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THE EFFECT OF NEUTRON IRRADIATION ON VANADIUM ALLOYS*

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Abstract

Neutron-irradiated vanadium alloys were evaluated for their susceptibility to irradiation hardening, helium embrittlement, swelling, and residual radioactivity, and the results were compared with those for the austenitic and ferritic stainless steels. The VANSTAR-7 and V–15Cr–5Ti alloys showed the greatest hardening between 400 and 600°C while V–3Ti–1Si and V–20Ti had lower values that were comparable to those of ferritic steels. The V–15Cr–5Ti and VANSTAR-7 alloys were susceptible to helium embrittlement caused by the combination of weakened grain boundaries and irradiation-hardened grain matrices. Specimen fractures were entirely intergranular in the most severe instances of embrittlement. The V–3Ti–1Si and V–20Ti alloys were more resistant to helium embrittlement. Except for VANSTAR-7 irradiated to 40 dpa at 520°C, all of the vanadium alloys exhibited low swelling that was similar to the ferritic steels. Swelling was greater in specimens that were preimplanted with helium using the tritium trick. The vanadium alloys clearly exhibit lower residual radioactivity after irradiation than the ferrous alloys.

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Introduction

The structural first wall of a tokamak-type fusion reactor poses a number of severe demands on the selected material. Radiation damage from a neutron spectrum with 14 MeV energies is often considered the most serious problem [1]. In the past, vanadium alloys have been considered for use as a fuel cladding material in fission reactors and the results from a number of neutron irradiation experiments using these alloys have already been reported [2—10]. One of the difficulties associated with vanadium alloys is their tendency to pick up light elements (C, N, and O) which embrittle the material. In many experiments, this has made it difficult or impossible to separate the effects of irradiation from those due to contamination. This problem was recently overcome by encapsulating vanadium specimens in TZM tubes filled with lithium. The tubes were irradiated in the Materials Open Test Assembly (MOTA) of the Fast Flux Test Facility (FFTF) [11,12]. In this paper, key results from that work for V—15Cr—5Ti, VANSTAR-7, and V—3Ti—1Si, as well as those for other vanadium alloys reported in the literature, are compared with the ferritic and austenitic stainless steels. The results are divided into four major neutron irradiation effects that have been observed in the vanadium alloys: (1) irradiation hardening, (2) helium embrittlement, (3) swelling, and (4) residual radioactivity. In all but the last case, features of the irradiated microstructure as viewed by transmission and scanning electron microscopy (TEM and SEM, respectively) are correlated with the respective tensile or physical properties.

Irradiation Hardening

Neutron irradiation increases the yield strength of an alloy or "hardens" it by creating defects in the vanadium alloy microstructure. These defects are generally divided into three types: dislocation loops, network dislocations, and cavities (voids). Previous investigators have shown that the sizes and densities of these defects correlate reasonably well with the amount of irradiation hardening [13]. Other hardening mechanisms such as irradiation-induced precipitate hardening or transmutation product hardening (e.g., helium or hydrogen bubbles) may also contribute to this phenomenon. An example of a vanadium alloy microstructure that has been hardened by irradiation is shown in Fig. 1. A high density of small dislocation loops and segments were observed in this V—15Cr—5Ti specimen irradiated to 10 dpa in the MOTA. A small number of cavities was also present but these were not visible in the micrograph under the diffraction contrast imaging conditions used. There were no helium bubbles present because this specimen was not preimplanted with helium. The dense array of defects impedes the motion of dislocations through the microstructure and thus hardens the alloy. The typical effect of irradiation hardening on tensile behavior is shown in Fig. 2 for VANSTAR-7 irradiated in the MOTA to 40 dpa at 420°C. Note first that a control specimen aged in the lithium-filled capsules at 420°C for 257 days had a stress-strain curve that was essentially the same as the unirradiated starting material. This indicates that no appreciable hardening due to precipitation, interstitial pickup, or any other mechanism took place in the vanadium during thermal aging. The hardening due to irradiation was evident in the increased yield strengths after damage levels of 10 and 40 dpa were reached. These increases in yield strength were accompanied by decreases in total elongation. The continuous increase of yield strength with damage level up to ~40 dpa does not necessarily apply to all vanadium alloys and, for some, the yield strength increases saturated at lower damage levels [12]. At first glance, it would appear that helium, preimplanted by using the tritium trick [14], also increased the hardening because specimens with helium exhibited somewhat higher yield strengths. However, consideration of tensile data for this and a number of other vanadium alloys showed that the situation was reversed as often as not [12]. Therefore, the effect of preimplanted helium on irradiation hardening did not appear to be significant. Caskey [15] has observed yield strength increases in austenitic stainless steels containing helium implanted by the tritium trick, but the helium levels and distributions differed considerably from those in the vanadium alloys [14].

Irradiation hardening may reach a point where specimen ductility is reduced to very low levels and failure occurs by cleavage fracture as illustrated in Fig. 3. The micrograph shows the fracture surface of an unimplanted V—15Cr—5Ti specimen that was tensile tested after irradiation in FFTF to 10 dpa at 420°C. Cleavage fracture surfaces tend to have flat fan-shaped surfaces covered with "treelike" markings or "river patterns." The cleavage failure of a hardened metal is characteristic of vanadium alloys and other bcc materials, but does not occur in the fcc alloys such as austenitic stainless steel. The fracture behavior is temperature dependent and the temperature at which a change from ductile to brittle (cleavage) failure takes place is called the ductile-brittle transition temperature (DBTT). The relationship between irradiation hardening and fracture behavior can be visualized in simple terms by consulting the type of schematic diagram used by Tetelman and McEvily [16]. Such a diagram is shown in Fig. 4 where both fracture stress (σ_f) and yield stress (σ_y) are plotted as a function of temperature. The intersection of the yield stress curve for the starting material [$\sigma_y(1)$] with the σ_f curve is the DBTT(1). When the yield stress of the vanadium alloy is increased from $\sigma_y(1)$ to $\sigma_y(2)$ due to irradiation hardening, the intersection with σ_f also increases the DBTT. Depending on the vanadium alloy and its fracture stress curve it is possible to raise the DBTT, which is generally below room temperature, to elevated temperatures such as shown for V—15Cr—5Ti in Fig. 3. Of course, a DBTT determined by a tensile test will differ from one obtained by the Charpy impact test that takes place at much higher strain rates. The DBTT derived from the tensile test will generally be lower than that from impact tests.

The irradiation-hardening behavior for several vanadium alloys was quantified and compared with other first wall candidate materials as shown in Fig. 5. For this presentation, irradiation hardening was defined as the difference between the unirradiated and irradiated yield strengths and is shown as a function of irradiation temperature for a number of vanadium alloys as well as bands representing common austenitic and ferritic steels. Whenever possible, the yield strength values of respective thermally aged controls were used instead of unirradiated controls to give the best measure of true irradiation hardening. However, in most cases the difference between the two controls was not large anyway. Alloys represented in the ferritic band include: HT-9 [17], 2.25Cr-1Mo [17], 12Cr-1MoVW [18], and 9Cr-1MoVNb [19]. These alloys exhibit irradiation hardening which drops off rapidly above ~400°C until yield strengths actually decrease after irradiation (irradiation softening). The alloys represented in the annealed austenitic band include: 304 [20], SA 316 [21], and 347 [22]. These alloys were more prone to harden under neutron irradiation in the temperature range investigated and hardening values decreased gradually above ~400°C to just above zero at 700°C. However, the 20%-cold-worked 316 [23] behaved more like the ferritics. Superimposed on the stainless steel data are hardening curves for four vanadium alloys. The V—15Cr—5Ti, VANSTAR-7, and V—3Ti—1Si specimens were irradiated in the FFTF [12] while V—20Ti was irradiated in EBR-II [8]. All of the alloys contained ~80 appm He, but the V—20Ti was implanted at room temperature with a cyclotron while the others were implanted at 400°C using the tritium trick. It is seen that a large variation in irradiation hardening behavior exists between the different vanadium alloys. VANSTAR-7 showed the greatest susceptibility to hardening followed by V—15Cr—5Ti and V—3Ti—1Si, while the V—20Ti fell within the band for ferritics. The reason for these differences in behavior between the vanadium alloys is not clear at this time. However, lower susceptibility to irradiation hardening may be related, in part, to higher titanium content in the alloy as shown by Böhm [2]. From a technological standpoint, the vanadium alloys showing less susceptibility to hardening would be more desirable candidates for a first wall because they retain higher ductility after prolonged exposure to neutron irradiation. In addition, alloys that harden less also appear to resist helium embrittlement better, as will be discussed in the next section. Therefore, the alloy development strategy for reducing irradiation hardening should probably favor the simple V-Ti alloys like V—3Ti—1Si and V—20Ti.

Helium Embrittlement

Some of the vanadium alloys have shown a marked sensitivity to helium embrittlement, especially when the helium is implanted using the tritium trick [12]. One of the more sensitive alloys is V—15Cr—5Ti. Rather high densities of helium bubbles form on the grain boundaries during the tritium trick. Subsequent irradiation in FFTF to 40 dpa coarsened the grain boundary bubbles as shown in Fig. 6 for a specimen irradiated at 520°C. Weakening of the grain boundaries due to these bubbles, combined with irradiation-hardened grain matrices, caused the alloy to fail intergranularly with little or no elongation as shown in Fig. 7. Irradiation to a damage level of 17 dpa reduced the total elongation of V—15Cr—5Ti from 24 to ~8% with 74 appm He and to zero with 300 appm He. Irradiation to 40 dpa created more irradiation hardening as demonstrated by the increase in yield stress and the specimen failed after less than 3% strain. At the lower elongations (<~3%), specimens exhibited fracture surfaces that were entirely intergranular as shown in Fig. 8. Specimens with somewhat greater ductilities had fractures that were mixtures of intergranular, cleavage, and ductile modes [12].

The relative susceptibility of four vanadium alloys to helium embrittlement is shown in Fig. 9 and compared with the austenitic and ferritic stainless steels. Comparison between the V—15Cr—5Ti, VANSTAR-7, and V—3Ti—1Si should be quite reliable because these alloys contained nearly the same amounts of helium (~80 appm) and were irradiated together in the FFTF to 40 dpa [12]. The embrittlement data for V—20Ti was obtained under somewhat similar conditions with a comparable helium level (90 appm), but a lower damage level (17 dpa). The results show a high sensitivity of V—15Cr—5Ti to helium embrittlement from ~400 to 600°C. Data points at 400 and 700°C from Grossbeck [10] on the same alloy irradiated in EBR-II agree quite well with the curve based on FFTF specimens. VANSTAR-7 was also severely embrittled at 520°C but had some ductility left at both 420 and 600°C. The V—3Ti—1Si and V—20Ti alloys both resisted helium rather well and their curves fall between results for the ferritic and austenitic stainless steels.

Comparison of the results for the vanadium alloys with those for the stainless steels must be done very carefully because of the wide differences in irradiation conditions, alloy helium levels, alloy impurities, etc. Therefore, the reader should view Fig. 9 as a comparison between present existing data fields for the different material groups even though there is not a continuity in conditions between groups. To prevent the drawing of incorrect conclusions, it is important to outline, in general terms, the conditions represented by the data bands. The band for austenitic stainless steels was taken from Maziasz [24] for a number of annealed and cold-worked 316 stainless steels and PCA irradiated from 10 to 22 dpa. Helium levels generated in the alloying <u>during</u> irradiation ranged from 5 to 15 appm in EBR-II to

1750 appm in HFIR. Included also are data points from the work of de Vries et al. [25] for DIN 1.4948 (similar to AISI type 304) with 7 appm He and strain rates of $3 \times 10^{-7}\ s^{-1}$. The ferritic band includes data from the same investigators [17–19] used in the ferritics band on irradiation hardening in Fig. 5. In addition, some results from Klueh and Vitek [26] on 12Cr-1MoVW and 9Cr1MoVNb with up to 2 wt % nickel added are used. Except for this last case in which nickel was added for the purpose of creating helium (up to ~50 appm), the other ferritic alloys contained little or no helium. Essentially no helium embrittlement has been observed in the ferritic stainless steels, but again, the levels generated in past fission reactors have been very low.

In Fig. 9, severe helium embrittlement with 100% intergranular fracture occurs in the curves or bands below ~3% total elongation. The V–15Cr–5Ti, VANSTAR-7 alloy around 500°C, and several PCA and 316-type austenitic stainless steels fall into this category. Above this ductility level, helium may be acting to reduce total elongation somewhat but is probably not technologically significant. However, new data that is being generated for higher damage and helium levels may tend to push many of the curves and data bands downward.

It is interesting to note that the same vanadium alloys that exhibited the greatest irradiation hardening (i.e., V–15Cr–5Ti and VANSTAR-7) were also more susceptible to helium embrittlement. These results strengthen the author's belief that the mechanism of helium embrittlement involves the hardening of grain matrices (due to irradiation) and weakening of the grain boundaries due to the presence of helium or helium bubbles. In the extreme cases, irradiation hardening can cause brittle cleavage failure with no helium present [12] or relatively high levels of helium can cause 100% brittle intergranular failure without any irradiation [14]. However, from the standpoint of alloy development, there are vanadium alloys that appear to resist irradiation hardening and seem to accommodate larger amounts of helium without deleterious effects. Again, the development effort should probably focus on simple V-Ti alloys such as V–20Ti and V–3Ti–1Si. The effect of the silicon addition on the neutron irradiation hardening and embrittlement is not clear and it should be investigated because of its apparent positive contribution.

Swelling

The results of cavity swelling for a number of different vanadium alloys are compared with the general swelling behavior for the austenitic and ferritic stainless steels in Fig. 10. Swelling is plotted as a function of damage level. The austenitic band is based on a summary for a number of annealed and cold-worked 316-type stainless steels irradiated from ~400 to 650°C in HFIR and EBR-II [27]. The ferritic band along the bottom of the graph includes the results of Vitek and Klueh [28], Loomis [29], and Gelles and Thomas [30]. The data points for V–15Cr–5Ti, VANSTAR-7, and V–3Ti–1Si that were irradiated to 40 dpa in the FFTF experiment [12] and the V–20Ti irradiated to 17 dpa in EBR-II [8] are shown as filled points because these materials were implanted with helium to levels of ~80 appm. For simplicity, the data point for maximum swelling in any given temperature series is plotted along with that temperature. For example, the maximum swelling of V–15Cr–5Ti after 40 dpa was ~0.28% at 600°C, while that for V–3Ti–1Si was ~0.09% at 400°C. Other data for the same four alloys [5–7,9,31,32] are shown as open points to denote that those specimens did not contain implanted helium. Although only a few points are shown in Fig. 10, all of the unimplanted vanadium alloy specimens irradiated in FFTF, regardless of alloy or irradiation temperature, exhibited lower swelling than identical specimens with ~80 appm He. Except for VANSTAR-7 irradiated to 40 dpa at 520°C, all of the other vanadium alloys were low swelling as they fell in the region just above the axis which also corresponds to the ferritic band. The cause of the high swelling in the one VANSTAR-7 specimen is not clear at this time. Swelling values for VANSTAR-7 at 420 and 600°C were less than 0.1% [12]. It is concluded that the vanadium alloys are generally low swelling under neutron irradiation and comparable to the ferritic stainless steels.

Residual Radioactivity

After neutron irradiation, any first wall amterial will have residual radioactivity associated with it. The level of the activity will impact on the areas of maintenance, safety, and most importantly, waste management [33]. Since waste management will most likely influence public acceptance of fusion reactors for power generation [33], low activation characteristics of the first wall will make it easier to meet goals in these areas. Vanadium alloys are generally low activation materials and are clearly superior to both the ferritic and austenitic stainless steels as shown in Fig. 11. The vanadium alloys reach an induced activation equal to that of newly enriched uranium at least five times sooner than the stainless steels. Even with substitution of lower activation elements for those with high activities, the stainless steels will not compare favorably with the vanadium alloys. However, the impurity levels of certain high activation elements, such as nitrogen and niobium, must be controlled in all alloy classes in order to maintain reasonably low activation behavior.

Summary

The behavior of vanadium alloys following neutron irradiation was examined in the areas of irradiation hardening, helium embrittlement, and swelling, and residual activity, and the results were compared with those for the austenitic and ferritic stainless steels.

The tendency for vanadium to harden under neutron irradiation varied widely from one alloy to another. VANSTAR-7 and V–15Cr–5Ti specimens exhibited the greatest hardening. Several V–15Cr–5Ti specimens were hardened so severely after irradiation at 420°C that they failed in a cleavage mode with low total elongations. On the other hand, V–3Ti–1Si and V–20Ti showed lower hardening in the range of 400 to 600°C and were comparable to the ferritic alloys and cold-worked 316 stainless steels.

The V–15Cr–5Ti alloy, and to a lesser extent, VANSTAR-7, were quite sensitive to the ~80 appm of helium preimplanted using the tritium trick. Large helium bubbles formed on the grain boundaries, weakened them, and in conjunction with irradiation hardening of the grain matrices, caused premature specimen failure due to intergranular separation. Similar results have been observed in some austenitic stainless steels at higher helium levels. The V–3Ti–1Si and V–20Ti alloys were more resistant to helium embrittlement due to reduced hardening and better accommodation of helium. No helium embrittlement has been observed in the ferritic steels, but they have been irradiated with relatively low helium levels compared to the austenitic or vanadium alloys.

Except for VANSTAR-7 irradiated to 40 dpa at 520°C, all other vanadium alloys exhibited low swelling that was comparable to the ferritic stainless steels. Preimplanted helium slightly increased the swelling observed in all of the vanadium alloys.

The vanadium alloys clearly demonstrate lower residual radioactivity after irradiation than either type of steel, which is an advantage for the waste management, maintenance, and safety of a first wall material.

The development of neutron irradiation resistance vanadium alloys should favor simple V-Ti alloys such as V–3Ti–1Si and V–20Ti which showed the best performance of the alloys investigated.

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LIST OF FIGURES

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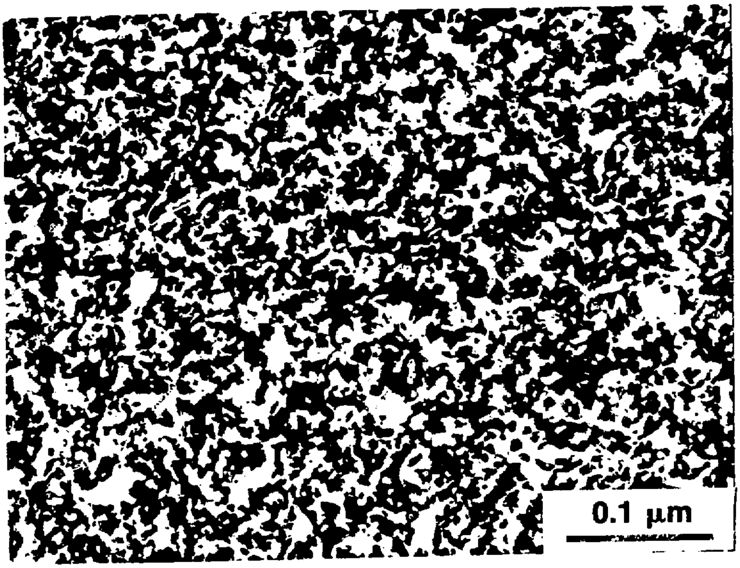

Fig. 1. Irradiation-hardened microstructure of a V–15Cr–5Ti specimen that was irradiated in FFTF to 10 dpa at 420°C.

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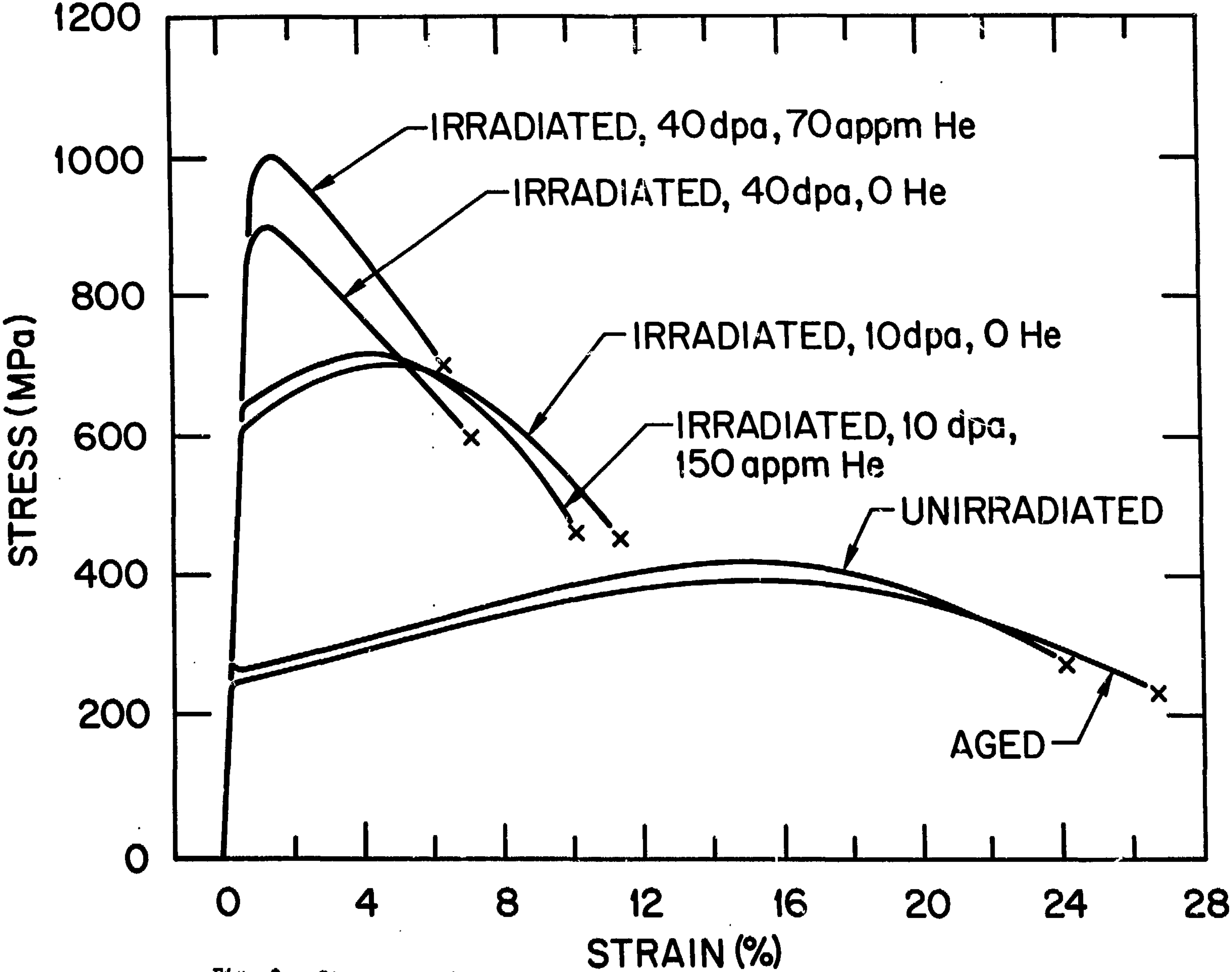

Fig. 2. Stress-strain curves for unirradiated, aged (257 days), and irradiated VANSTAR-7 specimens illustrating yield stress increases due to irradiation hardening. Aging, irradiation, and tensile tests

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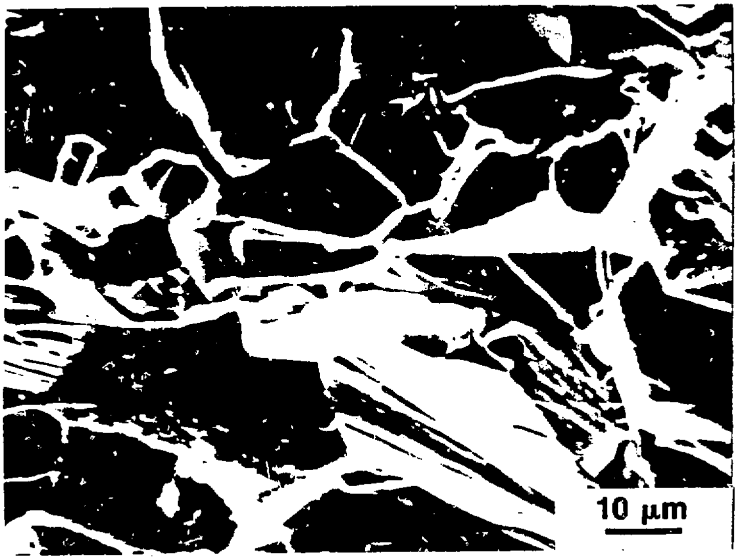

Fig. 3. Brittle cleavage fracture in a V–15Cr–5Ti specimen without preimplanted helium that was irradiated in FFTF to 10 dpa at 420°C and tested in tension at 420°C.

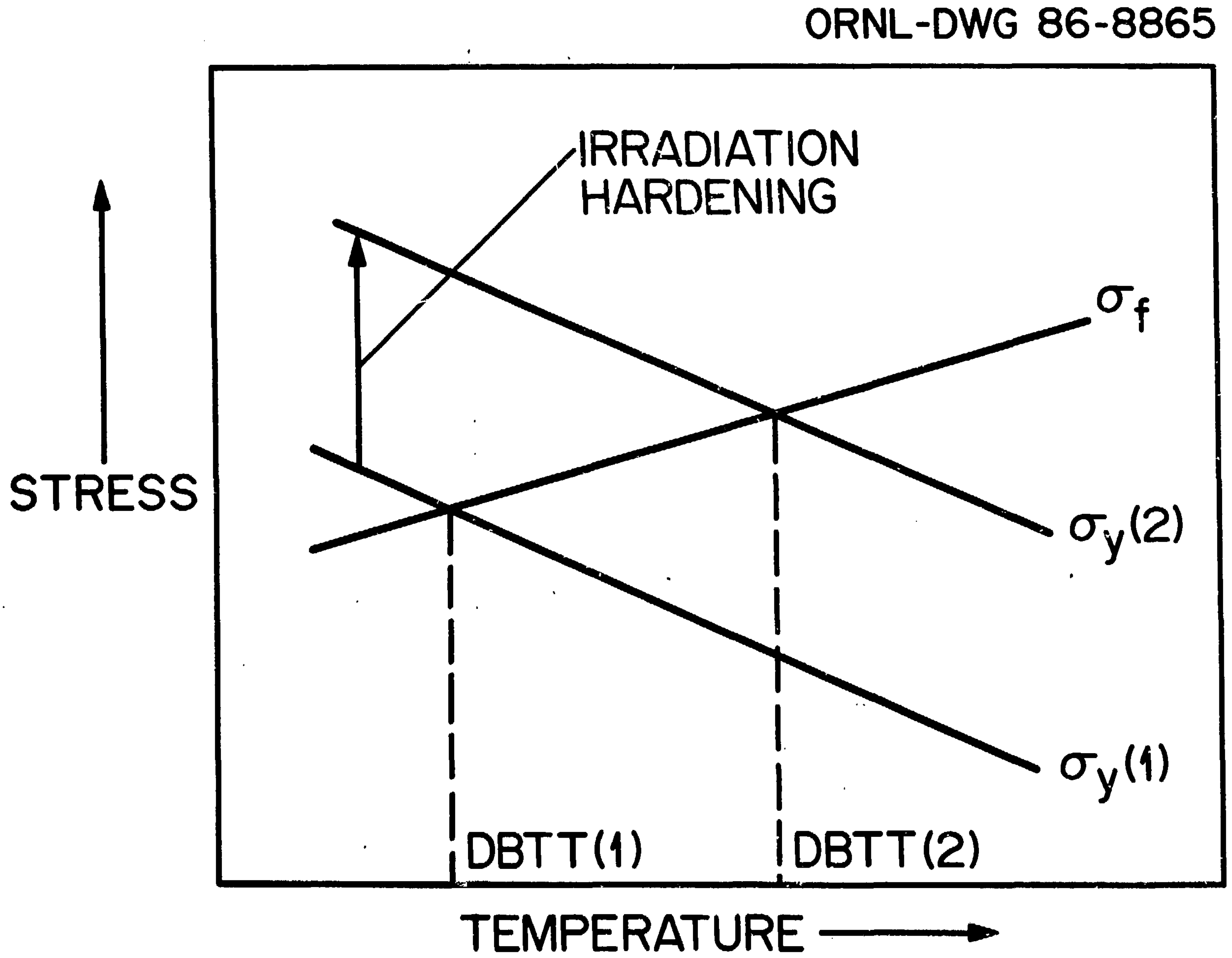

Fig. 4. Schematic diagram showing how irradiation hardening raises the DBTT of a vanadium alloy (after Tetelman and McEvily [16]).

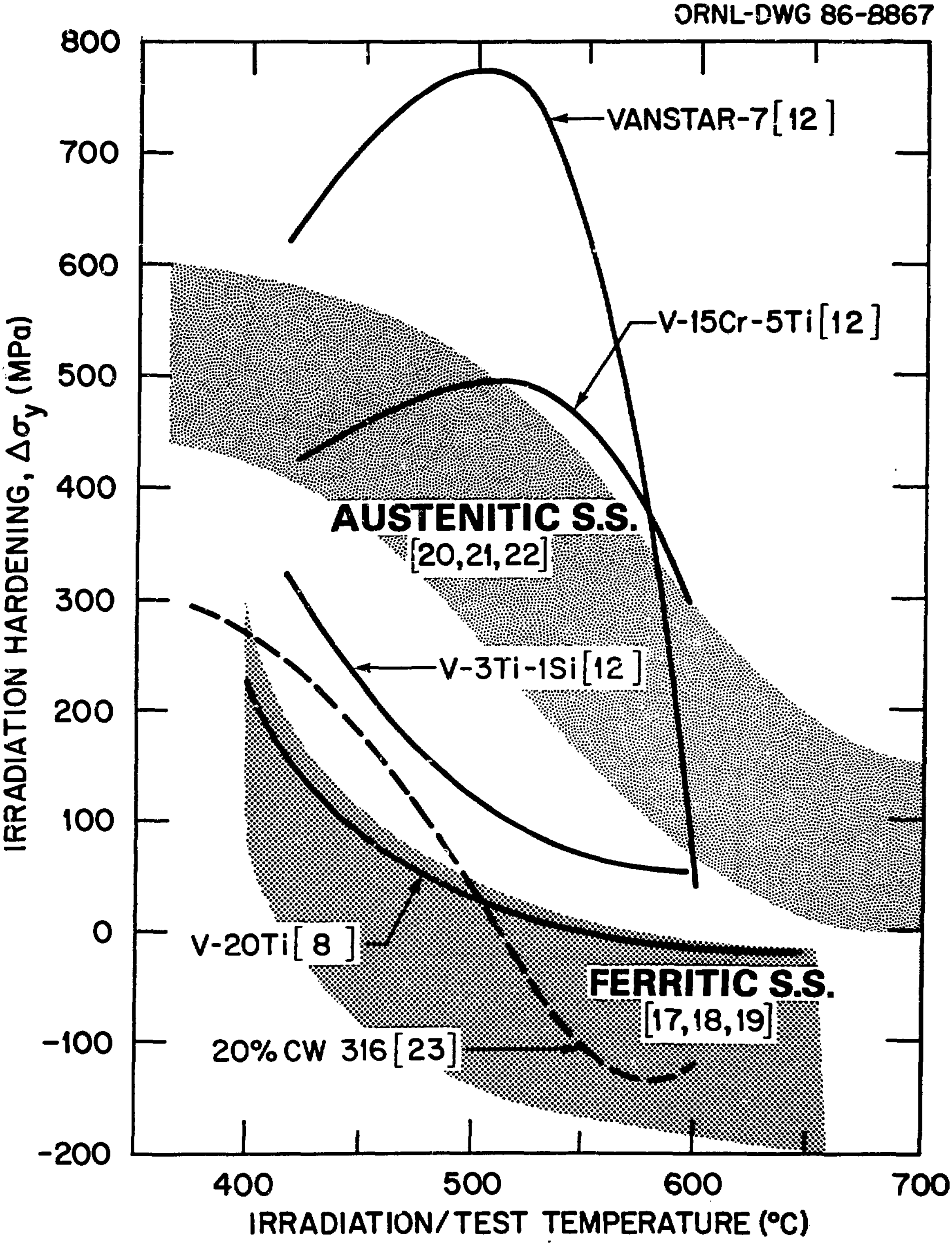

Fig. 5. Irradiation hardening (change in yield stress) as a function of irradiation/test temperature for vanadium alloys compared with similar data for austenitic and ferritic stainless steels. Reference numbers are in brackets.

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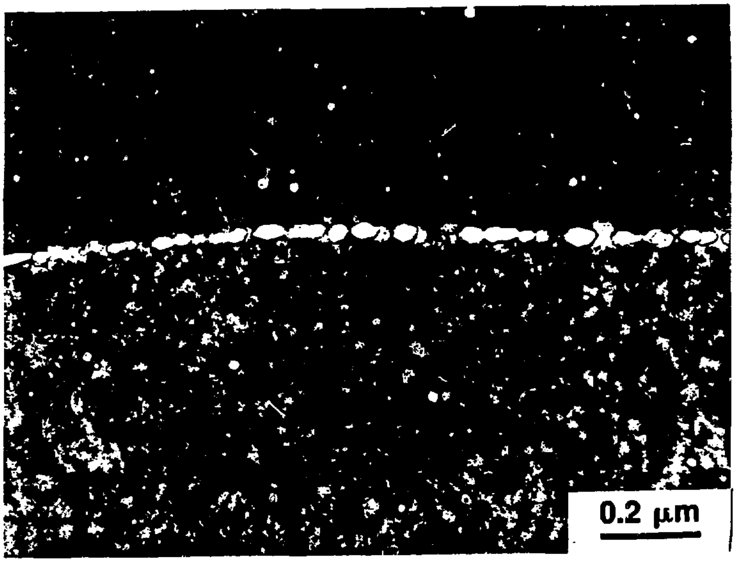

Fig. 6. Grain boundary bubbles in a V–15Cr–5Ti specimen with 74 appm He after irradiation in FFTF to 40 dpa at 520°C.

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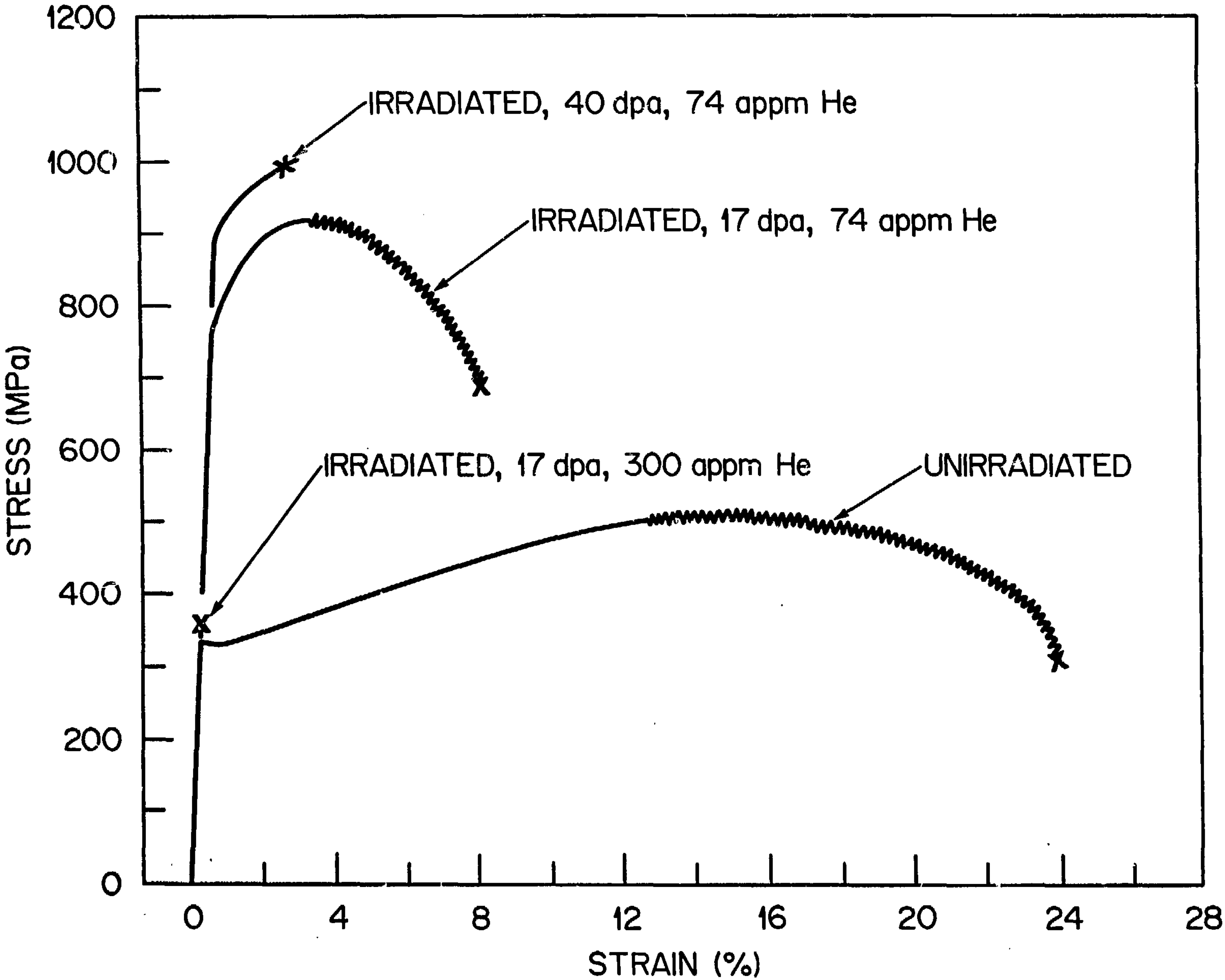

Fig. 7. Stress-strain curves for unirradiated and irradiated V–15Cr–5Ti specimens showing the effects of helium embrittlement. Specimens were irradiated in FFTF at 520°C. (From ref. [12]).

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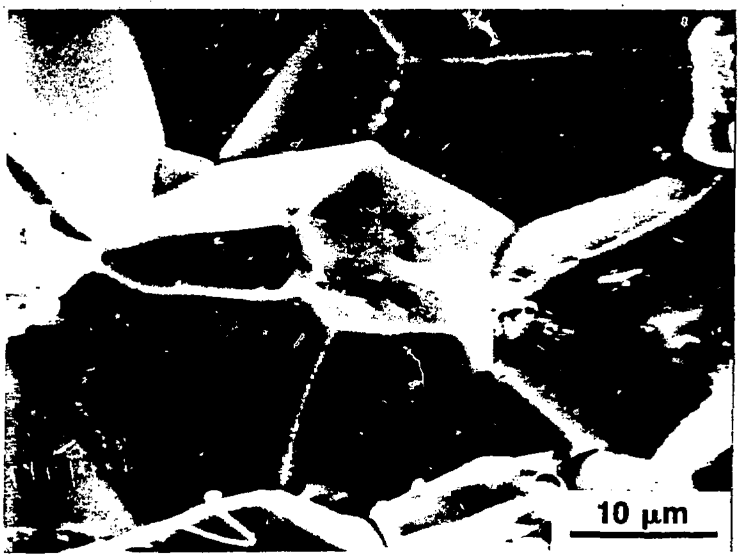

Fig. 8. Brittle intergranular fracture in a V–15Cr–5Ti specimen containing 300 appm He that was irradiated in FFTF to 10 dpa at 420°C and tested in tension at 420°C.

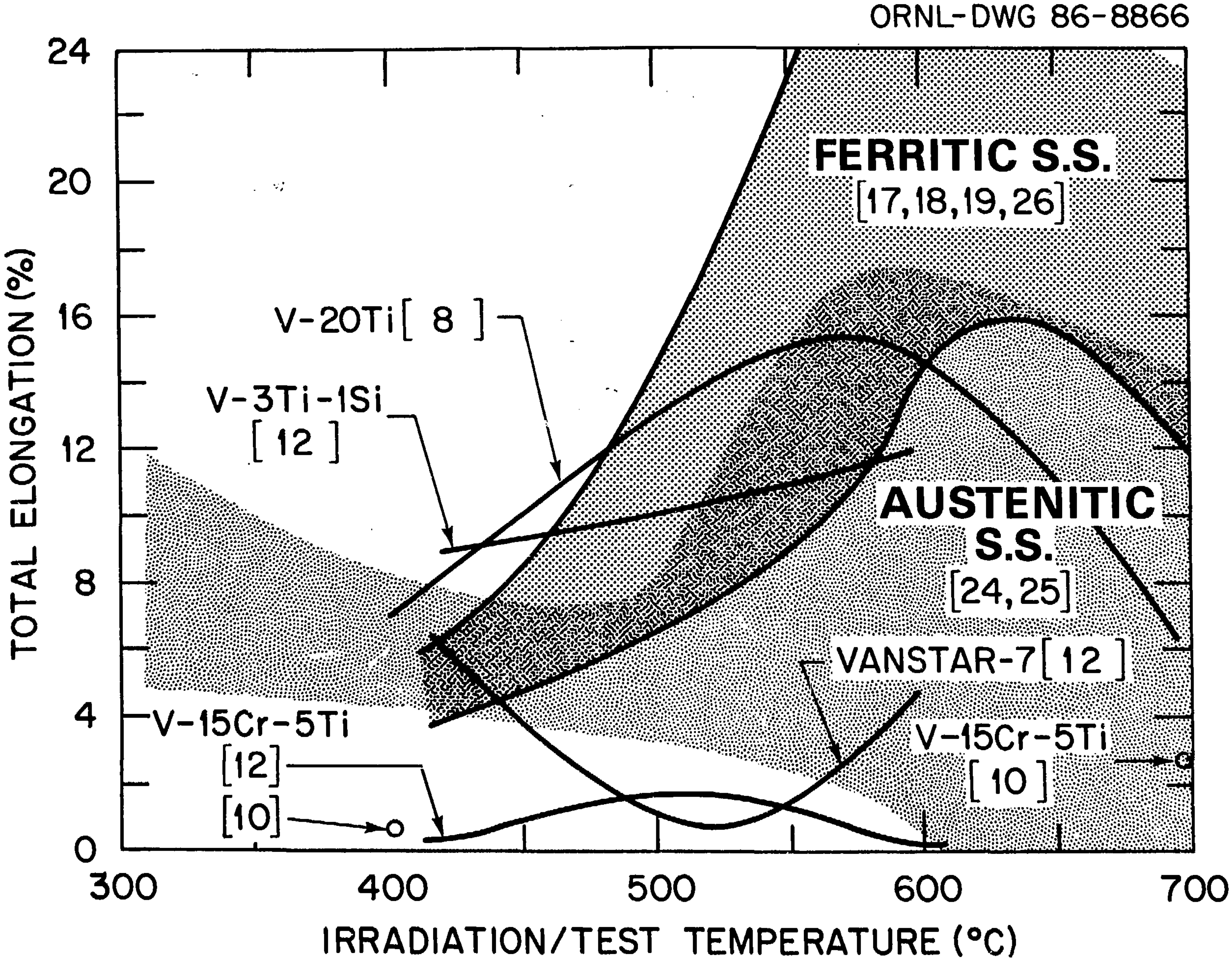

Fig. 9. Total elongation as a function of irradiation/test temperature for vanadium alloys compared with similar data for austenitic and ferritic stainless steels. Reference numbers are in brackets.

Fig. 10. Cavity swelling in vanadium alloys as a function of damage level compared with similar data for austenitic and ferritic stainless steels. The numbers are irradiation temperatures in °C and the reference numbers are in brackets.

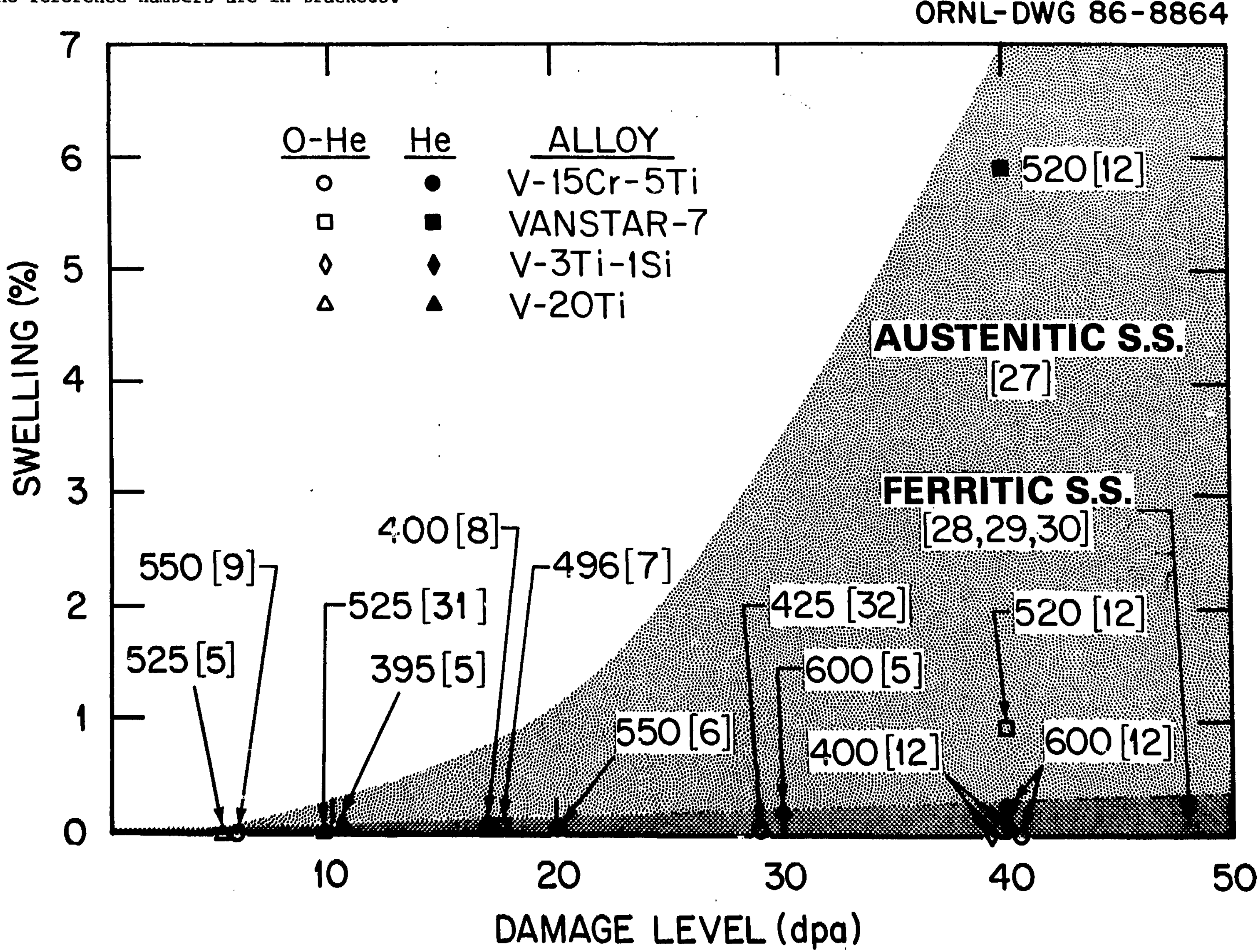

Fig. 11. Induced activation of vanadium alloys, ferritic stainless steels, and austenitic stainless steels as a function of time after reactor shutdown. Calculated curves assume a total fluence of ~1×10^{27} n/m^2 or ~100 dpa in an iron alloy first wall (after Wiffen and Santoro [33]).

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