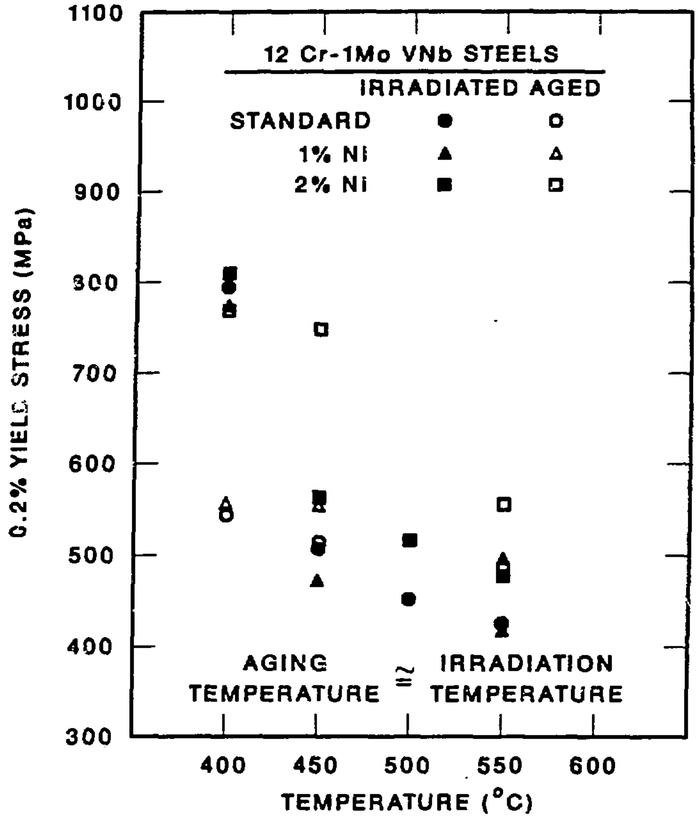

Fig. 4. The 0.2% yield stress as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels. Tests were at the aging and irradiation temperatures.

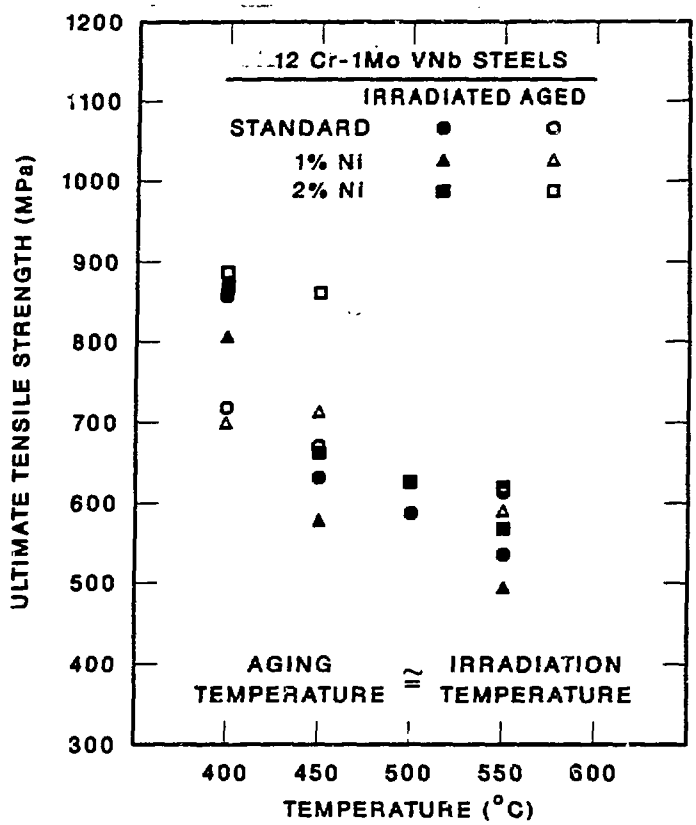

Fig. 5. The ultimate tensile strength as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels. Tests were at the aging and irradiation temperatures.

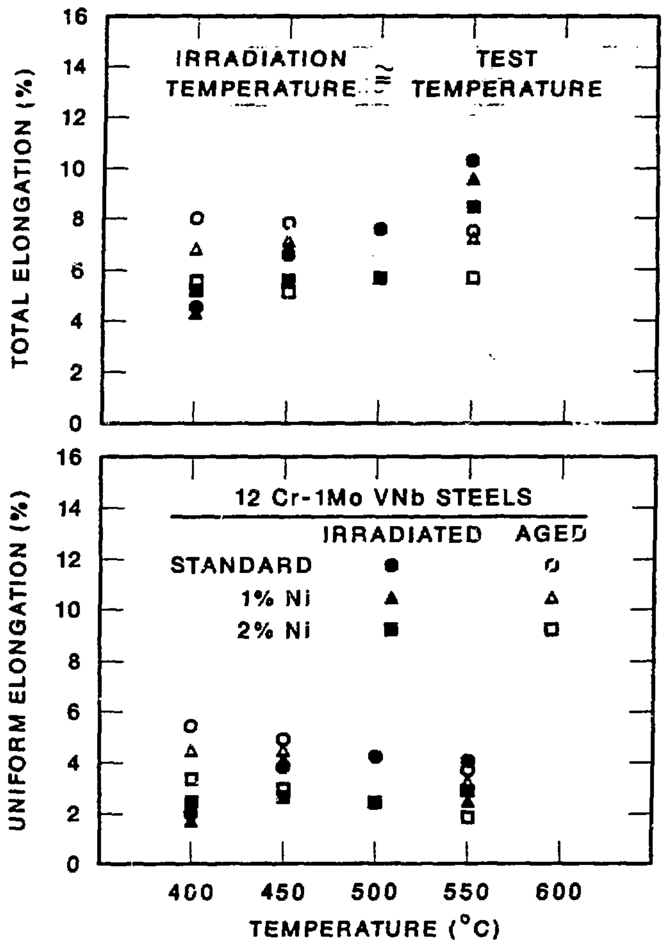

Fig. 6. The uniform and total elongation as a function of test temperature for thermally aged (5000 h) and irradiated (~16 dpa) 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels. Tests were at the aging and irradiation temperatures.

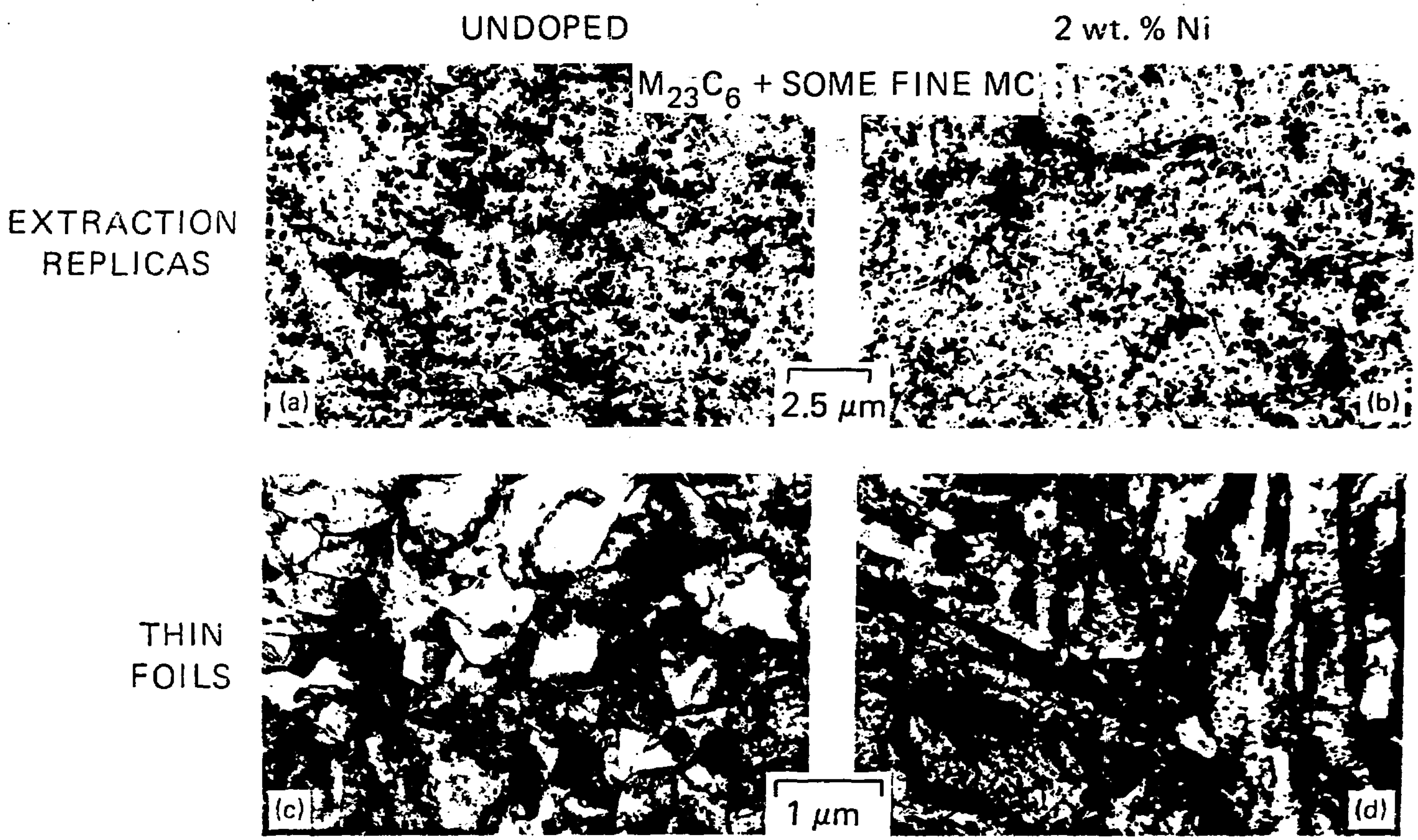

Fig. 7. Transmission electron micrographs of normalized-and-tempered 12Cr-1MoVW steel (left) and 12Cr-1MoVW-2Ni steel (right).