HELIUM EFFECTS ON NEUTRON-IRRADIATED Cr-Mo FERRITIC STEELS:

A REVIEW OF RECENT RESULTS*

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ABSTRACT: Large amounts of transmutation helium will be produced in the first wall of a fusion reactor by the high-energy neutrons from the fusion reaction. Since no fusion reactor is available, the effect of simultaneous helium production and displacement damage from neutron irradiation must be simulated. One method that has been used in ferritic steels is to add nickel to the steels and irradiate them in a mixed-spectrum reactor. In such reactors, the fast neutrons produce displacement damage, while helium is produced by a two-step reaction of ^{58}Ni with thermal neutrons. This technique has been used to investigate the effect of helium on swelling, tensile properties, impact properties, and elevated-temperature embrittlement. Results indicate that helium accelerates swelling and affects tensile and impact properties of Cr-Mo ferritic steels below $\sim 450^\circ\text{C}$. However, these steels are highly resistant to elevated-temperature helium embrittlement.

KEY WORDS: ferritic steels, helium effects, displacement damage, swelling, tensile properties, impact properties, irradiation damage, neutron irradiation.

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The irradiation resistance of ferritic (martensitic) steels, such as the 9Cr-1MoVNb and 12Cr-1MoVW steels, have made them candidates for fusion reactor applications. In the first wall of a fusion reactor, large amounts of transmutation helium will be produced by the high-energy neutrons (up to 14.1 MeV) released during the fusion reaction. The general effects of helium, especially on austenitic stainless steels, have been reviewed [1-5]. Until recently, however, little information was available on ferritic steels.

Because an adequate source of fusion neutrons does not exist, an alternative method must be used to simulate the effects of 14 MeV neutron irradiation. In alloys containing nickel, the effects of irradiation in fusion reactors can be simulated in mixed-spectrum fission reactors, where the spectrum contains both thermal and fast neutrons [6]. Helium is produced by the following two-step transmutation reaction of ^{58}Ni with thermal neutrons: $^{58}\text{Ni}(n,\gamma)^{59}\text{Ni}$ followed by $^{59}\text{Ni}(n,\alpha)^{56}\text{Fe}$. Helium production is accompanied by the formation of displacement damage by the fast neutrons of the spectrum.

Figure 1 shows the relationship between helium concentration and displacement damage for austenitic and ferritic steels irradiated in a fast reactor [e.g., the Experimental Breeder Reactor (EBR-II) or the Fast Flux Test Facility (FFTF)], in the mixed-spectrum High Flux Isotope Reactor (HFIR), and in the first wall of a tokamak reactor. If the 12Cr-1MoVW ferritic steel contains about 2% Ni and is irradiated in HFIR, the He:dpa ratio will approximate that expected in the first wall of a tokamak. Standard 12Cr-1MoVW steel contains ~0.5% Ni.

Over the past eight years, nickel doping has been used to investigate the effect of helium on swelling, tensile behavior, and impact properties of the 9Cr-1MoVNb and 12Cr-1MoVW steels [7-17]. The results from those studies will be reviewed in this paper.

Other techniques can be used to study the effect of helium. In one technique, ^{10}B is added to the steel, and helium is produced by the $^{10}\text{B}(n,\alpha)^7\text{Li}$ reaction [18]. Ion-beam irradiation has also been used [19]. However, ion-beam techniques require small specimens, which makes mechanical-properties studies difficult. Further, because of the large differences between ion bombardment and neutron irradiation (e.g., damage rate, damage volume, temperature shift, cascade size, spectrum, etc.), the ion-bombardment results cannot be used to predict behavior during neutron irradiation [19]. This review will concentrate on the results from neutron irradiations.

Experimental Procedure

Much of the discussion in this paper will involve studies on 9Cr-1MoVNb and 12Cr-1MoVW steels and these steels doped with 1% Ni (12Cr-1MoVW-1Ni) or 2% Ni (9Cr-1MoVNb-2Ni and 12Cr-1MoVW-2Ni). Chemical compositions for the heats of these steels that were used are given in Table 1. Note that the 12Cr steels contained approximately twice as much carbon as the 9Cr steels (0.2% vs 0.1%). Also given in Table 1 is the composition of DIN 1.4914, a German martensitic steel somewhat similar to the 12Cr-1MoVW steel, but with less molybdenum and carbon and some boron.

For all experiments, the steels were in the normalized-and-tempered condition. The normalizing treatment for the 9Cr steels was 0.5 h at 1040°C and for the 12Cr steels 0.5 h at 1050°C, after which they were cooled in air or flowing helium [8]. The 9Cr-1MoVNb was tempered 1 h at 760°C; the 12Cr-1MoVW and 12Cr-1MoVW-1Ni steels 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 produced by these heat treatments. Details on the heat treatment and on the optical microstructures have been published [8].

TEM studies were conducted to determine if nickel had an effect on the microstructures of the steels [13]. In the normalized-and-tempered condition, there were no basic differences between the tempered martensite of the nickel-doped and undoped steels: both contained an abundance of coarse $M_{23}C_6$ precipitates and many finer MC particles [13]. The steels with 2% Ni had a finer average precipitate size, a finer cell size, and a higher dislocation density than the standard steels [13]. These differences were attributed to the different tempering treatments. A lower tempering temperature was required for the steels with 2% Ni because nickel raises the A_{C_1} temperature [8].

Discussion

Swelling Studies

Neutron irradiation of Cr-Mo ferritic steels in fast reactors to fast neutron fluences of 17.6×10^{26} n/m² (80 dpa and <10 appm He) revealed that these steels swell much less than the commercial

austenitic stainless steels exposed to similar conditions, although significant amounts of irradiation-induced precipitation formed [20,21]. When standard 9Cr-1MoVNB (~0.2% Ni) and standard 12Cr-1MoVW (~0.5% Ni) steels were irradiated in HFIR up to 36 to 39 dpa and 30 and 100 appm He for the 9Cr-1MoVNB and 12Cr-1MoVW steels, respectively [10,11,13], more void formation was observed than after irradiation in fast reactors to much higher damage levels [20-22].

In both steels, void swelling, as estimated by TEM, was a maximum after irradiation at 400°C. Swelling at 400°C in the 9Cr-1MoVNB steel was about 0.2% [11,13] and in the 12Cr-1MoVW steel about 0.1% [10,13]. Voids (unstable, supercritical cavities) were nonuniformly distributed in patches. After irradiation at 300°C, a few fine helium bubbles (stable, subcritical cavities) were observed [13]. Irradiation at 500 and 600°C resulted in small bubbles forming preferentially at lath boundaries, prior austenite grain boundaries, and dislocations. When compared to results in fast reactors, where little helium was produced, the HFIR results indicated that helium assisted the nucleation of bubbles and voids [10,11,13].

To study the effect of higher helium concentrations on swelling, the nickel-doped steels were irradiated in HFIR to 36 to 39 dpa [13]. In the 9Cr-1MoVNB-2Ni steel containing ~410 appm He, a relatively uniform distribution of voids was observed after irradiation at 400 and 500°C. The number density was greater than that observed for the 9Cr-1MoVNB steel with 32 appm He, although the average void size was about the same. Void swelling in the 9Cr-1MoVNB-2Ni steel at 400°C was estimated to be 0.3 to 0.4% and at 500°C ~0.2%. At 500°C, voids

appeared inside coarse χ -phase particles. Significantly more and larger bubbles were produced on lath boundaries and dislocations at 500 and 600°C in the 9Cr-1MoVNb-2Ni steel than in the 9Cr-1MoVNb steel. In the 9Cr-1MoVNb-2Ni steel at 300°C, a high concentration of very fine bubbles was observed that was not present in the 9Cr-1MoVNb. Examples of the cavity structures in 9Cr-1MoVNb-2Ni steel are shown in Fig. 2 [13].

For comparison with the HFIR results, a 9Cr-1MoVNb-2Ni steel specimen irradiated in the FFTF at 400°C to 47 dpa and ~5 appm He was also examined. The FFTF-irradiated specimen contained the same precipitation that the HFIR-irradiated specimen contained, but there were no bubbles or voids (Fig. 3), indicating that the high-helium concentrations generated in HFIR were responsible for the bubbles and voids [13].

In the 12Cr-1MoVW-2Ni steel irradiated in HFIR to 39 dpa and 430 appm He, large numbers of bubbles were also observed at all irradiation temperatures and in much greater numbers than in the 12Cr-1MoVW steel [13]. As was true for the 9Cr-1MoVNb-2Ni steel, voids were observed only at 400 and 500°C. At 500°C, voids formed in coarse χ -phase particles that formed during irradiation, similar to the observation on 9Cr-1MoVNb-2Ni steel. In addition to the 12Cr-1MoVW and 12Cr-1MoVW-2Ni steels, a 12Cr-1MoVW-1Ni steel was also irradiated at 400°C. Swelling in the nickel-doped 12Cr steels appeared to be similar to or less than that of the undoped steel. This followed because the greater number of bubbles in the nickel-doped steels acted as sinks, and thus, a smaller percentage of bubbles in the nickel-doped

steels converted to voids than in the undoped steels. The subgrain structure of the 12Cr-1MoVW-2Ni steel in the normalized-and-tempered condition was smaller than that of the other two 12Cr steels, and there were fewer voids in the sample with small subgrains than for the samples with larger subgrains. This observation suggested that the subgrain boundaries in these steels could be acting as strong sinks, thereby hindering void formation [13].

Void formation was not an artifact caused by the nickel additions. This followed from the observation that there was no difference in the precipitate microstructures of 9Cr-1MoVNb-2Ni steel irradiated at 400°C in HFIR and FFTF (Fig. 3) [13]. Only the void structures differed, indicating that helium strongly enhances void formation in HFIR, and that the main reason for the absence of voids in FFTF was the very low helium generation rate [13]. The effect of irradiation on precipitation in these steels is discussed in another paper in this volume [23].

It was suggested [13] that these results may indicate that helium could shorten the incubation period for void swelling in the ferritic steels to lower displacement-damage levels than the extremely low values obtained from fast-breeder reactor data [20]. Since these studies [13] were for swelling conditions that were still in the incubation period, answers concerning the effect of helium on post-incubation, steady-state swelling will come only with longer irradiations.

Helium bubbles were also observed in the German martensitic steel DIN 1.4914 when it was irradiated at 590°C in the mixed-spectrum BR2 reactor in Belgium [24]. After irradiation, the steel contained about 90 appm He, and large numbers of small bubbles were observed on lath boundaries, prior austenite grain boundaries, and in the matrix [24].

Tensile Behavior

The effect of helium on the tensile properties of 9Cr-1MoVNB and 12Cr-1MoVW steels was investigated by irradiating the undoped and nickel-doped steels in HFIR [7,8,15,17] and EBR-II [12]. Little helium is produced in steels irradiated in EBR-II, and these specimens were used as baseline tests.

The 9Cr-1MoVNB, 9Cr-1MoVNB-2Ni, 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels were irradiated in EBR-II at 390, 450, 500, and 550°C to approximately 16 dpa (< 10 appm He), after which tensile tests were conducted at the irradiation temperature. The results were compared to normalized-and-tempered and thermally aged controls.

When irradiated at 390°C, the 9Cr-1MoVW, 12Cr-1MoVW, and 12Cr-1MoVW-1Ni hardened, but little change occurred in the steels with 2% Ni [12]. Therefore, after irradiation at 390°C there was much less difference in the strength of the steels, regardless of their nickel content. After irradiation at 450, 500, and 550°C, the strength of all of the steels decreased relative to the normalized-and-tempered and the aged controls. At each temperature, there was less difference in the strengths of the irradiated steels with different nickel content than there was before irradiation. The strength of the

9Cr-1MoVNb-2Ni and 12Cr-1MoVW-2Ni steels had decreased somewhat more than the strengths of the two standard steels and the 12Cr-1MoVW-1Ni steel.

Figure 4 shows the yield stress (YS) behavior for the two 9Cr steels. Similar behavior was observed for the ultimate tensile strength (UTS). The ductility behavior followed as expected from the strength behavior (i.e., an increase in strength was accompanied by a decrease in ductility and vice versa) [12].

It was concluded that the steels with 2% Ni showed little change in strength during irradiation at 390°C compared to the larger changes for the 9Cr-1MoVNb, 12Cr-1MoVW, and 12Cr-1MoVW-1Ni steels because of the different tempering temperatures used for the different steels (the steels with 2% Ni were tempered at a lower temperature and had a higher preirradiation strength) [12]. Strengthening by irradiation at 390°C has been attributed to the formation of irradiation-induced dislocation loops and precipitates [13,20]. This causes the strengthening in the two standard steels and the steel with 1% Ni. From the lack of a significant strength change for the steels with 2% Ni, it was concluded that, in these steels, strengthening caused by the formation of an irradiation-induced structure was offset by an irradiation-aided tempering that advanced the tempering process started at 700°C [12]. The larger decrease in strength of the steels with 2% Ni than for the other steels after irradiation at 450, 500, and 550°C was similarly explained (i.e., more tempering in the steels with 2% Ni). This approach to a similar strength level for steels with the different

nickel contents was taken as an indication that nickel has essentially no effect on the postirradiation strength of these steels in a fast reactor [12].

Tensile specimens of the steels with and without nickel additions were irradiated in HFIR to ~11 dpa to produce up to 103 appm He at 300, 400, and 500°C [15]. Specimens were tested at the irradiation temperature, and the results were compared with results from unirradiated, normalized-and-tempered specimens and results from thermally aged specimens. Thermal aging for 2500 h (approximately the time in reactor) caused little change in strength or ductility at 300, 400, and 500°C.

Figure 5 shows the changes in YS caused by irradiation in HFIR; changes were determined relative to the thermally aged specimens [15]. When irradiated at 300 and 400°C in HFIR, the YS of all the steels increased [15]. The strength changes at 300°C became greater as the nickel (helium) content increased. At 400°C, the effect of nickel was no longer systematic among the 12Cr steels, while for the 9Cr steels, the nickel-containing steel showed a larger increase in strength. At 500°C, irradiation produced little or no strengthening, and relative changes among the steels varied. Strength decreases occurred for the steels containing 2% Ni. UTS behavior was similar. There was no large effect on ductility at any temperature in HFIR.

Although the results of the HFIR study [15] were in general agreement with the EBR-II results [12] discussed above, there were differences that could be attributed to helium. The magnitude of the strength increase in HFIR at 300°C (Fig. 5) increased with nickel concentration, and therefore, with helium concentration. This was true for both 9Cr and 12Cr steels. Irradiation in HFIR at 400°C caused an increase in strength in all steels relative to aged control samples,

whereas the same steels with 2% Ni showed little change after irradiation to 16 dpa in EBR-II [12]. HFIR irradiation at 400°C produced increases in strength for the 12Cr-1MoVW and 12Cr-1MoVW-1Ni that were greater than those found after irradiation in EBR-II.

To explain the observations from HFIR at 300°C and the differences between the specimens irradiated at 400°C in EBR-II and HFIR, the possible role of helium was examined [15]. In the HFIR experiments at 300°C, the maximum amount of helium produced after 11 dpa, even in the nickel-doped steels, was close to the amount of helium obtained in the undoped steels irradiated in HFIR to 36 to 39 dpa, where TEM examination showed little or no evidence of voids or bubbles [13]. Even when the steel contained 2% Ni and over 400 appm He after 36 to 39 dpa, the bubbles were extremely small at 300°C (1 to 2 nm or less in diameter) [13]. Therefore, for the specimens irradiated at 300°C in HFIR to ~11 dpa, the helium would either be incorporated in extremely small bubbles below the resolution limit of TEM (1 to 2 nm) or remain trapped in vacancies in the matrix. There is thus far no evidence for additional precipitation occurring during HFIR irradiation at 300°C [13]. Increases in strength after irradiation at 300°C are therefore expected to be caused primarily by the formation of fine dislocation loops, which were observed in abundance [13]. The additional increment of strength increase for the higher helium-containing alloys (Fig. 5) could be due to hardening caused directly by either unresolved helium bubbles or helium-vacancy complexes. It could also be indirectly caused by helium trapped in vacancies interfering with recombination

of interstitials and vacancies, causing slightly more interstitial loop formation.

The principal cause of the increase in strength at 400°C appears to be a combination of precipitate and dislocation effects [15]. However, the increase in strength of the irradiated steels relative to the aged materials was significantly greater in HFIR irradiations than for the same steels irradiated to 16 dpa in EBR-II, which may be attributed to helium [15]. This difference is shown in Table 2. The strength increase was most pronounced in the 12Cr-1MoVW-2Ni steel, which produced the greatest amount of helium. TEM examination indicated that irradiation in HFIR at 400°C to 39 dpa produced network dislocations, a fine dispersion of M_6C precipitates, and an abundance of fine bubbles [13]. These effects should also contribute to the strengthening of the steels irradiated to 11 dpa, including the possible strengthening by bubbles with perhaps only a few helium atoms per bubble, although bubbles of this size could not be detected by TEM.

The fact that the 12Cr-1MoVW-2Ni steel did not show a strength increase at 400°C as large as the other steels was attributed to irradiation-enhanced tempering, which advanced the tempering process started during the normalizing-and-tempering treatment. That is, the irradiation-induced strength increase caused by precipitation, dislocation loops, and helium was offset by the tempering, which caused coarsening of the $M_{23}C_6$ and MC precipitates and the subgrain structure [13]. The 9Cr-1MoVNb-2Ni steel, however, did not show this same behavior; in fact, it showed the highest relative strength increase (Fig. 5). For the 39 dpa irradiation [13], the coarse $M_{23}C_6$ produced

by the preirradiation temper dissolved and finer and more abundant $M_{23}C_6$ particles precipitated with very fine M_6C particles as well [13]. By contrast, the 9Cr-1MoVNb showed no additional precipitation during irradiation at 400°C, while its as-tempered precipitate structure appeared to be partially dissolved [13].

Strength decreases were observed in the 500°C tests in the EBR-II experiment [12], which agree with observations in the HFIR experiment [15]. Furthermore, there was little difference in strength between steels with 0 and 2% Ni additions. At this temperature, therefore, little effect of the helium on strength was detected. As the dislocation density decreases and both the bubble and matrix precipitate distributions coarsen with increasing irradiation temperature, the contribution of these components to the strength of the alloys is decreased.

The nickel-doped steels were also irradiated in HFIR at ~50°C (the HFIR coolant temperature), and the effect of fluence (0 to ~25 dpa) and helium concentration (up to 327 dpa) was determined [7,8,17] by room-temperature tensile tests.

Results for the 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels indicated that the YS (Fig. 6) increased with increasing displacement damage. Above about 5 dpa, the rate of strength increase with fluence decreased as the damage level increased. Hardening increased with nickel content. The UTS shows a similar behavior.

At low fluences a strength increase was accompanied by a ductility decrease [17]. The total elongation showed indications of a

minimum after displacement-damage levels of 5 to 10 dpa. Above 15 dpa, all three steels had greater elongations than at 10 dpa [17].

Results for the 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels irradiated in HFIR at 50°C were similar to those for the three 12Cr steels [17].

There are indications that at higher temperatures ($>390^{\circ}\text{C}$), hardening or softening due to irradiation saturates in Cr-Mo steels [25] and in austenitic stainless steels [26]. These low-temperature results for the 9Cr and 12Cr steels indicated that irradiation caused the rate of increase in strength to decrease significantly with increasing fluence between about 5 and 10 dpa [17]. Although data were limited, the results indicated that after the rather rapid decrease in hardening rate beyond ~ 5 dpa, saturation had not yet occurred, and there was still a finite positive slope to the strength-displacement-damage curves. Even after ~ 25 dpa the strength was still increasing with fluence (Fig. 6) [17].

An effect of helium on low-temperature strengthening can be demonstrated by examining the YS-dpa relationship for the 12Cr-1MoVW and 12Cr-1MoVW-1Ni steels (Fig. 6) [17], which had similar strengths in the unirradiated condition. After irradiation to ~ 20 dpa, the YS of the 12Cr-1MoVW-1Ni steel increased about 100 MPa more than that for 12Cr-1MoVW steel. Although the sparse amount of data available at low fluences did not allow an accurate determination of when the YS of the 12Cr-1MoVW-1Ni steel became greater than that of the 12Cr-1MoVW steel, it was higher at 5 dpa, which corresponds to about 35 and 15 appm He for the 12Cr-1MoVW-1Ni and 12Cr-1MoVW, respectively.

Therefore, it was concluded that the difference between the 12Cr-1MoVW and 12Cr-1MoVW-1Ni steels after irradiation must be caused by the larger amounts of transmutation helium present in the 12Cr-1MoVW-1Ni steel [17]. A helium effect can also be deduced from the difference between the curves for the 12Cr-1MoVW-2Ni and 12Cr-1MoVW-1Ni. The YS of the 12Cr-1MoVW-2Ni steel was about 135 MPa above that for the 12Cr-1MoVW-1Ni steel in the unirradiated condition and about 175 MPa at 20 dpa (Fig. 6).

Based on the available data, the rate of increase in strength with dpa decreased abruptly somewhere around 5 dpa. This occurred for both the YS and the UTS for all of the steels [17]. By around 10 dpa, the slope of the strength-displacement-damage curves was much less than it was below 5 dpa. This observation was interpreted to mean that a saturation in strength due to displacement damage must be approached for the steels somewhere in the region where the slope of the strength-displacement-damage curves suddenly decreased (Fig. 6). The strength at which this decrease in slope occurred depended on the helium concentration: the higher the helium concentration, the higher the strength [17].

Mechanisms by which helium affects strength are only speculative [15,17], especially at low temperatures where there are few experimental data. When helium is in an interstitial position, it can readily diffuse through an alloy [27]. However, the interstitial helium becomes immobilized when trapped by a vacancy into a substitutional position. At temperatures below 100°C, detrapping can occur either

directly or by irradiation (by a neutron collision or by the knock-on process) [27]. At somewhat higher temperatures, detrapping can involve replacement by a self-interstitial, but at these low temperatures self-interstitials may be unable to recombine with vacancies containing trapped helium atoms. Helium-vacancy clusters can form when both the helium and vacancies are mobile at higher temperatures [28]. There are also indications that helium refines the scale of loop nucleation and prolongs loop stability to high fluences [29]. These results, however, have generally been obtained at higher temperatures than those of the experiments discussed here.

At temperatures below 100°C, helium-vacancy clusters will be immobile and continue to form as the helium concentration increases during irradiation. To the extent that this interferes with Frenkel pair recombination by mobile self-interstitials, either more self-interstitials will be present at any instant in time, or more self-interstitials will migrate and cluster to form and grow sinks (i.e., loops). This would constitute an effect that would cause additional hardening as well as be proportional to the buildup of helium. The excess self-interstitials would likely cause either more loop nucleation and/or growth relative to low He:dpa ratio irradiation, although experiments to verify this are very difficult to do. However, helium-enhanced loop formation and growth together with a buildup of helium-vacancy clusters in the matrix could logically give rise to additional strengthening that would be expected to eventually saturate.

Impact Properties

Irradiation can cause the ductile-brittle transition temperature (DBTT) of ferritic steels to increase by as much as 200°C [30]. To determine if helium may affect this process, miniature Charpy impact specimens of the nickel-doped and undoped 9Cr-1MoVNb and 12Cr-1MoVW steels were irradiated in HFIR at 50°C to up to 9 dpa [16]. As seen in Table 3, helium appeared to affect the impact properties for the 12Cr steels, where for a given dpa the increase in DBTT (Δ DBTT) increased as the helium concentration (nickel concentration) increased [16]. Also, the differences in the results for experiments RB2 and T3 on 12Cr-1MoVW-2Ni steel are noteworthy (Table 3). For the 12Cr-1MoVW-2Ni steel, the Δ DBTT for the tests from RB2 was 91°C and from T3 was 76°C. Both sets of specimens were irradiated to similar displacement-damage levels, but the specimens from RB2 contained 93 to 114 appm, while those in T3 contained 36 to 58 appm He. Further, the Δ DBTT for the 12Cr-1MoVW-2Ni irradiated in T3 was similar to that for the 12Cr-1MoVW-1Ni irradiated in RB2. Equivalent displacement-damage levels and helium concentrations were achieved for both materials under these test conditions. The DBTT shift for the 9Cr-1MoVNb-2Ni steel was also greater than that for the 9Cr-1MoVNb steel [16].

Although these data suggest a helium effect, relatively few data were collected and considerable scatter was inherent in the data. Only with further extensive testing can these preliminary data be verified.

If helium has an effect on impact properties at these low irradiation temperatures, the mechanism would be expected to be similar to

the effects on strength discussed above. This follows because the $\Delta DBTT$ is related to the increase in YS . A possible saturation of the $\Delta DBTT$ caused by displacement-damage effects has been discussed [30,31]. Although the helium effect would be expected to saturate, the observation that the yield stress increase saturates at a higher level when helium is present suggests that saturation might occur at a much larger $\Delta DBTT$ than observed to date in fast reactors where little helium is generated [14,32].

Helium Embrittlement

For irradiation temperatures $T_i > 0.6T_m$, where T_m is the absolute melting temperature, displacement damage is no longer stable, and flow properties are basically unaffected by irradiation. However, in certain alloys that contain helium, the ductility after irradiation decreases [1]. Intergranular fracture occurs, and the loss of ductility is thought to be caused by helium on grain boundaries [1]. For the austenitic stainless steels, this helium embrittlement can occur with only a few appm He — even the small amounts formed during fast-reactor irradiation [1].

To determine whether the 9Cr-1MoVNb and 12Cr-1MoVW steels might be susceptible to helium embrittlement, irradiated nickel-doped and undoped steels were tested [9]. After irradiation at 50°C to ~6 dpa to produce up to 49 appm He in the steels with 2% Ni, tensile tests were conducted at 700°C, a temperature where small amounts of helium can severely embrittle austenitic stainless steels [33]. Tests were

at a low strain rate ($4 \times 10^{-5} \text{ s}^{-1}$), since embrittlement becomes more severe as the strain rate is decreased [1].

The ductilities of the steels were essentially unaffected by irradiation. There was no apparent decrease in total elongation of either the 9Cr or 12Cr steels with up to 49 appm He. The lack of any effect of irradiation and helium on the ductility was quite evident when the tensile curves were compared: similar curves were obtained for the irradiated and unirradiated specimens [9].

For comparison, this irradiation experiment also contained specimens of 20% cold-worked type 316 stainless steel (316 SS) [9]. Because of the higher nickel content, the 316 SS contained ~230 appm He after irradiation. When tested at 700°C, the total elongation decreased from 8.5 to 0.3%.

Although the 316 SS contained considerably more helium than even the steels with 2% Ni, this alone could not explain the different helium effect. Previous work indicated that helium at levels as low as 1.3 to 3 appm caused the ductility of cold-worked 316 SS to decrease to only 0.8% when irradiated and tested at 700°C [33]. Solution-annealed steel showed a decrease from 49% to 9% under the same irradiation and test conditions.

Some information is available on a ferritic steel somewhat comparable to 9Cr-1MoVNb and 12Cr-1MoVW steels, namely, a 13Cr-2Mo-1W steel with and without 4% TiO_2 added for dispersion strengthening [34]. This steel had a duplex microstructure of martensite and δ -ferrite and was irradiated in the BR2 material testing reactor in Belgium, a mixed-spectrum reactor; after irradiation, specimens contained <10 appm He.

For specimens irradiated at 60 and 700°C and tensile tested at 700°C, a decrease in ductility was observed. However, the total elongations after irradiation were still greater than 10%, whereas the total elongation for solution-annealed 316 SS and the high-nickel austenitic Alloy 800 in the same experiment decreased from >70% to 2.5 and 0.6%, respectively [34].

The tensile results on the ferritic (martensitic) steels [9,34] indicate that they are highly resistant to elevated-temperature helium embrittlement relative to most other materials. The ferritic stainless steels types 405 and 430 showed a decrease in elongation when they were tested at 700°C following cyclotron injection by helium to ~40 appm He [35]. The total elongation of type 405 was decreased from 52 to 33%, whereas that of type 430 decreased from 89 to 48% [35]. However, in similar tests on type 316 stainless steel, a high-nickel alloy, Inconel X750, and a niobium-base alloy, Cb 752, final total elongations of 15, 2, and 2%, respectively, were observed after the addition of 40 appm He [35] (elongations before helium injections were 46, 8, and 12%, respectively). Vanadium alloys are also embrittled by helium [36].

The most severe conditions for helium embrittlement occur at the low strain rates of a creep test. In a creep experiment designed to compare the effect of helium embrittlement on an austenitic stainless steel and a martensitic steel under severe conditions, the martensitic steel was found to be much more resistant to embrittlement [37]. The German austenitic steel DIN 1.4970 (Fe-14.5Cr-15.0Ni-1.94Mn-0.1C) was irradiated at 600°C and creep tested at 650°C; the martensitic steel

DIN 1.4914 (Table 1), which has some similarities to the 9Cr-1MoVNB and 12Cr-1MoVW steels, was irradiated and creep tested at 600°C. Irradiation was in the mixed-spectrum BR2 reactor, and ~90 appm He was generated in both steels, primarily from the nickel and boron in the steels [37]. The austenitic steel was irradiated in the 15% cold-worked condition that had been aged for 2 or 23 h at 800°C, and the martensitic steel was in the normalized-and-tempered condition [37].

Although both steels showed a reduced rupture elongation, the results clearly indicated that the martensitic steel was much more resistant to helium embrittlement than the austenitic steel [37]. The rupture elongation of the irradiated austenitic steel was considerably less than for the unirradiated steel for all test conditions. As the strain rate decreased (rupture life increased), the ductility became progressively smaller, becoming <1% for rupture times $>10^4$ h. The unirradiated steel showed little change in ductility to 10^4 h [37], remaining around 10%.

The ductility of the irradiated martensitic steel was also less than that of the unirradiated steel [37]. However, the rupture elongation showed only a small decrease with decreasing strain rate (increasing rupture life). An elongation of about 5% was observed for a rupture life of $\sim 10^4$ h. Further, the rupture elongation for the unirradiated steel showed a much larger rate of decrease with rupture life than the irradiated steel. As a result, when the data were extrapolated to long rupture times, the curves appeared to merge, resulting in little difference in elongation of the irradiated and unirradiated steels near 10^4 h [37].

Creep experiments have also been conducted on 90- μm -thick specimens of the DIN 1.4914 martensitic steel that were implanted by α -particles in an accelerator [38,39]. Both in-beam and post-implantation experiments were conducted at 600°C on steels that contained 10 to 1000 appm He. Again, there was only a small loss of ductility, which was attributed to hardening by the implanted helium. Fractures were always transgranular, compared with those for type 316 SS tested under the same conditions, which failed intergranularly [38,39].

The reasons for the immunity of the martensitic steels to elevated-temperature helium embrittlement are not obvious. This immunity is not inherent in the body-centered-cubic crystal structure (compared to the face-centered-cubic structure of austenitic stainless steels), because helium embrittlement occurs in vanadium and niobium alloys above $0.5 T_m$ [35,36]. It appears more likely that this immunity is related to the microstructure of the martensitic steels.

In the normalized condition, the martensitic microstructure has a fine lath structure with each lath containing a high density of dislocations [40,41]. During tempering the dislocation density decreases, and a ferrite matrix containing carbide particles and a lower dislocation density results. However, the distinctive lath structure is still evident and consists of long laths grouped in packets with most laths separated by low-angle boundaries; the packets and some of the laths are separated by high-angle boundaries [40]. Prior austenite grain boundaries are also present.

This type of fine subgrain structure should allow helium atoms to disperse to the various boundaries, as well as distribute helium to the precipitate interfacial boundaries. Such a wide distribution of helium among many sites should effectively keep the helium concentration at any given high-angle grain boundary location relatively low. Various workers have shown that embrittlement in austenitic stainless steels is related to helium accumulation to form cavities along such boundaries [2,42,43]. The small amount of helium on high-angle boundaries in the ferritic steels reduces the probability of intergranular failure.

The large difference between the embrittlement of cold-worked and solution-annealed austenitic steel may indicate that some grain boundary migration (by recrystallization or grain growth) during irradiation or testing may be necessary to obtain extremely low elongations, such as those found in cold-worked 316 SS [9,33,34,38] and other austenitic stainless steels [34,37]. The difference between the cold-worked and solution-annealed 316 SS during HFIR irradiation, for example, has been explained by observations of grain boundaries "collecting" helium during recrystallization of the cold-worked structure [44]. Recrystallization also explains the observations on the relative embrittlement comparison of V-10Ti-10Nb, V-10Ti-20Nb, V-20Ti-10Nb, and V-3Ti-1Si alloys [36]. After irradiation, all but the last alloy showed a marked loss of ductility when tested at temperatures above $0.5 T_m$. It was also found that the V-3Ti-1Si alloy, which did not experience helium embrittlement, had a markedly higher recovery and recrystallization temperature than the other three high-titanium alloys. Evidence of

recrystallization was found to be associated with the fractures of the three high-titanium alloys.

If the above conclusions are correct, then we would expect a much larger effect of helium on pure alpha-iron or on a steel with a polygonal ferrite structure — as opposed to a martensitic or possibly a bainitic microstructure. This follows because the large number of subgrain boundary collection sites available in martensite or bainite are not present in pure iron. Furthermore, the grain boundaries in precipitate-free microstructures such as pure alpha-iron are more mobile at elevated temperatures. Indeed, this may partially explain the reduced ductility values observed on the dual-phase steel [34] and on the ferritic stainless steels [35]. Ferritic (martensitic) steels such as 9Cr-1MoVNb and 12Cr-1MoVW might be embrittled if they were irradiated in the cold-worked condition and recrystallized prior to or during testing; an alternative severe embrittlement test would be the recrystallization of the martensitic steel during elevated-temperature irradiation.

Summary and Conclusions

Helium generation during neutron irradiation of ferritic steels has been found to produce effects beyond those attributable to displacement damage alone. By irradiating nickel-doped and undoped 9Cr-1MoVNb and 12Cr-1MoVW steels in a mixed-spectrum reactor, the simultaneous effects of displacement damage and transmutation helium have been studied. The results generally indicate that helium has

less influence on the properties of Cr-Mo ferritic steels studied to date than on the properties of austenitic steels.

Void swelling is essentially absent in ferritic steels irradiated to almost 100 dpa in the absence of helium. When helium is produced simultaneously with the displacement damage, however, small amounts of void swelling are observed. Voids were produced at 400 and 500°C in 9Cr-1MoVNb and 12Cr-1MoVW steels irradiated to 39 dpa and containing up to 440 appm He. Helium bubbles were observed following irradiation at 300 to 600°C to 39 dpa.

Tensile studies on specimens irradiated at 50°C to ~25 dpa and ~327 appm He showed that, in addition to the strengthening caused by the displacement damage, a further increment of strength was caused by the helium. Helium also affected the hardening caused by irradiation at 300 and 400°C. Irradiation of Charpy impact specimens in HFIR at 50°C indicated that helium affected the increase in the DBTT.

Although the major concern for helium on the mechanical properties of austenitic stainless steels is elevated-temperature helium embrittlement, tests on ferritic steels indicate that they are quite resistant to this effect.

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TABLE 1--*Composition of 9Cr-1MoVNb and 12Cr-1MoVW Steel*

Element	Concentration, ^a wt %					DIN 1.4914
	9Cr-1MoVNb		12Cr-1MoVW			
	0% Ni ^b	2% Ni	0% Ni ^b	1% Ni	2% Ni	
C	0.09	0.064	0.21	0.20	0.20	0.11
Mn	0.36	0.36	0.50	0.47	0.49	0.35
P	0.008	0.008	0.011	0.010	0.011	...
S	0.004	0.004	0.004	0.004	0.004	...
Si	0.08	0.08	0.18	0.13	0.14	0.11
Ni	0.11	2.17	0.43	1.14	2.27	0.7
Cr	8.62	8.57	11.99	11.97	11.71	11.3
Mo	0.98	0.98	0.93	1.04	1.02	0.5
V	0.209	0.222	0.27	0.31	0.31	0.3
Nb	0.063	0.066	0.018	0.015	0.015	0.25
Ti	0.002	0.002	0.003	0.003	0.003	...
Co	0.013	0.015	0.017	0.015	0.021	...
Cu	0.03	0.04	0.05	0.05	0.05	...
Al	0.013	0.015	0.030	0.017	0.028	...
B	<0.001	<0.001	<0.001	<0.001	<0.001	0.007
W	0.01	0.01	0.54	0.53	0.54	...
As	<0.001	<0.001	<0.001	0.002	<0.002	...
Sn	0.003	0.003	0.002	0.001	0.002	...
Zr	<0.001	<0.001	<0.001	<0.001	<0.001	...
N	0.050	0.053	0.020	0.016	0.017	...
O	0.007	0.006	0.005	0.007	0.007	...

^aBalance iron.^bStandard steel with no nickel added.

TABLE 2--*Strength change of irradiated steels relative to
thermally aged steels for irradiation
in HFIR and EBR-II at 400°C*

	<u>12Cr-1MoVW</u>		<u>12Cr-1MoVW-1Ni</u>		<u>12Cr-1MoVW-2Ni</u>		<u>9Cr-1MoVNB^a</u>	
	YS	UTS	YS	UTS	YS	UTS	YS	UTS
HFIR	359	227	365	248	263	171	274	218
EBR-II	249	138	215	107	38	-18	93	43

^aNo 9Cr-1MoVNB-2Ni steel was irradiated at 400°C in EBR-II.

TABLE 3--Charpy impact properties of unirradiated
and irradiated^a 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and
12Cr-1MoVW-2Ni steels

Steel	Experiment	Conditions dpa (appm He)	DBTT ^b (°C)	ΔDBTT (°C)	USE (J)
12Cr-1MoVW	CC	Unirradiated	-35	..	26
	RB1	4.3 (15)	-33	2	20
	RB2	7.0-9.0 (30-39)	-2	31	25
	T3	6.0-9.0 (11-17)	3	36	24
12Cr-1MoVW-1Ni	CC	Unirradiated	-37	..	23
	RB1	3.9 (26)	30	67	20
	RB2	8.2 (66)	37	74	23
12Cr-1MoVW-2Ni	CC	Unirradiated	-25	..	17
	RB1	4.0-4.5 (50-55)	37	62	11
	RB2	6.0-7.3 (94-115)	66	91	13
	T3	5.7-9.1 (36-58)	51	76	15

^aIrradiation was at 50°C.

^bMeasured at 9.2 J on miniature (½-size) Charpy specimens.

^cUnirradiated control specimens.

LIST OF FIGURES

FIG. 1--*Relationship between helium concentration and displacement damage for austenitic and ferritic steels irradiated in HFIR, EBR-II, and the first wall of a fusion reactor.*

FIG. 2--*Microstructures of 9Cr-1MoVNb-2Ni steel irradiated at 37 to 39 dpa and 410 appm He at (a) 300°C, (b) 400°C, (c) 500°C, and (d) 600°C.*

FIG. 3--*A comparison of the microstructures of 9Cr-1MoVNb-2Ni steel irradiated in (a) FFTF at 407°C to ~47 dpa and 5 appm He and (b) HFIR at 400°C to ~37 dpa and 410 appm He. Precipitate and dislocation microstructures are similar, but void formation increases with helium content.*

Fig. 4--*A comparison of 0.2% yield stress as a function of test temperature for 9Cr-1MoVNb and 9Cr-1MoVNb-2Ni steels as thermally aged (5000 h) and as irradiated in EBR-II to ~16 dpa. Tests were at the aging and irradiation temperatures.*

FIG. 5--*Change in 0.2% yield stress caused by irradiation of ferritic steels in HFIR; the change was measured relative to the thermally aged specimens.*

FIG. 6--*The 0.2% yield stress of 12Cr-1MoVW, 12Cr-1MoVW-1Ni, and 12Cr-1MoVW-2Ni steels as a function of fluence for low-temperature irradiation in HFIR.*

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