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Pros and cons of nickel- and boron-doping to study helium effects in ferritic/martensitic steels

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Abstract

In the absence of a 14 MeV neutron source, the effect of helium on structural materials for fusion must be simulated using fission reactors. Helium effects in ferritic/martensitic steels have been studied by adding nickel and boron and irradiating in a mixed-spectrum reactor. Although the nickel- and boron-doping techniques have limitations and difficulties to estimate helium effects on the ferritic/martensitic steels, past irradiation experiments using these techniques have demonstrated similar effects on the swelling and Charpy impact properties that are indicative of a helium effect. Although both techniques have disadvantages, it should be possible to plan experiments using the nickel- and boron-doping techniques to develop an understanding of the effects of helium on mechanical properties.

1. Introduction

Unlike the development of materials for fission reactors where test facilities with potential operating conditions were available, the testing of materials for a fusion reactor first wall must proceed without the availability of the 14 MeV neutrons that will be present in a fusion facility. Therefore, radiation effects on structural materials for fusion applications must be simulated with fission neutrons or ion irradiation. Displacement damage effects can be studied satisfactorily with fission irradiation. However, because of different neutron spectra in fission and fusion, transmutation product production in materials differ in the two environments. Large amounts of transmutation helium form in the fusion neutron environment, and it is necessary to understand the effect of the simultaneous formation of displacement damage and large amounts of helium on the properties of a potential structural material.

To study the simultaneous effect of neutron displacement damage and helium effects on ferritic/martensitic steels, two techniques that have been used are the addition of either nickel or boron to the steels that are then irradiated in a mixed-spectrum fission reactor, such as the high flux isotope reactor (HFIR) or the high flux reactor (HFR). This paper will summarize observations made using these techniques and examine the advantages and disadvantages (shortcomings) of the techniques to assess their appropriate use in future work.

2. Nickel doping studies

Helium forms from ^{58}Ni during irradiation in a mixed-spectrum reactor by the following two-step reaction with thermal neutrons: ^{58}Ni (n, γ) ^{59}Ni (n, α) ^{56}Fe. Natural nickel contains 68% ^{58}Ni, and steel with up to 2% natural nickel has been used in HFIR irradiations because that concentration gives a He:dpa ratio similar to that in a first wall of a DT tokamak reactor [1].

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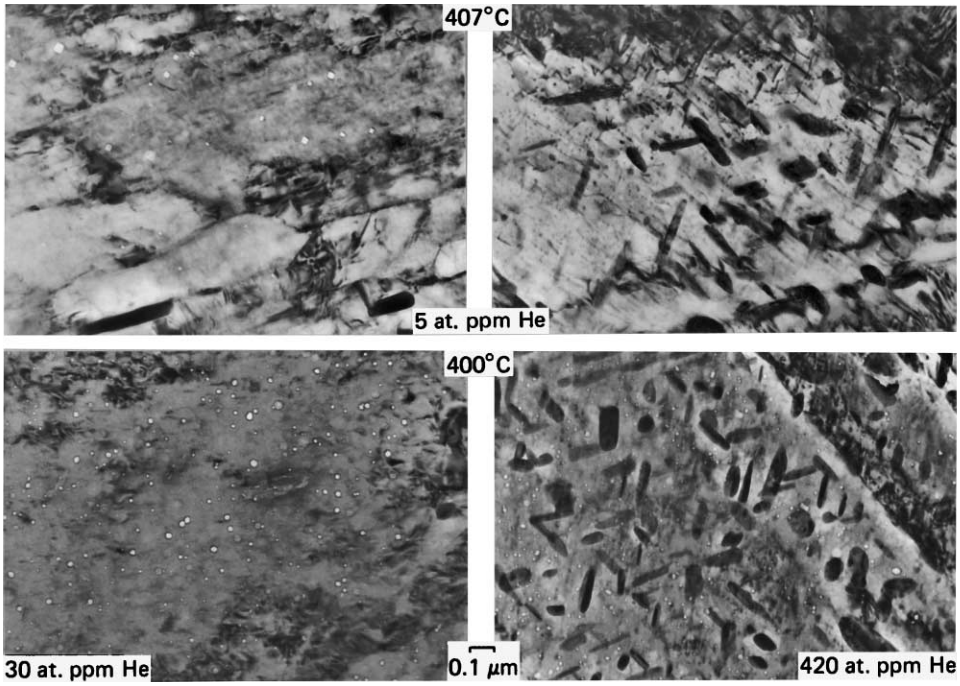

Fig. 1. Microstructure of 9Cr–1MoVNb (left) and 9Cr–1MoVNb–2Ni (right) steels irradiated in FFTF at 407 °C to 47 dpa (top) and in HFIR at 400 °C to 37 dpa (bottom) showing increased void formation after HFIR irradiation; the micrographs on the right indicate little difference in the precipitate microstructure of the 9Cr–1MoVNb–2Ni after irradiation in FFTF (top) and HFIR (bottom) [5].

2.1. Microstructure: swelling and precipitation

The first studies on nickel-doped steels were on the modified 9Cr–1Mo (9Cr–1MoVNb) and Sandvik HT9 (12Cr–1MoVW) steels to determine the effect of helium on swelling and to determine the effect of nickel on the microstructure before and after irradiation [2–5]. The nickel did not have a large effect on the martensite microstructure, although the 2% Ni addition lowered the A_{C1} temperature to around 720 °C [6]. This meant the steel containing 2% Ni could not be tempered at the standard tempering temperatures for 9Cr–1MoVNb (760 °C) and 12Cr–1MoVW (780 °C) steels. A 700 °C tempering temperature (for a longer time) was chosen [2–6], which meant that although the same precipitates (primarily $M_{23}C_6$ and MX) formed in the steels with and without nickel, the morphologies of the precipitates (especially the average size of the $M_{23}C_6$) were slightly different [7]. In addition, recent work indicated that when F82H with and without nickel was given similar heat treatments, the steel with nickel had a slightly smaller lath size [8].

After irradiating the 9Cr–1MoVNb and 9Cr–1MoVNb–Ni steels at 400 °C in HFIR to ≈38 dpa, swelling increased with increasing helium (nickel) as shown in Fig. 1 (bottom micrographs) [2–5]. When these steels were irradiated at 407 °C to a comparable fluence (≈46 dpa) in the fast flux test facility (FFTF), where little helium formed, very little swelling occurred (Fig. 1, top micrographs) [4]. Irradiation of the nickel-doped steel produced a somewhat different precipitate structure than in steel without nickel. The nickel-containing steel contained a high number density of nickel-enriched M_6C particles [4,5]. However, as seen in Fig. 1 (micrographs on the right), the microstructures generated in HFIR and FFTF after similar irradiations were similar (except for the cavities), indicating that the increased helium generation in HFIR must cause the higher swelling in the steel irradiated in that reactor.

For irradiations of 9Cr–1MoVNb and 9Cr–2WVTa (Fe–9Cr–2W–0.25V–0.07Ta–0.1C) steels with and without 2% Ni to ≈12 dpa at 400 °C in HFIR, both steels with the higher helium (nickel) concentration showed higher swelling [8]. A higher number density of dislocation loops formed in the nickel-containing steels. Before irradiation, all four steels contained similar precipitates, mainly $M_{23}C_6$ with a few MC particles. After irradiation, no change in tempering-produced precipitates occurred in the steels without nickel, but irradiation-induced precipitates formed in the nickel-containing steels. The 9Cr–1MoVNb–2Ni contained a low number density of large M_2X type (a chromium- and nitrogen rich Cr_2N precipitate) particles (5×10^{20} m^{-3}, mean size ≈ 54 nm) and the 9Cr–2WVTa–2Ni contained a high density of small of M_6C (2×10^{21} m^{-3}, mean size $\approx$ 7 nm). The M_6C precipitate agrees with the results discussed above for the nickel-containing steels, including 9Cr–1MoVNb–2Ni [8]. The presence of M_2X type precipitate instead of M_6C in the latter irradiation experiment may have been due to the nitrogen in the 9Cr–1MoVNb–2Ni along with a lower fluence in the latter experiment.

Gelles irradiated Fe–12Cr alloys containing 0% Ni, 1.5% natural nickel, 1.5% ^{59}Ni, and 1.5% ^{60}Ni to $\approx$7 dpa in HFIR at 300 and 400 °C [9]. About 45 appm He was produced from the natural nickel, 80 appm He from ^{59}Ni, and no helium from ^{60}Ni. A high density of helium bubbles formed in the alloys with natural nickel and ^{59}Ni irradiated at 300 °C. Lower cavity densities were observed in the alloys with 0% Ni and 1.5% ^{60}Ni additions, indicating that helium promoted swelling. At 400 °C, an unidentified precipitate formed in the nickel-containing alloys that hindered the observation of bubbles, while the alloy without nickel contained some cavities [9]. From these past studies, it is concluded that nickel doping of ferritic/martensitic steels can play a role in precipitate development during irradiation, but that the helium generated from the nickel affects the swelling behavior.

2.2. Tensile properties

Several attempts have been made to examine the effect of helium on tensile properties (strain rates of $\simeq 10^{-3}$ s^{-1}) using the 9Cr–1MoVNb and 12Cr–1MoVW steels with and without nickel additions [10–12]. Irradiations were in HFIR at 300–600 °C, where helium was generated in the nickel-containing steels, and at 390–550 °C in EBR-II, a fast reactor where little helium formed with or without nickel. At temperatures above $\approx$425 °C where no hardening occurs in the steels without the nickel additions, no hardening was observed in the nickel-doped steels either. Under conditions where hardening occurs (<425 °C), no effect of helium could be definitively established, nor was there any evidence of a change in fracture mode for the specimens with high-helium concentrations [10–12].

Uncertainty exists on the use of nickel doping for low-temperature irradiations. Kasada et al. [13] irradiated a 9Cr–2W reduced-activation steel with and without 1% Ni in JMTR to 0.15 dpa at 170 and 220 °C. After irradiation at 170 °C, an increase in the room temperature yield stress of up to 350 MPa was observed for the nickel-containing steel, compared to a 120 MPa increase for the steel without 1% Ni. No difference in strength increases was observed for the steels with and without nickel when the steels were irradiated at 220 °C. These steels were also irradiated to 2.2 and 3.8 dpa at 270 and 348 °C, respectively, in the Advanced Test Reactor, and the nickel-containing steel hardened about 20% more than the steel without nickel at 270 °C, but strengths were similar after the irradiation at 348 °C. TEM analysis indicated that nickel refined the size of the defect clusters, which were more numerous in the nickel-containing steel [14].

The conclusions from these results are that there is no discernable hardening effect due to helium or nickel at 300 and 400 °C, but at temperatures below $\approx$300 °C, hardening due to nickel may interfere with attempts to determine the effects of helium.

2.3. Charpy impact properties

After irradiation of the 9Cr–1MoVNb and 12Cr–1MoVW steels in EBR-II to 13 and 26 dpa at 390 °C, where little helium formed, the increase in DBTT (ΔDBTT) saturated with fluence at $\approx$54 and $\approx$144 °C, respectively [15]. These same materials were irradiated in the HFIR at 400 °C to $\approx$40 dpa which produced $\approx$30 appm He in the 9Cr–1MoVNb steel and $\approx$110 appm He in the 12Cr–1MoVW. The ΔDBTT was significantly higher than after irradiation in EBR-II to 13 and 26 dpa (Fig. 2) [17], 204 and 242 °C in the 9Cr–1MoVNb and12Cr–1MoVW steels, respectively. The difference was taken to indicate that the saturation in ΔDBTT observed in the EBR-II at 390 °C did not apply to the HFIR irradiation. Further, when the 12Cr–1MoVW–2Ni and 9Cr–1MoVNb–2Ni were irradiated in HFIR at 400 °C to $\approx$40 dpa and $\approx$370 appm He, the ΔDBTTs were >325 °C, which was considerably above the values for the steels without the nickel additions, and also greater than the value (90 °C) obtained for 12Cr–

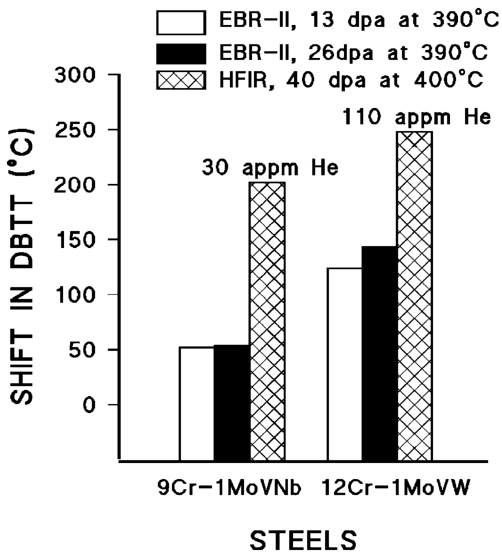

Fig. 2. A comparison of the shift in DBTT for 9Cr–1MoVNb (modified 9Cr–1Mo) and 12Cr–1MoVW (HT9) steels irradiated in EBR-II to 13 and 26 dpa and in HFIR to 40 dpa.

1MoVW–2Ni irradiated in EBR-II at 390 °C. Examination of fractured tensile specimens of 9Cr–1MoVNb–2Ni and 12Cr–1MoVW–2Ni irradiated to 74 dpa in HFIR provided an indication of intergranular fracture. Irradiation at 300 °C in HFIR indicated an effect of helium when the Charpy properties of the steels with 2% Ni were compared to those with no added nickel (no comparison with fast reactor irradiation was possible because of the low irradiation temperature) [16].

The conclusion from those results was that helium affects embrittlement [16] with the helium effect being in addition to the other effect caused by the hardening from dislocation loops and irradiation-enhanced precipitation. Because excess hardening by helium, as measured in tensile tests, was not sufficient to completely explain these observations, it was proposed that helium decreases the fracture stress, perhaps by promoting intergranular fracture, although this needs to be verified. Mechanisms by which this could occur have been proposed [16], but that subject is beyond the scope of the present discussion.

Alternative explanations to a helium effect have been considered but rejected [16]. The high number density of small irradiation-induced precipitates (M_6C, etc.) that were discussed above has most often been cited as possibly playing a role in these properties changes [15,17]. However, the same precipitates in similar amounts to that formed in the mixed spectrum of HFIR at 400 °C also formed during fast reactor irradiation in FFTF at 407 °C (Fig. 1) [16,18]. Since the ΔDBTT was much smaller after the fast reactor irradiation than the mixed-spectrum irradiation, the extra embrittlement could not be attributed to the precipitates. Likewise, no excess hardening could be attributed to those precipitates in tensile studies [14,15].

3. Boron doping studies

When boron-containing steels are irradiated, helium forms from the ^{10}B (n, α) ^{7}Li reaction. The cross section for this (n, α) reaction is greatest for low-energy neutrons, which means that irradiation in a mixed-spectrum reactor such as HFIR will result in all of the ^{10}B being reacted within ≃1 dpa, compared to a much longer time in a fast reactor. The energies of the lithium ion and the α-particle (0.87 and 1.53 MeV, respectively) produced by the (n, α) reaction create a significant number of atomic displacements as they slow down to thermal energy, and these additional displacements could cause an increase in the local defect concentration. Boron is highly reactive, so it segregates to interfaces and may be present in precipitate particles, such as B_4C and $M_{23}(CB)_6$ [19]. When the boron is contained in precipitates, neutron irradiation can lead to the cavities forming as halos around the boron-containing precipitate particles, with the size of the halos being characteristic of the energy of the α-particles released by the (n, α) reaction [19]. It has also been found that boron can affect the prior-austenite grain size, thus making it difficult to achieve a similar grain size for alloys with different boron concentrations given the same heat-treatments [20].

3.1. Microstructure: swelling and precipitation

Swelling was investigated in F82H steel containing 4–8 (F82H-Std) and 60 (F82H-2) ppm natural boron and 58 ppm ^{10}B (F82H-3) irradiated at 400 °C in HFIR to ≈7, 26, and 51 dpa [21,22]. For the 51 dpa irradiation, this produced ≈6, 62, and 325 appm He for F82H-Std, F82H-2, and F82H-3, respectively. The amount of swelling and the number density of cavities increased with helium concentration (Fig. 3), although the swelling of F82H-2 (1.2%) was slightly larger than F82H-3 (1.1%) because the high density of smaller cavities in F82H-3 could act as neutral sinks for both vacancies and interstitials. For all irradiation conditions, the cavities were relatively uniformly distributed on dislocations and in the matrix, with relatively few appearing on the lath boundaries.

For a 9Cr–2W–0.25V–0.05Ta–0.1C (9Cr–2WVTa) reduced-activation steel with and without 0.003% B

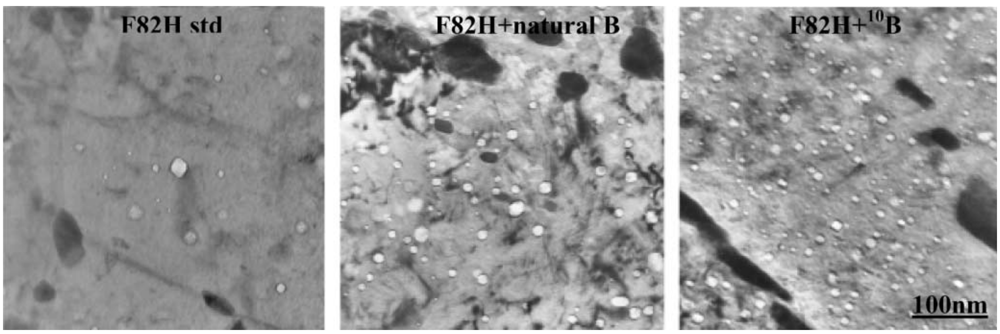

Fig. 3. TEM micrographs showing the difference in swelling of F82H that contain different boron content.

irradiated in the FFTF, the steel with boron showed as much as 0.2% swelling after irradiation to 40 dpa (30 appm He) at 420 °C, compared with negligible swelling for the steel without boron [23]. This observation was attributed to the higher swelling rate in the higher helium concentration in the boron-containing steel. In another FFTF irradiation [24], two 9Cr–2WVTa steels containing 0.02% Ti, one with a 0.003% B addition, and one without were irradiated to 60 dpa at 420 °C. The mean void size in the boron-containing steel with 30 appm He was much smaller than for the voids in the steel without boron, but the void number density in the steel with boron was about twice that of the steel without boron. Less swelling occurred in the steel with boron (0.45% vs. 0.77%) because the high density of smaller bubbles in this steel acted as neutral sinks (no bias for either vacancies or interstitials) [24].

3.2. Tensile properties

Tensile properties of the F82H-Std, F82H-2, and F82H-3 were determined after irradiation in HFIR at 400 and 500 °C to fluences between 11 and 34 dpa [25,26]. Tests at the irradiation temperature indicated that any effect of helium on strength or ductility was small, and that the differences generally fell within the scatter of the data. The F82H-Std and F82H-3 steels were also irradiated in the Japan materials test reactor (JMTR) to 0.8 dpa at 250–265 °C [26,27]. For tensile tests over the range from room temperature to 400 °C, irradiation hardening for the two steels was similar, with boron-doped (F82H-3) steel showing a slightly lower strength. However, the steel with the higher helium concentration had a considerably lower total elongation and reduction of area [27].

3.3. Charpy impact properties

After irradiation of ^{10}B-doped specimens and undoped specimens at 355–375 °C to 0.3–0.5 dpa in JMTR and the Japan Research Reactor (JRR-2) to produce 300 appm He in the doped F82H, the DBTT in the doped steel was ≈15 °C higher than in the undoped steel [26]. No shift in transition temperature was observed when irradiated at 500–590 °C. A larger effect using ^{10}B-doping was obtained for F82H irradiated at a lower temperature (260–360 °C) to 0.3–0.6 dpa in JMTR [27]. Although a complete Charpy curve was not determined (because of temperature limitations of the Charpy test rig) for the ^{10}B-doped steel with 100 appm He, the ΔDBTT appeared to be well above room temperature (Fig. 4) and much larger than for the standard F82H irradiated similarly.

In another experiment, the reduced-activation steel JLF-1 (a 9Cr–2WVTa steel) and this steel with 0.0022% B was irradiated in the HFR mixed-spectrum reactor to

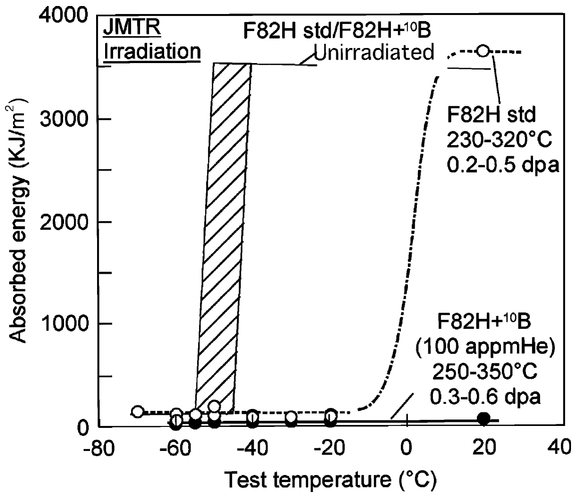

Fig. 4. Charpy impact data for standard F82H and ^{10}B-doped F82H before and after irradiation at 250–350 °C in JMTR [27].

2.5 dpa at 300 °C [28]. Before irradiation, the steels had similar Charpy curves. After irradiation, the DBTT of the boron-containing steel with ≈23 appm He was about 70 °C higher than for the steel without the boron addition.

The relative ΔDBTTs obtained from irradiation to 0.8 and 2.4 dpa at 250–450 °C in HFR of four reduced-activation steels, OPTIFER Ia, OPTIFER II, F82H, and ORNL 9Cr–2WVTa, and two conventional Cr–Mo steels, MANET I, MANET II, was attributed to the different levels of helium in each of these steels due to the different levels of boron contained in each steel [29,30]. It was observed that the higher the boron content, the higher the ΔDBTT for the individual steels. Maximum helium in the steels was calculated as 85, 70, 60, 60, <20, and <10 appm for MANET I, MANET II, OPTIFER Ia, OPTIFER II, F82H, and ORNL 9Cr–2WVTa, respectively. The ΔDBTTs for the steels scaled with the boron content and, therefore, the helium content; the ratio of ΔDBTT to helium was essentially the same for all of the steels at ≈2–3 °C/appm He [29,30].

4. Discussion

Despite some complicating issues with nickel and boron doping of ferritic/martensitic steels to study helium effects, the above review of past work indicates the doping technique has produced results that indicate helium does affect the impact properties. Furthermore, without a 14 MeV neutron source, these doping techniques are essentially the only tools available for such studies. Other techniques that have been proposed or used are helium implantation in a cyclotron [31] and replacing the natural iron in the steel with ^{54}Fe [32].

Cyclotron irradiation has been used, and a shift in DBTT similar to that observed in the nickel- and boron-doping experiments was observed and attributed to helium [31]. The problem with cyclotron implantation is that it is difficult to use to implant the helium in mechanical properties specimens because of the limitation of their size and expense. Irradiation of 54_{Fe} produces helium in a (n, α) reaction, and it appears to be an ideal technique, since the composition of the steel is essentially unchanged. Unfortunately, ^{54}Fe is also very expensive (about $20 000/g), which makes the cost of conventional (even miniature) mechanical properties specimens prohibitively expensive.

Given that the doping techniques are the primary tools available to study helium effects, it is necessary to determine how they may best be used to obtain the information required. The question is: How can single variable tests best be carried out with these techniques? The primary problems with the doping techniques involve the effect of the dopant as an alloying addition on the properties and microstructure of the steel.

From the data, the nickel-doping technique should not be used below about 300 °C. Although the precipitates in the nickel-doped steels are different from those in the undoped steels, the fact that similar microstructures are obtained in fast and mixed-spectra reactors for irradiations at 400 °C while the swelling and mechanical properties behavior differ in the two conditions appears to indicate that the effect of helium can be examined. Changes in microstructure in boron-doped steels are less obvious than in the nickel-doped steels, but the effect on prior-austenite grain size, possible segregation to grain boundaries and precipitates, and the formation of lithium from the (n, α) reaction must be kept in mind when evaluating the data.

One approach that will help minimize such uncertainty is to use isotopes of the dopants instead of natural nickel and boron. This lowers the amount of the dopant required for a given helium level. Helium is produced from the ^{58}Ni and ^{10}B and not from ^{60}Ni and ^{11}B. Thus, it should be possible to separate the effects of the nickel and boron from the effect of helium by irradiating steels with the two isotopes of either nickel or boron. This has not been done up to now for any mechanical properties tests.

Finally, the use of thermal-neutron-shielded and unshielded capsules in a mixed spectrum reactor can be used to vary the He:dpa ratio buildup during irradiation. This is illustrated in Fig. 5 for an unshielded and an Eu_2O_3-shielded capsule irradiated in HFIR. In the unshielded capsule, all ^{10}B is quickly transmuted to helium, whereas in the shielded capsule there is a much slower buildup, closer to what will happen in a fusion reactor. When the irradiation illustrated in Fig. 5 for ^{10}B in the shielded capsule is continued to about 13 dpa, this steel, the steel with ^{10}B irradiated in the unshielded capsule, and the nickel-containing steel in the unshielded capsule will all reach 300 appm, thus allowing for the comparison of the properties of the nickel- and boron-doped steels with similar helium concentrations but achieved by different techniques.

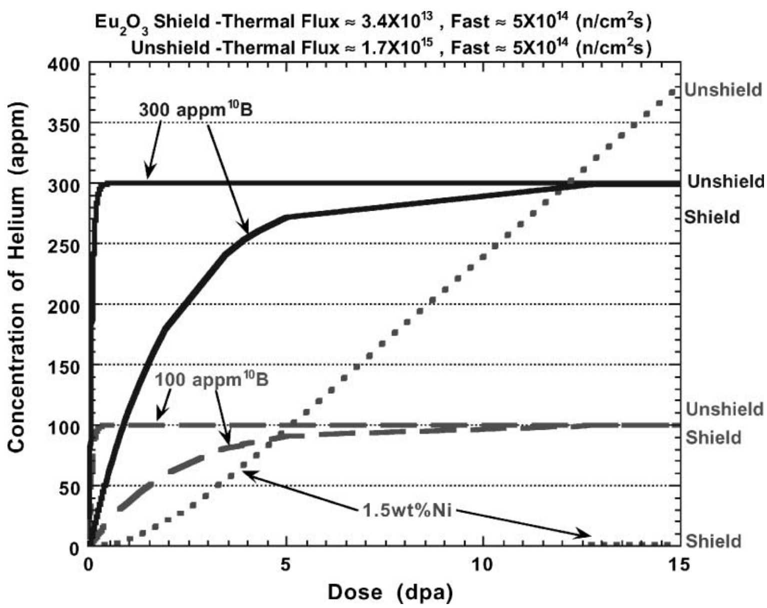

Fig. 5. Concentration of helium vs. irradiation dose for nickel- and boron-doped steels irradiated in HFIR in Eu_2O_3-shielded and unshielded capsules.

5. Summary

The effect of helium on mechanical properties of first-wall structural materials is important because large amounts of helium will form during irradiation with 14 MeV neutrons from the fusion reaction. Without a 14 MeV neutron source, simulation techniques using fission reactors must be used to study helium effects. The techniques that are most amenable for ferritic/martensitic steels are doping the steels with either boron or nickel. Past irradiation experiments using the techniques have demonstrated similar effects on the Charpy impact properties that have been attributed to helium. It is recognized that both techniques have disadvantages. Nickel lowers A_{C1} and promotes M_6C during irradiation. Boron is an active element that can promote precipitation and affect prior austenite grain size; during the transmutation reaction that produces helium, lithium is also produced. However, with proper planning and precautions, experiments using the nickel- and boron-doping techniques can be used to develop an understanding of the effects of helium on mechanical properties.

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