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Comparison of swelling and irradiation creep behavior of fcc-austenitic and bcc-ferritic/martensitic alloys at high neutron exposure

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Abstract

It is well-known that ferritic and ferritic/martensitic steels develop much less swelling than austenitic steels during neutron or charged particle irradiation. The prevailing assumption is usually that the steady-state swelling rate of bcc steels is inherently much lower than that of fcc steels, at least a factor of ten or more. It is shown in this paper that this perception is incorrect, with bcc steels having steady-state swelling rates perhaps only a factor two to four lower. It is thought to be significant that the creep compliance of the two types of alloys also differs only by a factor of about two. The lower swelling observed in bcc steels relative to fcc steels is shown in this paper to be primarily a consequence of much longer transient regimes prior to the onset of steady-state swelling. Several other commonly held perceptions concerning the swelling of both bcc and fcc steels are examined in this paper and also shown to require revision. These involve the effect of cold-work on swelling, the extent of the temperature regime of swelling, and the possibility that swelling might saturate eventually. It also appears that the use of well-controlled in-reactor materials tests that employ active temperature control tend to yield significantly lower values of void swelling compared to that obtained under more representative conditions typical of actual reactor operation. The role of previously ignored differences in displacement rate to determine the duration of the transient regime of void swelling is also shown to require reevaluation, especially for bcc steels. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

The majority of the papers presented at this workshop focus on the behavior of microscopic radiation-induced defects and defect clusters on very short time scales and at very low total damage levels. This focus is valid in that it seeks to illuminate those features of crystal structure which affect the production, survivability and aggregation of radiation-induced point defects. Better understanding of the crystallographic origins of these defect ensembles can then be applied to modeling the crystallographic differences driving macroscopic changes in either property or dimension that develop at much higher radiation exposures.

To some extent, this low to high exposure extrapolation is actually a circular process, in that large amounts of high exposure data are already available and have led to development of perceptions that influence our idea of what the crystallographic dependencies ‘ought’ to be on the small dose, small size, short time scale. An excellent example of this feedback loop can be seen in previous workshops in this series [1], with the growing recognition that the 1%/dpa swelling rate characteristic of austenitic steels could not be rationalized within the framework of the then-conventional wisdom. This deficiency prompted the development of the ‘production bias’ concept of defect production [2–4] which has been discussed and debated extensively in this forum series.

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This paper addresses the question of whether we can use the steady-state swelling and creep rates of fcc and bcc iron-base alloys at high damage levels to draw conclusions concerning the relative defect production, survivability, and agglomeration of point defects in the two crystal systems. Only iron-base alloys were chosen for this comparison in order to avoid strong differences in transmutation and other chemistry-related factors associated with other alloy systems.

Perhaps it may be possible to answer such a question and provide information for microscopic model development, but the most important criterion is to assure that we have the correct perception of these steady-state swelling and creep rates, as well as their range of applicability. Since perceptions, even incorrect ones, are often more powerful than facts, it is important to re-examine such perceptions periodically to assess whether current data continue to validate these perceptions.

At the current time, for instance, it is usually assumed that the steady-state swelling rates of bcc and fcc iron-based alloys are very different, perhaps by a factor of 10–20, and must therefore reflect some microscopic property dependent on their different crystal structures. In fact, however, the confidence underlying this assumption has been eroding recently. In particular, the perception that the generic bcc crystal structure confers an immunity on all bcc metals and alloys against the high swelling rates characteristic of fcc metals and alloys was recently called into doubt. This occurred when various vanadium-binary alloys were found to swell at very large rates, sometimes in excess of those observed in fcc systems [5,6]. This suggests that chemical and segregation processes can easily overwhelm crystallographic influences, or perhaps even more importantly, that the crystallographic influence may not have been as large as previously envisioned.

It will be shown in this paper that the differences in steady-state swelling rates of iron-based fcc and bcc alloys are not so very large, with the observed difference in swelling reflecting primarily the influence of crystal structure, composition, displacement rate, microchemical evolution and temperature history to determine the duration of the transient regime of swelling. It will also be shown that some perceptions concerning the swelling of fcc alloys also require modification based on acquisition of more recent data. The fcc alloys will be covered first, since the conclusions drawn from their reassessment set the stage for a much stronger revision of our perception of bcc behavior.

2. Swelling of iron-based alloys

As reviewed in Refs. [7–14] it is now generally accepted that the swelling of fcc alloys based on the Fe–Cr–Ni and Fe–Cr–Mn systems exhibit a well-defined and universal pattern during neutron irradiation at elevated temperatures. All these alloys eventually swell at a 'universal rate' of ~1%/dpa, regardless of composition. This universal rate manifests itself over a wide range of atomic displacement rates and irradiation temperatures, but is thought not to persist at this rate as the temperature decreases below ~430°C, with a near-zero swelling rate reached around ~350°C. The development of this universal swelling rate is preceded by a transient regime whose duration is primarily dominated first by the initial alloy composition, and second by the subsequent radiation-induced microchemical evolution of the alloy matrix. This evolution has been thought to *always* be delayed by cold-working and to be very sensitive to thermal–mechanical treatment, atomic displacement rate, applied stress state and irradiation temperature history.

The 1%/dpa steady-state swelling rate persists to very high swelling levels, approaching 100% in some experiments, but has been observed to decrease eventually and sometimes reach a saturation of swelling under some limited sets of conditions. Such situations arise from the segregation-induced fcc to bcc decomposition that occurs in Fe–Cr–Mn alloys [12–14], from spinodal-like decomposition that occurs in solute-bearing Invar alloys [15] and from some previously unidentified phenomenon that occurs in Fe–Cr–Ni ternary alloys at relatively low irradiation temperatures and relatively high nickel levels [8]. Saturation can also occur in bcc systems but appears to be associated with the development of void lattices [16,17] which do not usually occur in fcc metals and alloys. (Pure nickel and Ni–Al alloys [17] have recently been shown to be exceptions to this generalization, however, and pure aluminum was earlier shown to develop void lattices [18–20]).

Recently published studies on fcc iron-based alloys by Garner and coworkers, however, have shown that several features of the above collection of conventional wisdom are in fact not always correct. In particular, cold-working does not always delay swelling and may even accelerate it; the temperature range of swelling and the existence of the 1%/dpa swelling rate may extend down to ~300°C and not only to 430°C; and observations of saturation of swelling in neutron-irradiated Fe–Cr–Ni ternary alloys [8] were in fact erroneous.

2.1. Swelling of stainless steels

Figs. 1 and 2 demonstrate several features of the swelling behavior of AISI 316 stainless steel as observed in two open-core materials tests conducted in the EBR-II [1] fast reactor. Note in Fig. 1 that in one heat of

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[1] EBR-II is the Experimental Breeder Reactor-II in Idaho Falls, ID. FFTF is the Fast Flux Test Facility in Richland, WA. MOTA is the Materials Open Test Assembly.

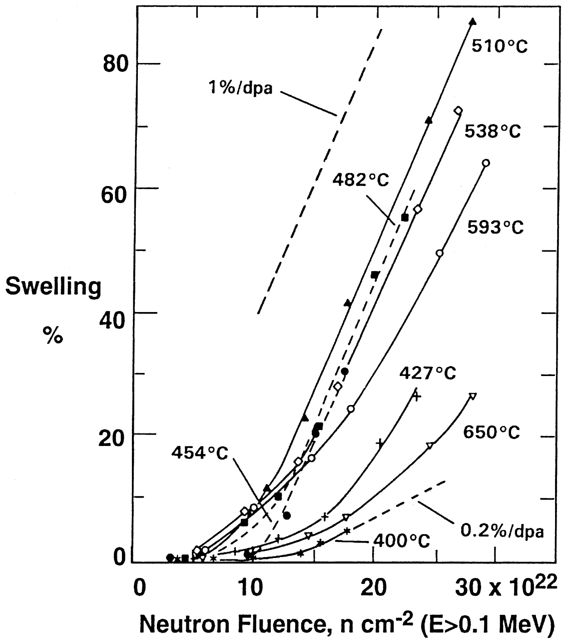

Fig. 1. Stress-free swelling of solid rods of 20% cold-worked N-lot AISI 316 during a materials test irradiation in the open core of EBR-II [21]. The lack of data scatter is due to repeated measurement and reinsertion of single specimens to further irradiation at each temperature.

20% cold-worked 316 stainless steel, the swelling rate of small rods exhibited a rather temperature-dependent transient duration of 10×10^{22}–25×10^{22} n cm^{-2} ($E >$ 0.1 MeV) or ~50–150 dpa before swelling thereafter at ~1%/dpa without saturation [21]. The very well-defined and invariant swelling rate in this experiment is easily demonstrated, arising from the lack of data scatter that results from making all measurements at a given temperature on a single specimen and then returning it successively to the reactor for continued irradiation.

Fig. 2 shows a second open-core experiment in EBR-II on multiple specimens of thin-walled tubes of the 'Core 4' heat of steel used in the construction of FFTF [22]. In this experiment, the temperature dependence of the transient regime in the range 475–600°C appears not to be as pronounced, primarily due to specimen-to-specimen data scatter, but the general behavior was very similar to that of Fig. 1. Note at the lower irradiation temperatures that the transient regime becomes progressively longer as the temperature decreases.

Similar behavior was also observed in another series of irradiations conducted in the open-core of FFTF on AISI 316 and D9, the latter a titanium-modified alloy, both in the cold-worked condition [23] as shown in Fig. 3. In this latter case, however, the data are derived from actual cladding of irradiated fuel pins, which were subject to often large and time-dependent variations in temperature. These three examples demonstrate the universality of the 1%/dpa behavior, independent of specimen form, test type, or test history. It is important to note, however, that all three of these experiments were subject to periodic negative deviations from the nominal test or irradiation temperatures.

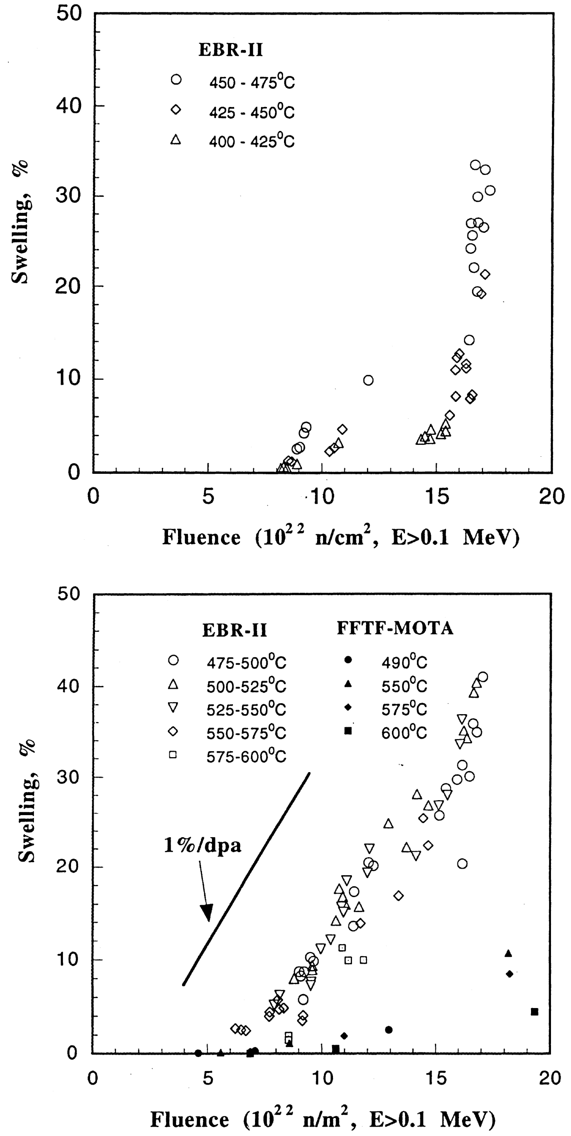

Fig. 2. Stress-free swelling of multiple specimens of thin-wall tubing of FFTF Core 4 heat of AISI 316 irradiated in EBR-II in another open-core experiment [22]. Also shown is the much lower stress-free swelling observed in the same heat of steel in the temperature-controlled Materials Open Test Assembly [24].

Note also in Fig. 3 that when the incubation period is prolonged, the transient tends to end very abruptly. In general, it has been observed that the longer the transient regime of swelling, the more abrupt is its

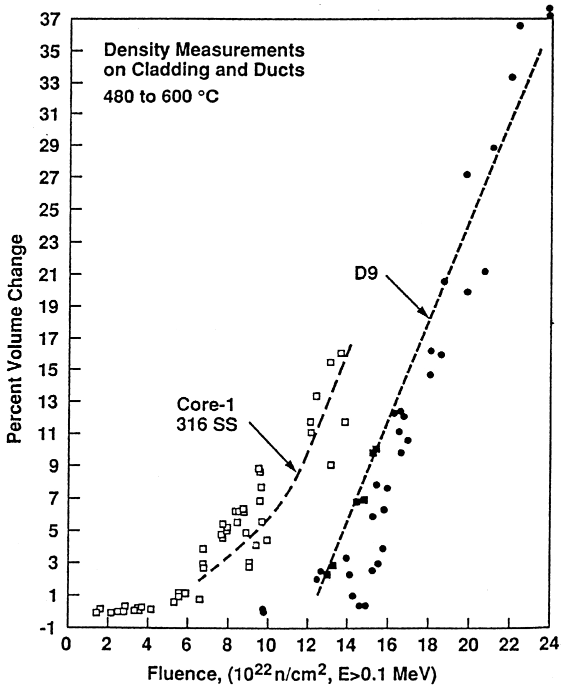

Fig. 3. Swelling observed in two cold-worked austenitic alloys after serving as fuel cladding in the open core of FFTF [23]. Note the increasing abruptness of the end of the transient regime as the transient duration increases in the D9 steel.

termination [7]. Also, the shorter the transient, the more curvature is observed in swelling rate during the transition to steady-state.

In creep tube tests conducted on the Core 4 steel in FFTF-MOTA using active temperature control to ±5°C, however, the stress-free swelling ranged only from 7–9% at 70 dpa in the range 490–600°C [24]. This is significantly lower than the >28% swelling observed in the passively controlled EBR-II experiments shown in Fig. 2. This effect of active temperature control to delay the onset of accelerated swelling will be shown to be an important observation in later sections when the swelling of model fcc and bcc alloys is discussed.

Note also in Fig. 1 that at 400°C the authors imply a significant reduction in steady-state swelling rate, based largely *only* on the perception that it should fall below 430°C. In fact, however, due to the coolant inlet temperature of ~370°C and to the large gradient in flux along the axis of the EBR-II core, it was impossible in either of the two EBR-II experiments to reach sufficient exposure to confidently preclude the possibility of an increase in swelling rate at higher exposure in the range 370–400°C. For light water power reactors, some portions of important components operate at these temperatures and can reach exposures of ~100 dpa [25].

In order to test the assumption that the steady-state swelling rate declines strongly in the range 350–430°C, one must generate high exposure data not in the second-generation types of fast reactors with relatively high coolant inlet temperatures (365–380°C in EBR-II, FFTF, PFR, Phenix, BN-600 [2]) but in the first-generation of fast reactors which have inlet temperatures in the range 270–300°C. Examples of the latter are DFR (270°C) BN-350 (285°C) and BOR-60 (300°C).

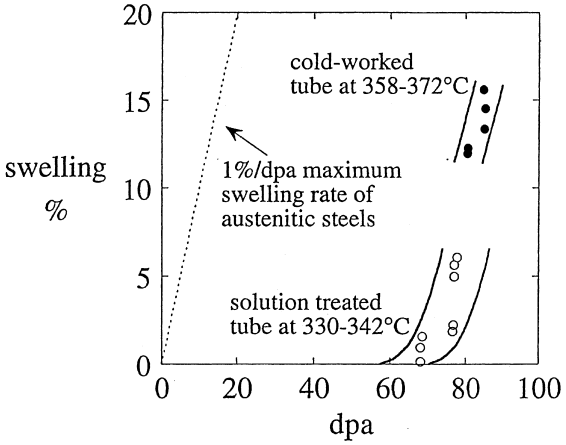

Fig. 4. Stress-free swelling behavior observed at relatively low temperatures in EI-847, a niobium-stabilized 16Cr–15Ni stainless steel irradiated in BN-350 [26].

There is at least one set of high exposure data at low temperatures that casts strong doubt on the decline of the swelling rate below 430°C. As shown in Fig. 4, a steel used in Soviet-design reactors for applications where AISI 316 would be used in the West appears to be developing swelling rates at 327–347°C and 355–380°C on the order of ~1%/dpa after very long transient periods when irradiated in the BN-350 reactor [26]. No previous studies have reached such high exposure levels at these low temperatures. Note also that the transient regime at the lowest temperature tends to end very abruptly, in a manner similar to that shown in Fig. 3.

Other studies on various Russian steels have recently shown that void swelling can extend down to temperatures of ~300°C, especially at the lower displacement rates experienced by the near-core austenitic components of pressurized water reactors [27–31]. It therefore appears that the temperature dependence of both swelling and the steady-state swelling rate of austenitic steels, especially at temperatures relevant to light water reactors (288–400°C), is not as pronounced as previously

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[2] PFR is the Prototype Fast Reactor and DFR is the Dounreay Fast Reactor, both in Dounreay, Scotland. Phenix, BN-600, BN-350, and BOR-60 are fast reactors in Marcoule, France; Beloyarsk, Russia; Shevchenko, Kazakhstan; and Dimitrovgrad, Russia.

envisioned. The 1%/dpa indeed appears to be a universal constant of very wide applicability, and in this case leads to the possibility that pressurized water reactors may experience significant and abrupt increases in swelling as they approach their 40–60 year lifetimes, reaching in some components maximum doses of 80–150 dpa.

2.2. Swelling in annealed ternary Fe–Cr–Ni alloys

In general, it is the inclusion of solutes like silicon, phosphorus and carbon that prolong the resistance to void swelling [7]. Thus, irradiations of simple solute-free model alloys allow us to observe the earliest onset of accelerated swelling. The most comprehensive and definitive studies on the compositional dependence of simple Fe–Cr–Ni ternary alloys were carried out in EBR-II and focused on the swelling of annealed, solute-free alloys [8,32]. These studies were completed in the early 1980s. Follow-on studies directed toward more specific and focused questions were conducted in both EBR-II and FFTF in the late 1980s and early 1990s.

The early EBR-II study showed that after a relatively short transient regime of −10 dpa, all Fe–15Cr–Ni alloys with 12–24% Ni at target temperatures in the range 400–510°C began to swell at ~1%/dpa as they were identical alloys at one temperature, as shown in Fig. 5(a). Increases in nickel level above 24% (Fig. 5(b)), increases in temperatures above 510°C, or decreases in chromium level all led to longer transient regimes, but eventually the ~1%/dpa rate was established at all compositions and temperatures.

At the termination of these studies in EBR-II, however, there was one perplexing and unresolved mystery. As shown in Figs. 6 and 7, it appeared that at the lower irradiation temperatures and higher nickel levels the post-transient swelling was either tending toward saturation or was falling in rate in a composition-dependent manner. Swelling at higher temperatures in this experiment did not exhibit such behavior, however. Since this experiment had been interrupted due to a funding reduction, there was no immediate prospect of resolving this behavior by measuring the swelling of other low temperature specimens that had been carried to higher exposure but not examined.

Some years later, portions of this experiment were resurrected and specimens irradiated to another 10–15 dpa were measured. To the surprise of Garner and Black [33], it was found that the tendency at low temperature toward saturation did not continue, but swelling 're-verted' to the characteristic ~1%/dpa rate, as shown in Fig. 8. At higher temperatures the swelling also continued at ~1%/dpa, but without any low swelling rate 'detour', as shown in Fig. 8. In order to produce such a startling result there is obviously some problem with the low temperature data that is not reflected in the data derived at higher temperatures.

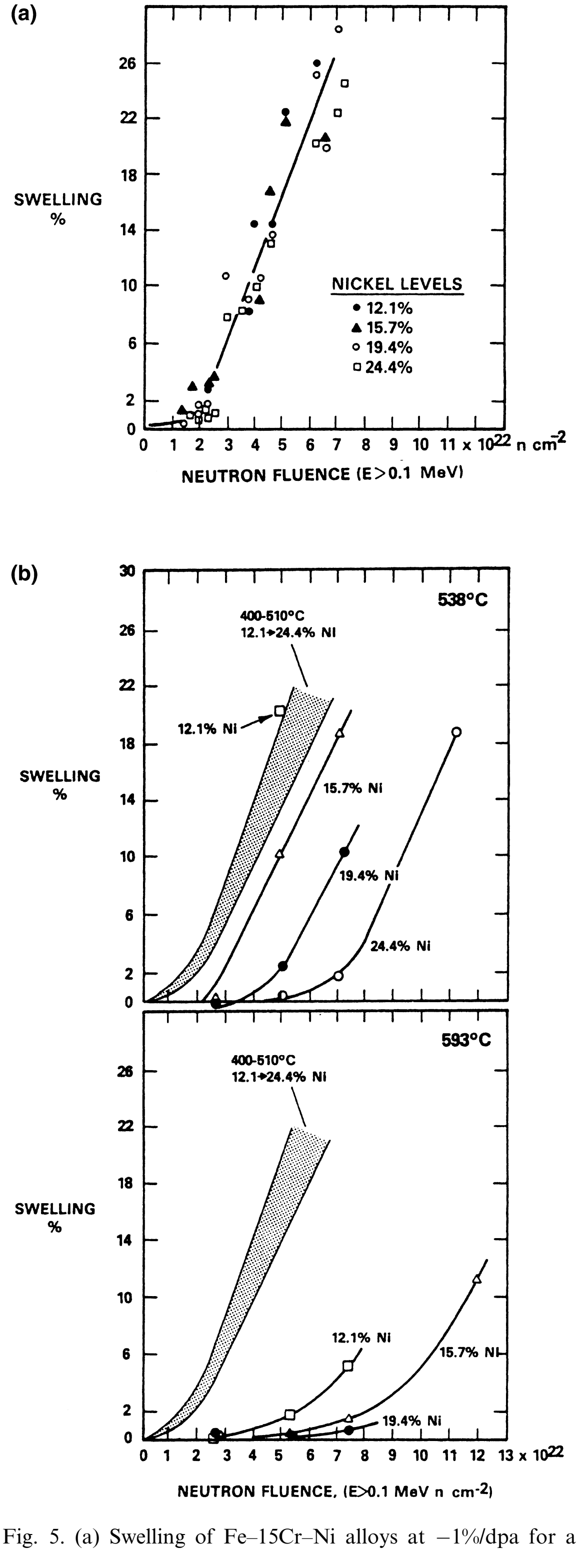

Fig. 5. (a) Swelling of Fe–15Cr–Ni alloys at −1%/dpa for a range of nickel levels and temperatures between 400°C and 510°C in EBR-II; (b) influence of nickel content at higher temperatures [8].

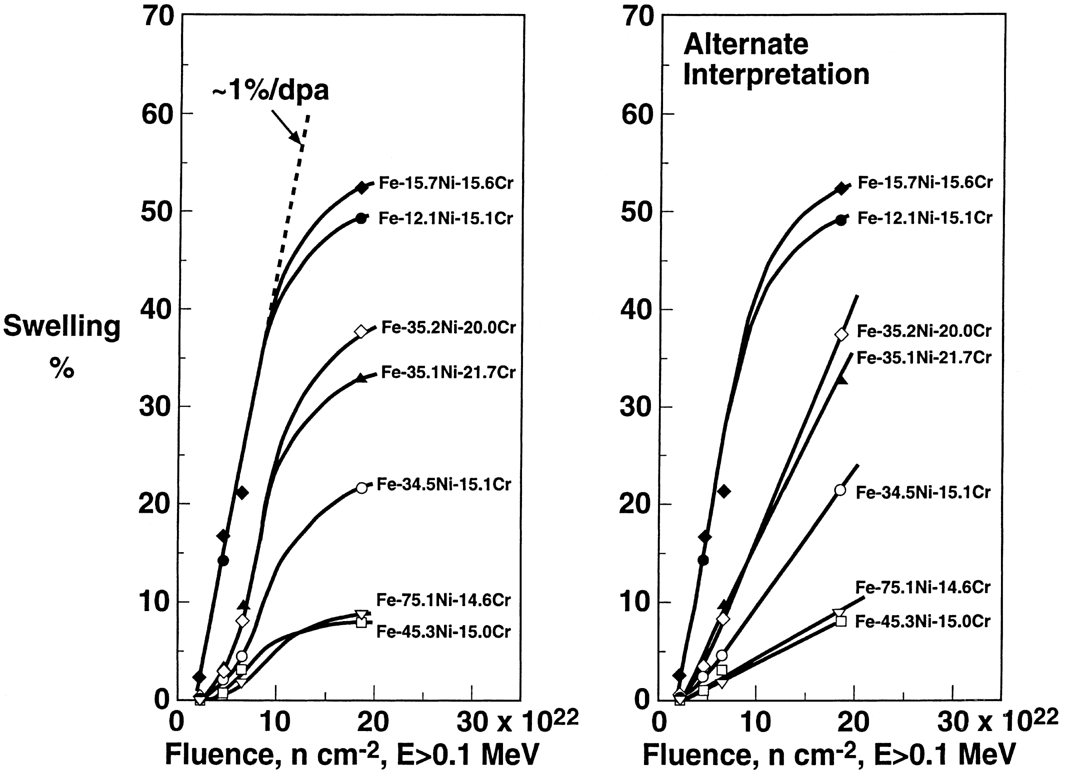

Fig. 6. Previously reported tendency toward saturation of swelling observed in Fe–Cr–Ni alloys at 427°C in EBR-II [8].

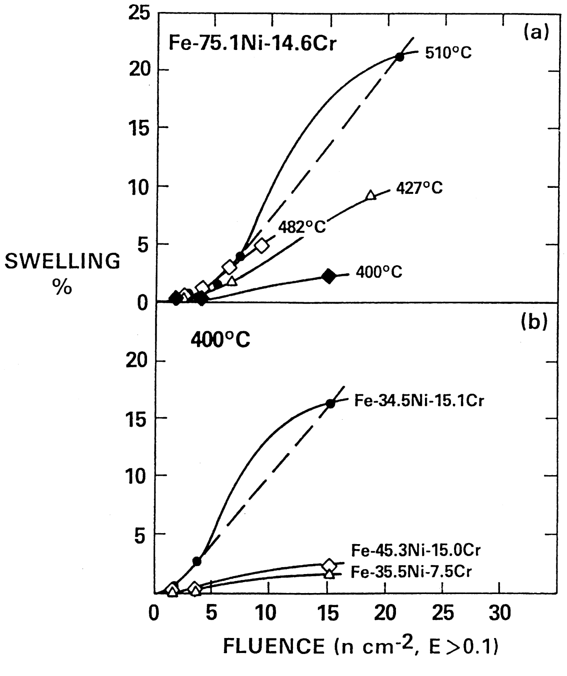

Fig. 7. Previously reported tendency toward saturation observed at higher nickel levels in Fe–Cr–Ni alloys in EBR-II [8].

A careful check of the archived specimen handling records revealed the source of the problem. Due to the large flux gradient along the EBR-II axis, the data at the lower temperatures, being accumulated at the axial extremities of the core, were falling behind in exposure relative to that developed in the in-core, higher temperature capsules. Therefore a programmatic decision was made to divide the low temperature specimens, which came in pairs, and return one-half of each pair to continued irradiation, while the other half was sent for density measurement. Thus the identity codes engraved on each specimen were no longer a unique description of the irradiation exposure. In the shut-down phase of the program the density changes for the lower exposure were erroneously assigned the neutron exposure of the higher dose specimens that were not yet examined. Therefore, the swelling data at the highest neutron exposures in Figs. 6 and 7 were simply misplotted at the wrong exposures. As shown in Figs. 9 and 10, saturation never developed at any temperature in this experiment. A similar conclusion was reached for irradiation at 400°C and 510°C.

As will be seen in a later section, it has been important in this paper to highlight the history of the dose assignment error, since this same error unfortunately

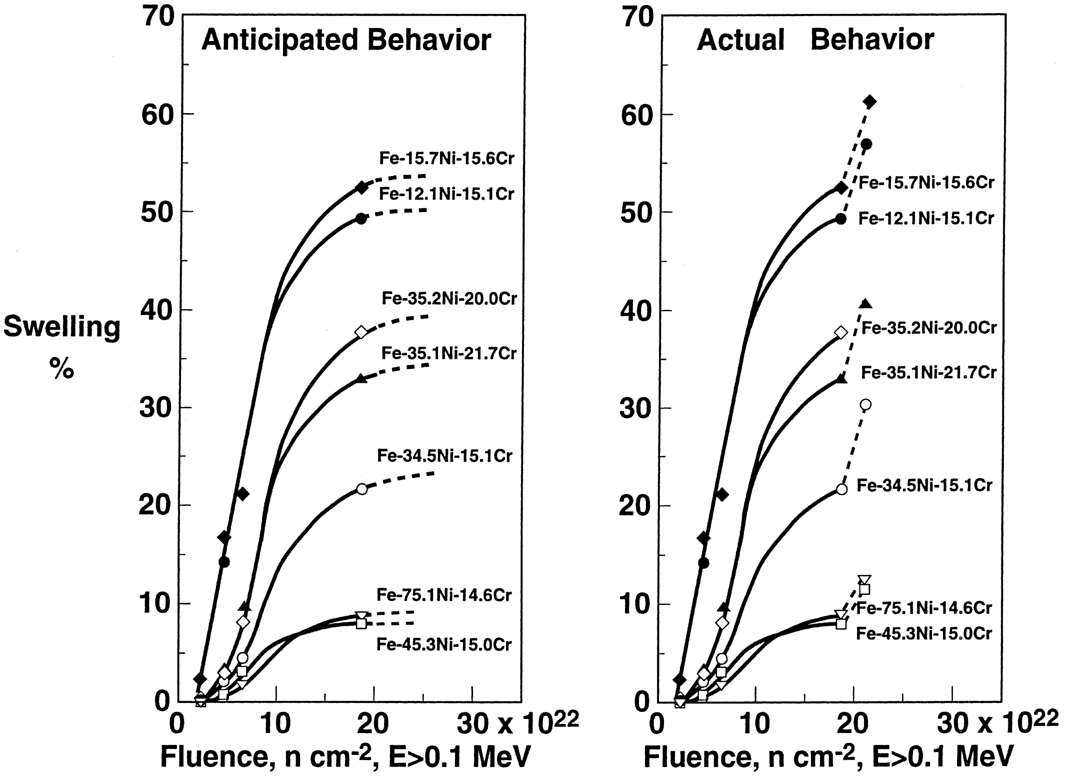

Fig. 8. Surprising swelling behavior at 427°C observed after irradiation to higher neutron exposures, indicating that some error was involved in specification of earlier neutron exposure levels [33].

had a much larger and more important impact on the radiation damage communities' perception of the swelling behavior of ferritic steels.

In summary, there is now no convincing evidence of swelling saturation to swelling levels often approaching 100% in any alloy that succeeds in resisting massive decomposition or phase change of the alloy matrix. This constancy of behavior again allows us to treat the 1% per dpa swelling rate as a truly universal constant for iron-based fcc alloys.

2.3. Effect of thermal–mechanical treatment on swelling of Fe–Cr–Ni ternary alloys

It is a near universally accepted perception that cold-working a metal or an alloy prior to irradiation leads to a reduction in its swelling. In recent history, however, both pure fcc nickel [11,34,35] and pure bcc molybdenum [36] have been shown to exhibit higher swelling after cold-work. In each case, void swelling was difficult to initiate in the absence of network dislocations, leading to relatively long transient regimes. Microchemical evolution is not a feature in these single element metals and therefore the causative mechanism must be physical rather than chemical in nature. The effect of cold-work to increase swelling in these metals was also more pronounced as the irradiation temperature increased. Cold-working followed by aging to produce stable dislocation cells was even more effective to increase swelling.

In solute-free, iron-based ternary fcc metals the microchemical evolution is also not a pronounced part of the swelling process. Might it therefore be possible that cold working, with or without aging, can also increase the swelling of these ternary alloys? Garner, Black and Edwards investigated this possibility, first in a second-generation experiment conducted in EBR-II and second, in another similar experiment conducted in FFTF [37].

As shown in Fig. 11, cold-working of fcc ternary alloys indeed decreases swelling at low temperatures, but tends to increase it at higher temperatures. Cold-work followed by aging tends in general to increase the swelling relative to that of annealed material, and often yields higher swelling.

Even more interesting, however, were two other features of these experiments. First, at lower nickel levels and lower temperatures, the transient regime developed in this second-generation EBR-II experiment was reduced nearly to zero, as evidenced by the attainment of ~30% swelling at ~30 dpa. This signals that there was

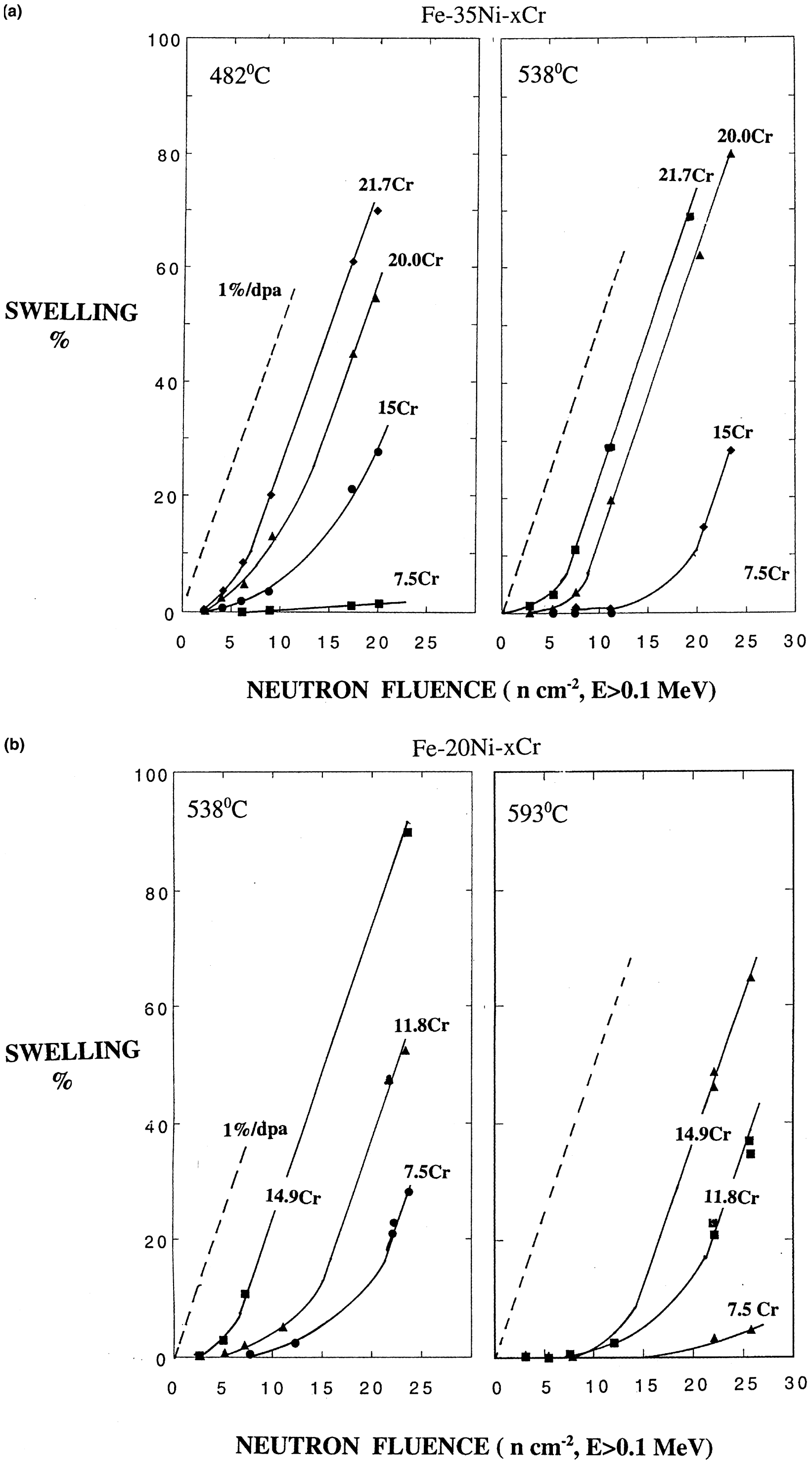

Fig. 9. Lack of saturation in swelling observed in a variety of Fe–Cr–Ni alloys at four irradiation temperatures where no error was made in dose assignments [33].

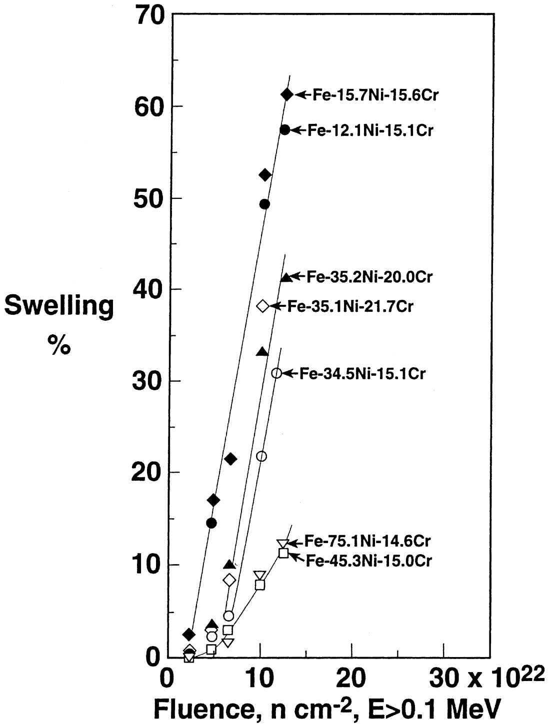

Fig. 10. Corrected swelling behavior of Fe–Ni–Cr ternary alloys at 427°C in EBR-II, showing no hint of saturation [33].

nothing particularly significant about the 10 dpa transient minimum observed in the original first-generation EBR-II experiment. This observation highlights the sensitivity of the transient regime to compositional, fabricational and environmental variables, especially the details of temperature history, and cautions against the expectation that the transient behavior is the same in every experiment, even when nominally identical specimens are used in each irradiation experiment.

Second, in FFTF the identical alloys swelled after a much longer transient regime, reaching only 10–17% swelling in ~36 dpa, implying transient regimes on the order of 19–26 dpa. In general, the swelling reached in FFTF by each alloy was less than that reached in EBR-II at every temperature. There is obviously some persistent difference in the two irradiations that produces such large and consistent differences in transient behavior for identical alloys. A similar behavior was observed in AISI 316 in the previous section, and similar behavior will be shown later for the Fe–Cr binary bcc alloys also included in these same experiments.

The difference is thought to be related to the very different ascent-to-power and operational temperature histories of the two reactor experiments and the resultant effect on void nucleation [37,38]. In EBR-II the temperatures were not actively controlled and fluctuated with power level. On each of the many ascents to power during the conduct of this experiment, the power was brought up in 4–5 stages and the reactor allowed to stabilize before ascending to the next power level. There were sometimes periods of operation at reduced power, superimposed on a tendency for the temperature to fall slowly during long-term irradiation [38]. In FFTF the temperatures were actively controlled in the Materials Open Test Assembly (MOTA) to ±5°C, however, and the operating temperatures were maintained during power fluctuations. Thus, there were no long periods of lower temperature operation in FFTF-MOTA that favored enhanced nucleation of voids. An extensive review of the impact of such temperature history differences on microstructural development was published earlier [38].

It is also important to note that FFTF operated at neutron flux levels that produced dpa rates that were approximately three times higher than that of EBR-II. The duration of the transient regime of swelling is known to be sensitive to the dpa rate, usually increasing with increasing dpa rate at relatively low irradiation temperatures [7].

2.4. Summary of important observations for solute-free fcc alloys

1. The dpa levels attained in some low-temperature subcapsules in an important EBR-II experiment were strongly overestimated, yielding low and misleading estimates of the swelling rates. When the exposure levels were correctly defined, no tendency toward saturation of swelling was observed.
2. Over a range of low temperatures the swelling of ternary alloys with a variety of compositions tend to behave as one material irradiated at one temperature.
3. Outside this composition range, variations in temperature, nickel and chromium result in an increased duration of the transient regime, but all alloys eventually swell at ~1%/dpa.
4. The duration of swelling transient regimes of both simple ternary alloys and AISI 316 stainless steel is in general longer in FFTF-MOTA than in the open core of EBR-II, with the difference attributed to the differences in temperature history and displacement rate.
5. Cold-working can increase rather than decrease swelling, especially when void nucleation is otherwise difficult, and acts to produce a shorter transient duration.

As will be seen in the next sections, each of these observations are pertinent in a much broader sense and will be used to modify our perception of the swelling behavior of iron-based bcc alloys.

3. Swelling of Fe–Cr binary alloys

Having established that there is a near-universal steady-state swelling rate for Fe–Cr–Ni alloys, can we

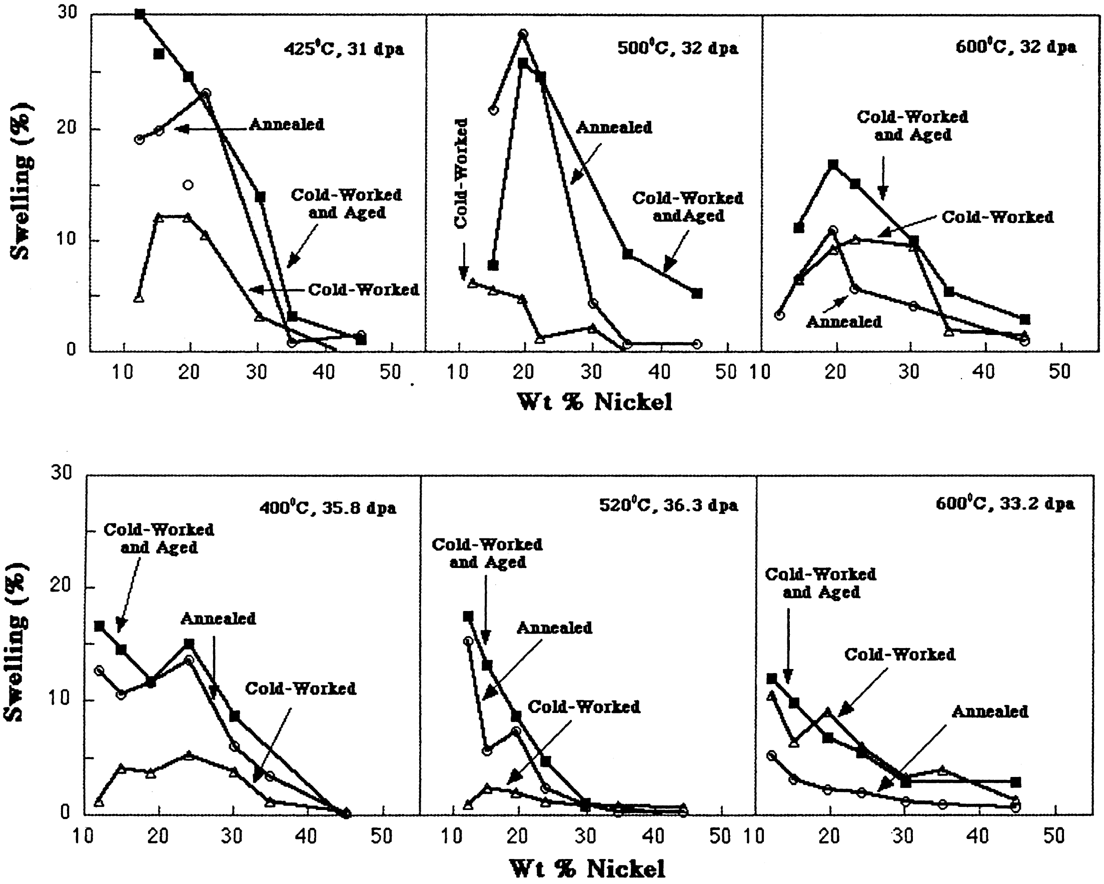

Fig. 11. Influence of starting state, nickel level and temperature on the swelling of Fe–15Cr–Ni alloys in two reactors [37]. EBR-II data are on the top row, FFTF data on the bottom.

define a comparable 'universal' rate for Fe–Cr ferritic alloys? Until recently, the answer to this question would probably be negative, but if there were such a universal rate, most knowledgeable persons would estimate it to be 10–20 times smaller than that of fcc Fe–Cr–Ni alloys.

The primary basis for this perception arises not so much from published data on commercial bcc alloys [39–44], but from swelling data on binary Fe–Cr alloys published by Gelles and coworkers first from EBR-II [45–47] and later from FFTF [48,49]. As shown in Fig. 12 the swelling rates in EBR-II of Fe–Cr alloys at 400–450°C [3] appear to be very low and to be dependent on both composition and temperature. The maximum swelling rate appears in EBR-II to be ~0.06%/dpa in this

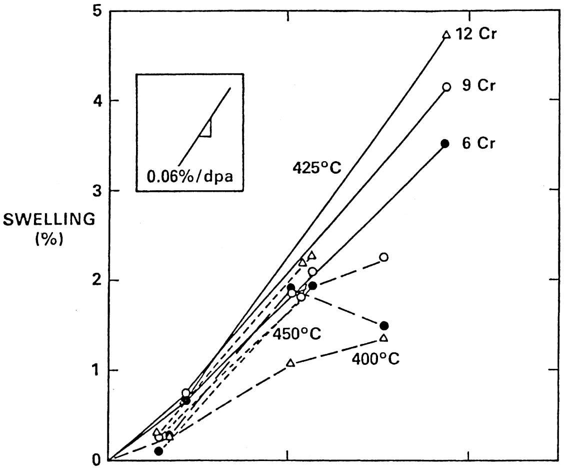

Fig. 12. Published data on the swelling of Fe–Cr binary alloys in EBR-II [45,46].

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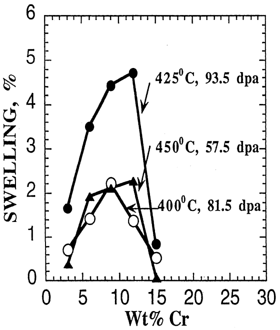
[3] Slight differences quoted in temperature (i.e. 425 vs 427, 450 vs 454°C) for fcc and bcc alloys in the same subcapsule reflect the tendency of Gelles to round off the temperatures from the nominal target temperatures, while still staying well within the uncertainties of the calculated temperatures.

data set. Based only on FFTF data at 200 dpa [50], Gelles concluded the maximum *average* swelling rate was ~0.1%/dpa, not significantly larger than that observed in EBR-II and still ten times smaller than that of fcc alloys.

It is important to note, however, that the EBR-II data on bcc Fe–Cr alloys were developed from specimens included in the very same low temperature packets and subcapsules as that of the fcc Fe–Ni–Cr alloys discussed in Section 2.2. In other words, the dpa assignments for the bcc Fe–Cr alloys are also incorrect at the last exposure level. Figs. 13 and 14 show the impact of correcting the dpa levels, with the magnitude of the correction being different at each irradiation temperature. The maximum average swelling rate at 425°C is observed to increase dramatically.

If we follow the pattern observed in the fcc alloys in this same experiment, we can group together the data over a range of Cr levels and all three lower temperatures in Fig. 15(a), and at higher temperatures in Fig. 15(b). It is now apparent that the general swelling behavior of the bcc Fe–Cr alloy series closely parallels the behavior of fcc Fe–Cr–Ni alloys in the same experiment. All data in the 6–12 Cr range and 400–454°C range appear to behave as if they belong to one alloy being irradiated at one temperature.



- Originally assigned dpa levels are too large.

T, °C	dpa
400	81.5 → 35.1
425	93.5 → 48.3
450	57.5 → 34.8

Fig. 13. Dependence of swelling in Fe–Cr alloys on chromium level, temperature, and dpa in EBR-II for Fe–Cr alloys, showing correction of dpa assignments.

It is not clear, however, if the steady-state swelling rate has yet been reached in this experiment. The steady-state swelling rate is at least 0.2%/dpa and may be higher. At least one more datum would be required at higher exposure to assess the possibility that the rate was not still increasing.

Similar Fe–Cr data generated by Gelles and coworkers [45–47] in FFTF-MOTA to doses in the range 15–200 dpa are shown in Fig. 16. The compositional dependence of swelling is essentially the same at all exposure levels. When plotted in comparison with the EBR-II data as shown in Fig. 17(a), however, it immediately becomes clear that while the transient regime in FFTF is very much longer than in EBR-II, the post-transient behavior is remarkably similar, with maximum swelling rates of at least 0.2%/dpa.

It is obvious that the major influence of chromium level lies in the duration of the incubation period with peak swelling arising as a consequence of the shortest transient regime at ~10% Cr. The formation of the α' phase is thought to be associated with the reduction in swelling at higher Cr levels [42,45].

The question then arises whether commercial ferritic/martensitic stainless steels will also exhibit the same behavior as the Fe–Cr binaries. In general most reported exposures for commercial steels never exceed 100–150 dpa and data usually exist at only one or two exposure levels. Therefore the best comparison to answer this question is the swelling observed by Toloczko, Garner and Eiholzer in HT9 (with 12% Cr) using pressurized tubes at ~400°C in FFTF-MOTA [51]. Seven dpa levels

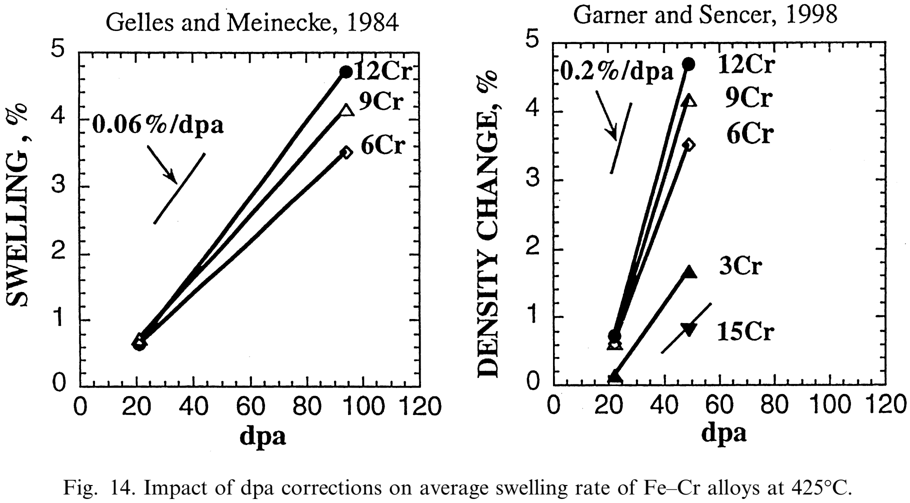


Fig. 14. Impact of dpa corrections on average swelling rate of Fe–Cr alloys at 425°C.

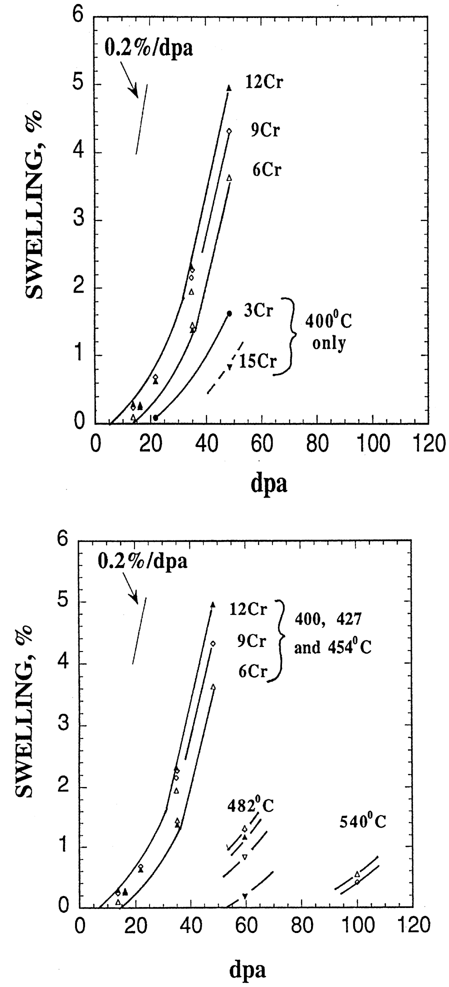


Fig. 15. Compilation of Fe–Cr data from EBR-II at all available temperatures, showing behavior similar to that observed in Fe–Cr–Ni alloys.

were involved in this experiment. These tubes resisted swelling for a long time but swelled from ~1% to ~3% at 208 dpa over a stress range of 0–200 MPa [51], as shown in Fig. 18. The alloy 9Cr–1Mo was also irradiated in this experiment and reached 2–3% swelling at 208 dpa. As shown in Fig. 17(b), both of these swelling ranges are relatively consistent with the Fe–(9,12)Cr behavior shown in Fig. 17(a) with some additional influence of solutes to extend the transient. The dependence of swelling in HT9 on applied stress is yet another indication of the inherent instability associated with the end of the transient regime.

Similar creep tube studies were conducted by Toloczko and Garner on various heats of HT9 irradiated side-by-side at ~400°C to 165 dpa [52,53]. These steels exhibited a heat-to-heat variability in the onset of swelling with an abrupt increase in swelling rate in the

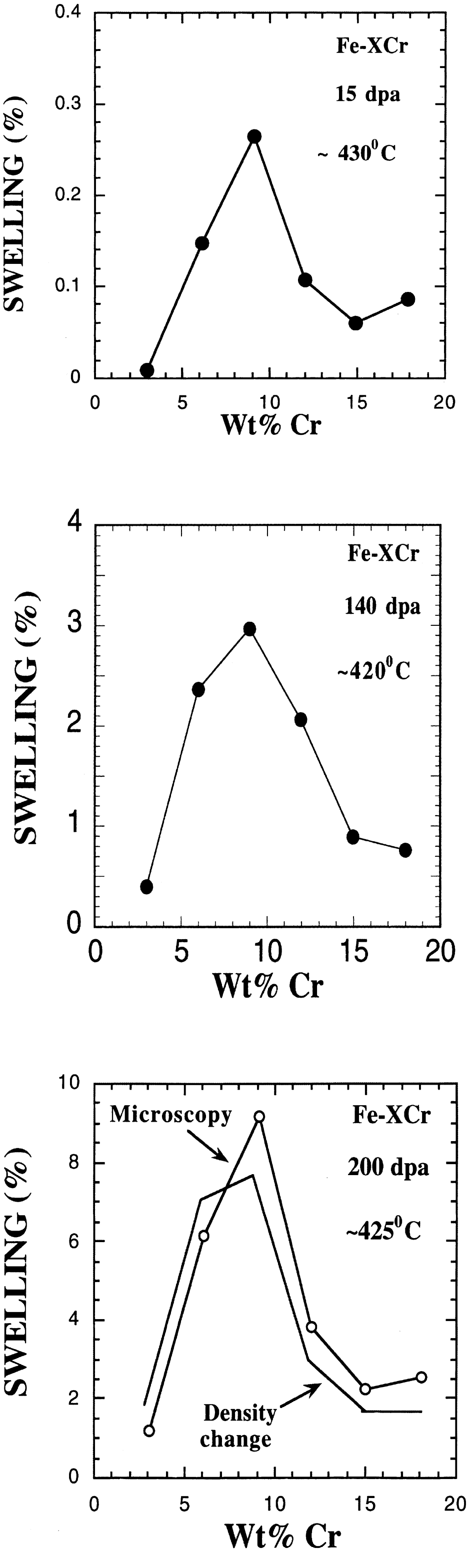


Fig. 16. Swelling of Fe–Cr alloys irradiated in FFTF-MOTA under active temperature control [45–47].

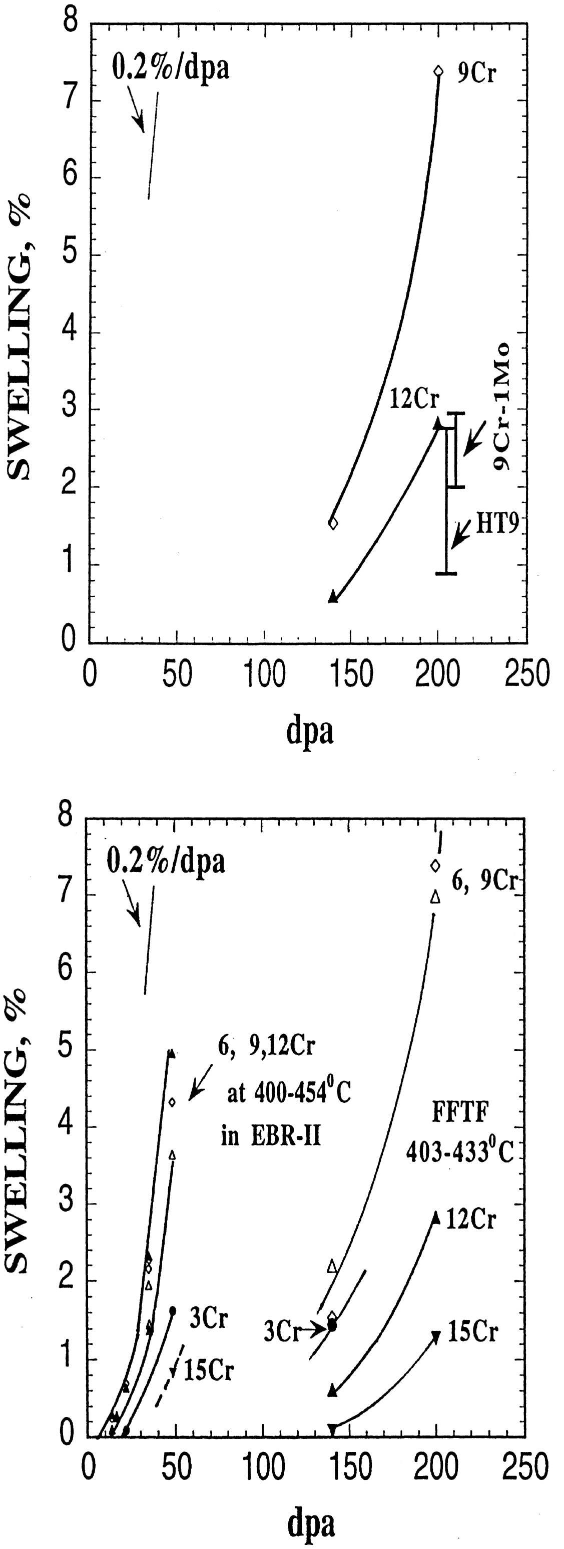


Fig. 17. (a) Comparison of swelling of Fe–Cr alloys in EBR-II and FFTF-MOTA; (b) swelling observed in HT9 and 9Cr–1Mo in FFTF-MOTA [51–53].

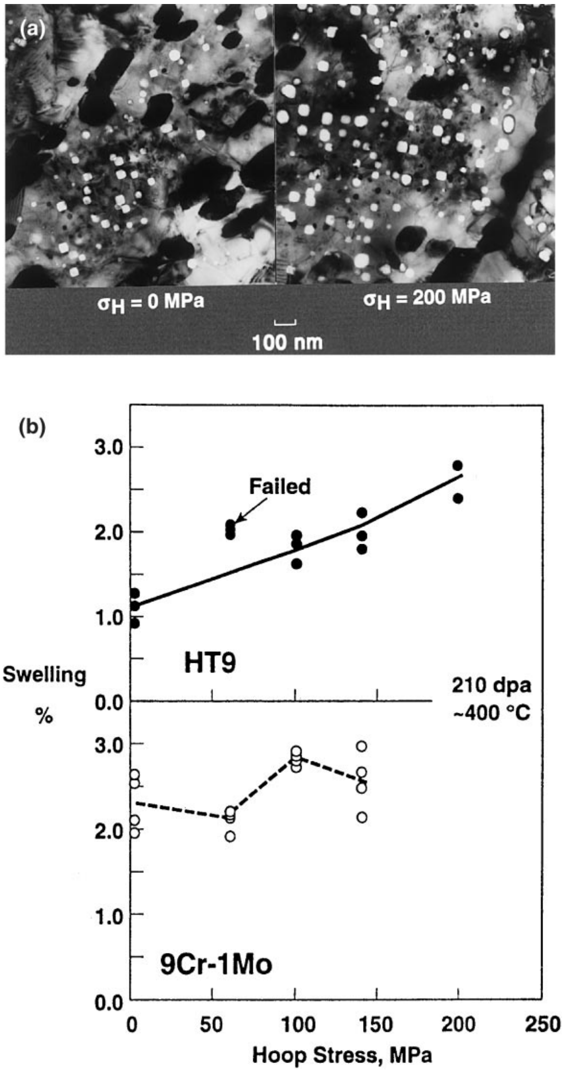


Fig. 18. Voids observed in HT9 pressurized tubes irradiated in FFTF to 208 dpa at ~400°C; (b) swelling observed in both HT9 and 9Cr–Mo as a function of applied stress [51].

more swelling-prone heats at ~100 dpa, behavior which also agrees with that of comparable Fe–Cr alloys irradiated in FFTF and shown in Fig. 17.

The foregoing discussion thus leads us to the conclusion that typical solute-bearing ferritic/martensitic steels can exhibit very long transient regimes of swelling, especially when irradiated under well-controlled temperature conditions, but eventually they will swell at an accelerated rate of ~0.2%/dpa and possibly greater.

3.1. Swelling of pure iron and Fe–Cr alloys

If Cr additions influence the transient regime of swelling, it would be very useful to observe swelling of pure iron at the same time, but none of the Fe–Cr experiments discussed in the preceding section included pure bcc iron. This is unfortunate, but if we extrapolate downward from 3% Cr it would be reasonable to conclude that pure iron would have very low swelling and thereby a very low swelling rate. This conclusion is probably misleading, however, even though it would require another inflection point for the swelling curve in the 0–3% Cr range to yield significant swelling for pure iron.

There was one comparable charged particle bombardment experiment which included both Fe and Fe–Cr alloys. The results of this Ni^+ ion bombardment experiment are shown in Fig. 19 and at first glance appear to support the assumption of low swelling in pure Fe. Johnston and coworkers [54], however, were rather concerned that the use of Ni^+ instead of Fe^+ ions may have distorted the swelling result, since this deposited a Gaussian distribution of nickel with a maximum concentration of 2.7%, peaking just 200 nm behind the position of peak damage. A smaller amount, 1.5% Ni, was deposited at the peak damage region, but it is expected that radiation-induced mixing and enhanced diffusion would have broadened the Gaussian profile somewhat. Whatever the time-averaged nickel distribution over the irradiated volume, however, it was not insignificant and there was some concern that it would have a strong effect on the swelling.

To investigate the effect of the deposited nickel, a second and more limited series of Fe–2Ni–Cr alloys were irradiated by Johnston. In going from ∼2% final deposited nickel to ∼4% total nickel there was a dramatic drop in swelling, as also shown in Fig. 19. This implies that the addition of the first 2% Ni by deposition probably had a very large effect to reduce swelling, and raised the possibility that both Cr and Ni additions may strongly depress the swelling of pure iron.

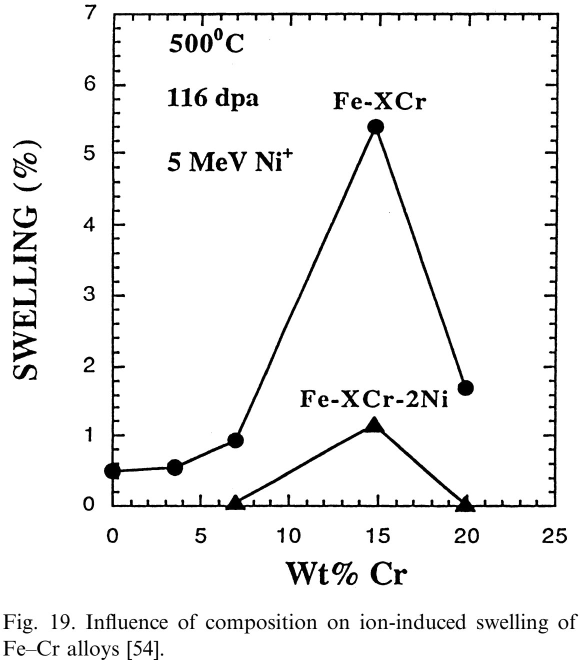


Fig. 19. Influence of composition on ion-induced swelling of Fe–Cr alloys [54].

The possibility of suppression of swelling in pure iron by Cr addition may be explored by examining several other experiments. Little and Stow [55] showed that, after neutron irradiation to 30 dpa of annealed pure iron and various Fe–Cr alloys, the first 1% Cr addition depressed swelling strongly, as shown in Fig. 20. The maximum swelling of annealed iron was not large, however, only ∼1% at 30 dpa, but illustrated the inflection behavior in the 0–3% range that was suggested earlier. The typical increase of swelling at Cr levels above 1% that was observed in other experiments was also observed in this study.

A similar inflection and suppression behavior was demonstrated by Horten and coworkers [56] in 'triple-beam' (D_2^+, He^+, Fe^+) irradiation to 10 dpa of annealed Fe and two Fe–Cr alloys at 800 K, but the levels of swelling were small (0.22% in Fe, 0.0% in Fe–5Cr and 0.16% in Fe–10Cr). In a related study to higher doses, Horten and Bentley produced ∼2.5% swelling in Fe–10Cr at 100 dpa and 850 K [57], demonstrating that binary alloys could eventually reach significant levels of swelling during ion bombardment.

Another more graphic suppression result was provided by Ohnuki and coworkers who irradiated pure iron and Fe–13Cr at 525°C to 118 dpa with 200 KeV C^+ ions [58]. This produced very large swelling, 14%, in pure iron but only <0.5% in Fe–13Cr. In this case it appears that iron can be induced to swell easily if the conditions are favorable. Ohnuki noted that there was considerable carbon addition during the irradiation, however, leading to extensive formation of Fe_3C precipitates, which suggests that carbon can accelerate swelling at such high temperatures. Garner and Sencer demonstrated this possibility to be true in EBR-II irradiation of Fe–12Cr and Fe–12Cr–0.1C alloys at 482–593°C and dpa levels of 74–131 dpa [59]. Swelling jumped from 0–1% to 6–10% at these irradiation conditions simply by adding 0.1%C. This behavior strongly suggests that an abrupt termination and shortening of a very long transient period had occurred. Together, these results imply that both pure iron and Fe–Cr alloys have potentially high intrinsic swelling rates that are camouflaged by very long transient regimes.

In general, most irradiations of annealed pure Fe by either neutrons or charged particles have yielded relatively small amounts of swelling and therefore do not admit the possibility of high steady-state swelling rates [60–64]. Neutron-induced swelling at relatively low exposure appears to peak at ∼400°C [64], as shown in Fig. 21, indicating a possible temperature dependence of the duration of the transient regime. While the temper-

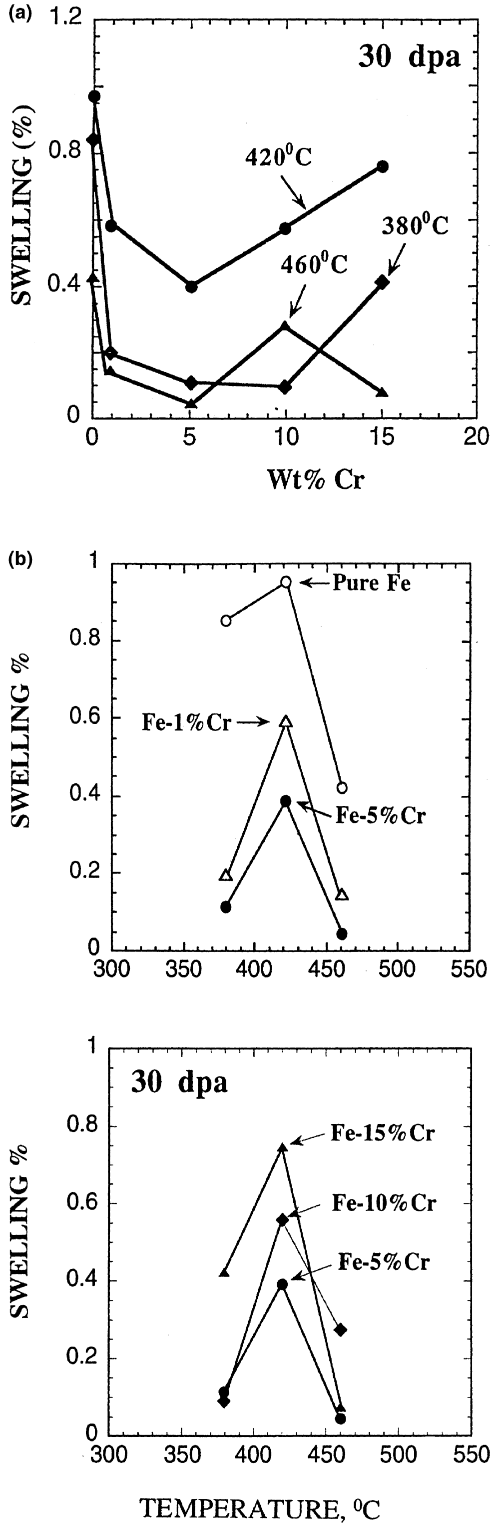


Fig. 20. Effect of (a) chromium level and (b) irradiation temperature of neutron-induced swelling of Fe–Cr alloys at 30 dpa in the DFR fast reactor [55].

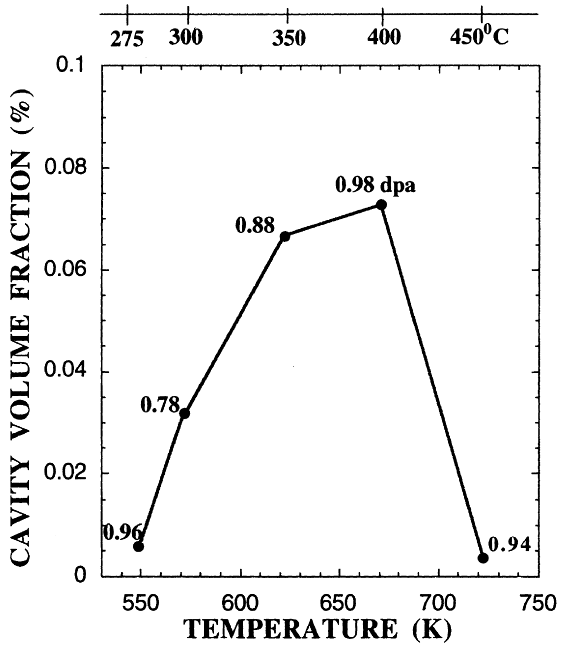


Fig. 21. Temperature dependence of pure iron at ~1 dpa in the ORR reactor [56].

ature dependence appears to be very pronounced in this latter study, it would take only a very slight dependence at low dpa levels to produce such a sharp peak with temperature in these data.

There was an indication in an electron irradiation study by Kitajima and coworkers [61], however, that swelling at low temperatures can be significantly increased by cold-working. This is an important and previously overlooked clue, since the effect of cold-working has not been studied in most pure metals, especially those with bcc structure.

In a study published recently by Porollo and coworkers [65], however, pure iron and various Fe–Cr alloys were irradiated in the BR-10 fast reactor, with the specimens in the ~10% cold-worked condition, reaching dpa levels of 5.5–7.1 dpa at 400°C. As shown in Figs. 22 and 23, pure cold-worked iron swelled 3% at 6.2 dpa as confirmed by microscopy. As might be expected, addition of 2% Cr to iron led to a dramatic suppression in swelling, but unexpectedly produced a large increase in the loop density. There was also a small peak in the swelling at 12% Cr, in agreement with the results of other studies.

The latter experiment suggests that cold-working of pure Fe led to an average swelling rate of ~0.5%/dpa, approximately one-half that of fcc Fe–Cr–Ni alloys. If any transient was involved, the post-transient rate would be >0.5%/dpa. Such a finding begs for confirmation, and the lead author of this paper and the group led by Porollo plan to examine in the near future a second discharge of this experiment at ~20 dpa.

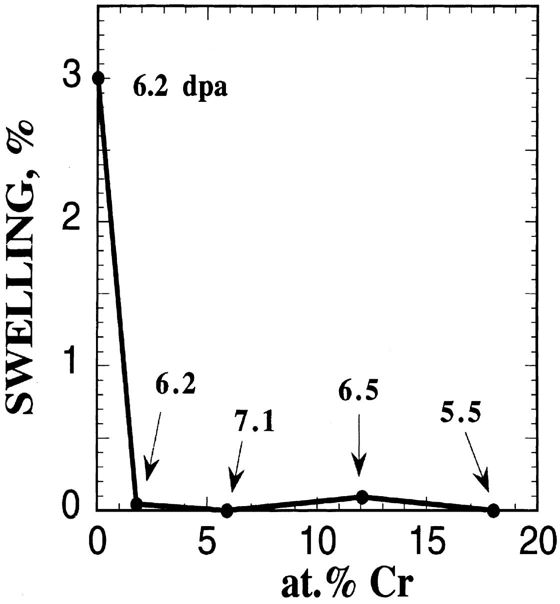


Fig. 22. Swelling observed in 10% cold-worked pure iron and Fe–Cr alloys in the BR-10 fast reactor at 5.5–7.1 dpa and 400°C [65].

Another experiment by Agapova and coworkers in the BOR-60 fast reactor at 460°C and ~37 dpa yielded 1.5% swelling in annealed pure iron and 1.0% swelling in 10% cold-worked iron [66]. In this higher temperature case, cold-working did not accelerate swelling. On the other hand, the reduction due to cold-work was not very large.

Based on the foregoing sections the possibility exists, that within a factor of two, the steady-state swelling rates of Fe and Fe–Cr alloys may eventually approach the rate of Fe–Cr–Ni alloys. The large differences in total swelling for fcc and bcc alloys are a consequence of the transient regimes being very much longer in the bcc metals. Johnston alluded to this possibility when he noted that Ni^+ ion irradiation of Fe–15Cr yielded a very large transient regime ($\geqslant$ 100 dpa) and ~6% and ~21% swelling at 116 and 240 dpa, respectively [54]. This yields an average swelling rate of 0.12%/dpa, which "are not sufficient data points to say that a steady-state swelling rate has been achieved". This is a particularly important observation when it is considered that the steady-state swelling rates developed in 3–5 MeV Ni^+ ion-bombardment experiments are known to be suppressed by approximately a factor of five due to the injected interstitial effect [67].

This conclusion was reached in an extensive comparison experiment between three ion bombardment groups (including Johnston's group) where it was shown

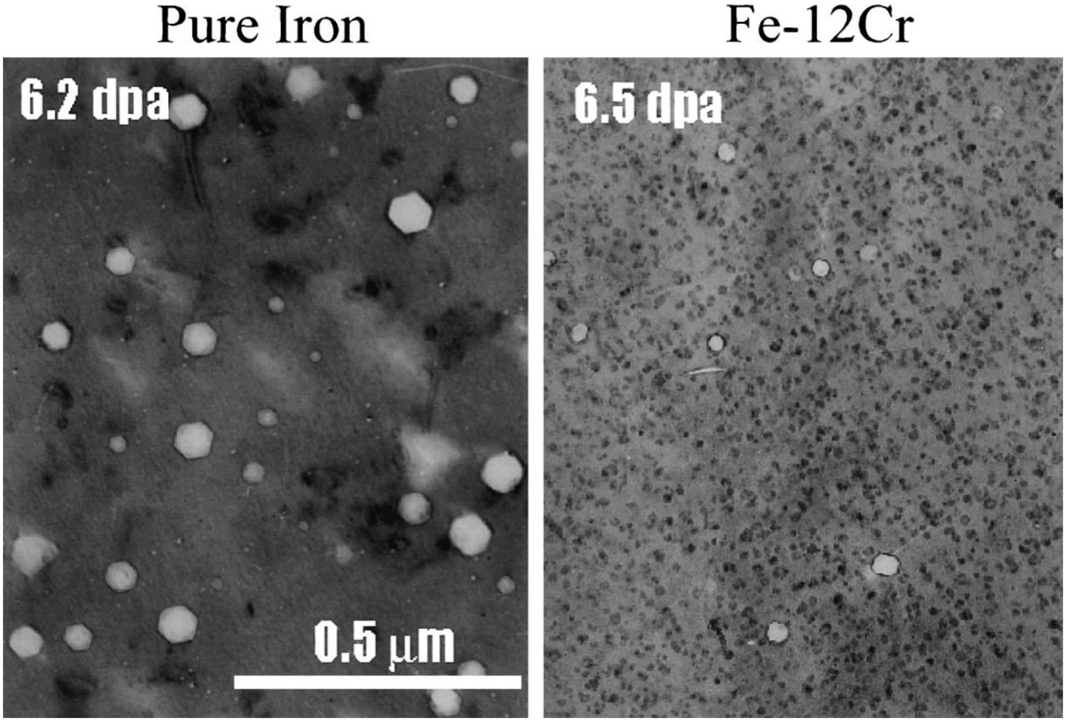


Fig. 23. Microstructures of pure iron and Fe–12Cr at ~6 dpa and 400°C [65].

that the Ni^+ ion-induced swelling rate in annealed Fe–15Cr–25Ni was reproducible by all groups but was only 0.2%/dpa [68]. This implies that the swelling rate of 0.12%/dpa in Fe–15Cr observed by Johnston at 116–240 dpa is at about half of that observed in the comparable fcc alloy. Although the swelling rate appears to be suppressed in both alloys by the injected interstitial effect, these results are again consistent with a possible difference of only a factor of two in the steady-state swelling rates of the two crystal structures.

3.2. Irradiation creep

An increasing amount of irradiation creep data on ferritic steels, especially HT9, has recently become available [51–53]. There has also been better resolution of the influence of important variables such as crystal structure, helium/dpa rate, displacement rate, thermal–mechanical treatment and composition on irradiation creep [7,11,24,51–53,69,70]. In summary, when the two most important creep coefficients, B_0 (describing creep in absence of swelling) and D (describing swelling-enhanced creep) are evaluated from data on various commercial and developmental alloys, it appears that the D coefficient is essentially equal for fcc and bcc steels at $\sim 0.6 \times 10^{-2}$ MPa^{-1}, reflecting the physical relationship between creep and swelling, but the B_0 coefficient of bcc steels appears to be about one-half that of fcc steels at $\sim 0.5 \times 10^{-6}$ $\mathrm{MPa}^{-1}\mathrm{dpa}^{-1}$. This conclusion is not as well supported as conclusions drawn from comparison of the swelling rates, but it does imply once again that the defect production and aggregation mechanisms in bcc iron-based alloys may be only a factor of two different in net efficiency to create microstructural alteration with consequent changes in dimension. In general terms then, both swelling and creep appear to be telling us the same story concerning the effect of crystal structure in iron-based alloys.

4. Discussion

Not only do the foregoing results have the potential to affect the microstructural modeling efforts that are one of the focal points of this workshop, but there are potentially strong engineering and design consequences of these conclusions as well.

The realization that bcc steels can eventually swell at rather high rates, with the primary swelling resistance residing more in the transient regime rather than in the steady-state swelling rate, requires that a larger degree of uncertainty must be allowed for the onset of swelling in previously uncharted irradiation environments. This consideration is well illustrated by the very large difference in transient duration in the not-so-isothermal irradiation conducted in the open-core EBR-II materials tests or FFTF fuel pin tests as compared to the nearly-isothermal tests conducted in FFTF-MOTA. It is important to remember that most operating reactor systems experience fluctuations in power and thereby temperature. This uncertainty is also foreshadowed by the heat-to-heat variability observed in the swelling of HT9 at 400°C and 165 dpa [52,53].

There are other possible ramifications of the results of this study. First, the data indicate that there exists a significantly larger resistance to void nucleation in bcc alloys, and that the origin of this resistance is sensitive to alloy composition and thereby also to microchemical evolution. Microchemical evolution in fcc systems has been shown to be very sensitive to many environmental factors such as temperature history, applied stresses, helium and hydrogen generation rates, as well as dpa rates, as reviewed in Ref. [7]. The study of such 'history effects' and their ability to accelerate swelling has led to the formulation of a general principle that 'the longer the transient regime of swelling, the greater the possibility and magnitude of potential decreases in transient duration and thereby acceleration of swelling' when new test environments are explored. The abrupt termination of long transients as shown in several of the preceding figures is another indication of the inherent instability of such systems.

Second, note that there is a suggestion in Fig. 17 that the transient regime of swelling in Fe–Cr alloys increases with temperature in a manner comparable that of fcc alloys. Sencer and Garner have recently shown that, in another irradiation series conducted in EBR-II, swelling of Fe–Cr alloys eventually accelerates at higher exposures for temperatures as high as ~600°C [59]. Since swelling was previously not anticipated at such high temperatures, other researchers have not carried irradiation of bcc Fe-base alloys at these temperatures to the high exposure levels required to initiate swelling. Sencer and Garner also showed that irradiation at lower dpa rates tended to shorten the transient regime compared to that produced in Fe–Cr alloys at higher dpa rates.

Third, since the resistance to swelling lies primarily in void nucleation during the transient regime, the onset of swelling in bcc steels may be much more sensitive to the high levels of helium and hydrogen generated in light water moderated reactors, fusion reactors, or spallation neutron environments [71], the latter two of which are currently unexplored territory with respect to neutron spectra, temperature, flux, and stress history. The majority of all high fluence data on bcc steels has been developed in fast reactors which have very low He/dpa ratios. In fusion and spallation environments, the helium to dpa ratios can be very large, 5–15 and ~150 appm/dpa, respectively, with hydrogen generation rates an order of magnitude higher. Gelles provided some support for this possibility in a comparison of HT9 irradiated in EBR-II and HFIR, where in the latter reactor, the early onset of void growth was attributed to higher helium levels [46,72].

On another issue, it is instructive to note that while the early Fe–Cr neutron data implied a factor of 10–20 difference in swelling rates, one of the earliest theoretical studies of the difference based only on crystal structure differences reached a conclusion much closer to that of the current study. Sniegowski and Wolfer [73] calculated crystal-dependent differences in dislocation and void biases for α-Fe and γ-Fe, coming to the conclusion that austenitic alloys might swell at a maximum of 1.4%/dpa and ferritic alloys at a maximum of 0.23%/dpa. Both of these estimates are remarkably close to the rates observed in the data derived from irradiations in EBR-II and FFTF.

The crystallographic basis for this predicted difference in swelling rates was primarily the differences in relaxation volumes and activation energies for diffusion in the two crystal systems for both interstitials and vacancies, which to the first order are dependent primarily on crystal structure and to second order on compositional considerations. This latter consideration was not considered in any depth in the study of Sniegowski and Wolfer, but their conclusions are consistent with the experimental conclusions reached in this paper.

Finally, for purposes of crystallographic modeling where the influence of compositional variations and microchemical evolution are usually ignored, it should be assumed that the crystallographic dependence of defect production and aggregation should not exceed a factor of two to four. Differences of larger magnitude are not supported by available data at high neutron exposure.

5. Conclusions

A number of previously accepted perceptions concerning the swelling of iron-base fcc alloys have been shown in this paper to not always be correct. These misperceptions include the effect of cold-work, the extent of the temperature regime of swelling, and the possibility of saturation in swelling magnitude.

Whereas it was previously perceived that the steady-state swelling rates of iron-based ferritic and ferritic/martensitic steels were perhaps 10–20 times smaller than that of iron-based fcc steels, it now appears that the difference may be as small as a factor of two to four. Due to very long transient regimes of swelling in bcc alloys, this possibility has not been previously recognized. The temperature regime of swelling for bcc alloys also appears to extend to much higher temperatures than previously anticipated. It is further concluded that well-controlled materials irradiation tests will probably yield conservatively low amounts of swelling for both fcc and bcc alloys compared to that where the temperature history is more typical of normal reactor operation.

It thus appears that differences in bcc and fcc alloys in irradiation creep rates in the absence of void swelling support only a factor of perhaps two in defect production efficiency to induce dimensional change by creep. Therefore, efforts to model the kinetics of defect production and aggregation in fcc and bcc alloys must incorporate this smaller than originally perceived difference in steady-state swelling and creep rates.

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