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The influence of neutron spectrum and irradiation history on microstructural evolution in fusion structural materials

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Abstract

The impact of solid transmutant generation and temperature transients during irradiation are new research subjects for application of fission neutron irradiation data to fusion materials development. Fundamental research for these issues is in progress, and includes such approaches as alloying the transmutant elements before irradiation and varying temperature irradiation with charged particles. In addition, novel uses of fission reactors are under way using neutron spectral changes to control solid transmutation, and highly controlled periodically-variable temperature irradiation. The understanding of microstructural processes in such complex and the variable irradiation conditions is growing.

1. Introduction

The current absence of a fusion neutron irradiation facility requires that various fission reactors should be used for testing the radiation response of fusion materials. The translation of the fission neutron irradiation data into the predicted materials response in fusion conditions has been pursued largely from mechanistic viewpoints only.

The neutron energy spectrum is the principal difference between the irradiation conditions in fission and the fusion reactors. The spectral effects on radiation damage can be categorized into the recoil energy effect and the transmutation effect. While most previous transmutation studies have concentrated on helium and hydrogen generation, increasing emphasis has been placed on the influence of solid transmutation products.

Recently, Garner and coworkers examined the transmutation of V, Mo, Re, W, Hf, Cu and others in fast reactors, mixed spectrum reactors, fusion reactors and spallation devices conditions [1–3]. The calculations showed significant differences in the transmutation rate between fission and fusion reactors. Furthermore, the spectral difference among fission reactors also leads to large differences in the transmutation rate. These facts complicate the fission–fusion correlation methodology, and suggest the need for systematic single-variable experiments to determine the impact of transmutation effects.

After Kiritani predicted and then demonstrated that minor temperature transients during reactor neutron irradiation could strongly influence the resulting microstructural evolution [4,5], various irradiation experiments have been carried out in Japan Materials Test Reactor (JMTR) in highly controlled steady or periodically-variable temperature conditions [6–10]. Consequently, the understanding of the mechanisms of temperature history effect at low dose levels (~0.2 dpa) was largely enhanced. Varying temperature irradiation experiments were also carried out with fusion neutrons [11], ions [12,13] and electrons [14], supporting the mechanistic understanding. Modeling and theory based on those studies may make it possible to predict the temperature history effect at high exposure levels. However, such model-based prediction should be verified experimentally. Therefore, varying temperature irradiation experiments at high neutron fluence levels are highly needed.

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In the Japan–USA fusion cooperation program (JUPITER project [15]), two subtask groups are organized which promote experiments on the above subjects. They are (1) "Combined Transmutation Effects" coordinated by Ohnuki and Garner, and (2) "Varying Temperature Irradiation Experiment" coordinated by Muroga and Garner/Zinkle. Under this framework, advanced irradiation experiments using HFIR have been either performed or designed. A number of studies, including charged particle irradiations, are also going on which are designed to support those experiments.

This paper reviews the present status and the future plan of research on the solid transmutant production effects and the varying temperature irradiation effects with emphasis on the activities in the JUPITER project.

2. Effect of solid transmutant production

2.1. Procedure of solid transmutant studies

For designing systematic solid transmutant studies, it is worthwhile to recall the various experimental approaches adopted for transmutant helium effect in fusion conditions [16–20], which are summarized as follows.

1. Fundamental studies of helium in materials, such as diffusion, interaction with defects, accumulation at grain boundaries and so on.
2. Injection of helium prior to neutron or charged particle irradiations.
3. Irradiation with high helium production rate such as nickel-bearing alloys in HFIR. Prediction of helium effect in fusion condition was attempted based on comparison of data at both high and low helium production rates.
4. Tailoring of helium production by either spectral tailoring or isotopic doping during neutron irradiations to achieve the fusion He/dpa condition. This approach includes co-injection of helium during charged particle irradiations.

An efficient experimental path for studies on solid transmutant effects may be summarized analogously as follows.

(1′) Fundamental studies of the transmutant elements in materials such as phase stability and defect-solute interactions.

(2′) Irradiation of materials alloyed with expected transmutant elements.

(3′) Accelerated transmutation during irradiation.

(4′) Tailoring of the transmutation by spectral change of the neutrons.The procedures (3′) and (4′) are possible for some transmutant elements using mixed-spectrum reactors.

In the framework of the JUPITER project, some experiments were carried out for copper and vanadium. They fall under categories (2′) and (3′) in the scheme shown above. A brief review of these experiments along with some fundamental studies are presented in the following.

2.2. Production of nickel and zinc in copper

Fig. 1 shows the production of nickel and zinc from copper calculated in FFTF (midplane), HFIR (PTP position) and fusion (STARFIRE) conditions [2,21]. The production of these solutes during irradiation can produce a pronounced decline in both the thermal and electrical conductivity of copper. Experimental results of electrical conductivity change after irradiation have been compared with the calculated contribution by transmutant nickel and zinc in several studies [21–23].

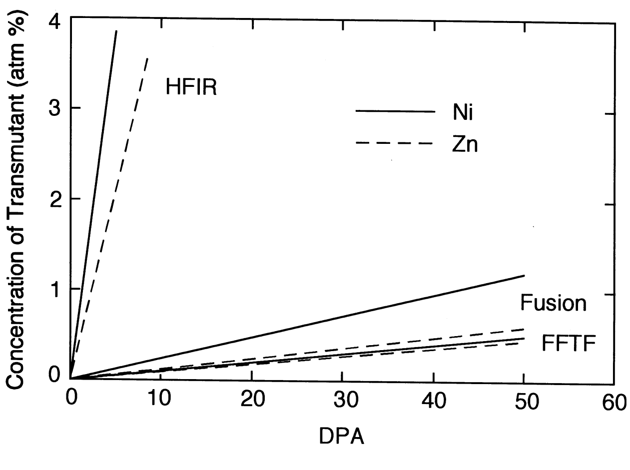

Fig. 1. Transmutant products calculated for copper in FFTF (midplane), HFIR (PTP) and fusion first wall (STARFIRE) [2,21].

Various studies of the effect of nickel solute on the radiation-induced microstructures in copper have been carried out. Studies of the zinc effects, on the other hand, have been limited. Electron irradiations have been carried out to examine nickel effects on dislocation evolution. Loop growth rate [24] and loop saturation density [25] were measured. The common conclusion from these two studies was that clustered nickel should trap interstitials, enhancing loop nucleation during irradiation. The observed loop densities in Cu and Cu–5%Ni obtained at a much lower damage rate with 750 MeV protons were in good agreement with the extrapolation of the electron data [25,26]. The effect of zinc solute on dislocation loop formation and growth was, on the other hand, small [25]. Electron irradiation also showed pronounced enrichment of nickel [25,27] and depletion of zinc [25] at grain boundaries.

Neutron irradiation of Cu and Cu–5Ni in FFTF at ~700 K to very high fluence showed only a small effect of nickel on void swelling [28]. However, lower dose irradiation at various temperatures showed that the nickel effect is highly temperature dependent [29,30]. At high temperature, the void size in Cu–5Ni increased significantly resulting in highly enhanced swelling. However, since the lowest temperature achievable in FFTF (~640 K) is already in the high-temperature region for void swelling in copper, ion irradiations were carried out over a wide temperature range to examine the overall temperature dependence [31]. Fig. 2 summarizes the neutron and ion irradiation data. Pure copper data from the ORR fission reactor [32] are also plotted in Fig. 2, which shows a systematic differences in the swelling temperature regime between neutron and ion irradiations by about 100 K, due to the difference in the damage rates. More importantly for the present discussion, the data shown in Fig. 2 also suggest that the addition of nickel and zinc shifts upward the void swelling temperature regime both in the neutron and ion irradiations.

A recent irradiation in HFIR-PTP as part of the JUPITER project provided data at a high solid transmutation rate for Cu and Cu–3.5Zn [33]. Post-irradiation chemical analyses showed that about 3.2%Ni and 3.4%Zn were produced by irradiation to 10.4 dpa [34]. Fig. 3 compares the void density and swelling after FFTF and HFIR irradiation. The figures show that, whereas the void densities of Cu–3.5Zn specimens irradiated in HFIR and FFTF are close to each other, the swelling levels are very different particularly when the higher dose for the HFIR irradiation is considered. It is understandable that the void densities of HFIR and FFTF specimens are similar to each other, because the void density would saturate during the early stages of irradiation [35] where the concentration of the solid transmutant is still low. The solid transmutants would mainly affect the swelling behavior at the later period of the irradiation.

2.3. Chromium production in vanadium

Fig. 4 shows chromium production from vanadium as calculated by Greenwood and Garner for HFIR-PTP, FFTF and Fusion conditions [1,36]. Also included in Fig. 4 are the chromium production in HFIR-RB* with Hf and Eu shield [1,37]. Chromium generation rate is much higher in HFIR-PTP than that in Fusion and FFTF.

Response of V–Cr alloys to neutron irradiation has been investigated with fast reactors (FFTF [36,38,39] and JOYO [40]). Similar to V–Fe, addition of chromium

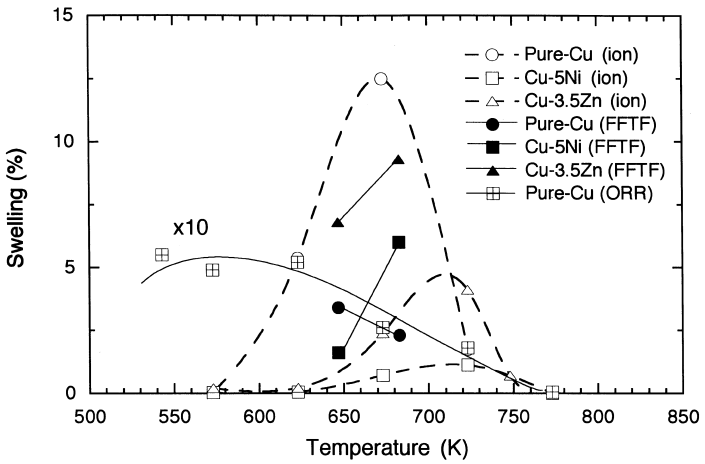

Fig. 2. The temperature dependence of swelling in Cu, Cu–5Ni and Cu–3.5Zn. Irradiations with 4 MeV heavy ions to 15 dpa [31], in FFTF Below Core Canister to 4.9–6.3 dpa [30] and in ORR to ~1.2 dpa [32] are compared. The damage rate of the ion irradiation was (6–15) $\times 10^{-4}$ dpa/s, and that of the neutron irradiations was typically ~10^{-7} dpa/s.

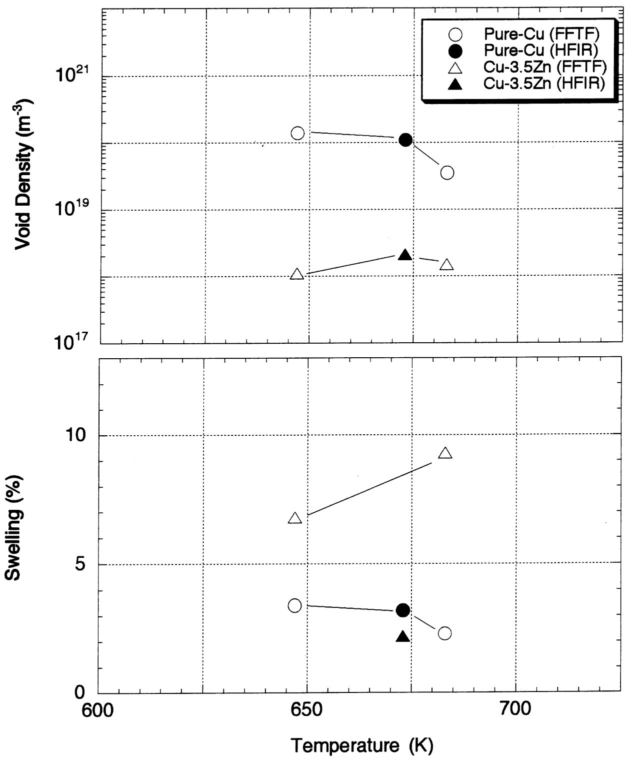

Fig. 3. The temperature dependence of void density and swelling in Cu and Cu–3.5Zn irradiated in FFTF Below Core Canister to 4.9–6.3 dpa [30] and HFIR-PTP to 10.4 dpa [33].

prior to irradiation significantly enhances void swelling in binary vanadium alloys, with an apparent peak effect occurring at concentrations below 10%.

A set of HFIR-PTP irradiations of V and V–1,5,10Cr was recently carried out in JUPITER program at 573, 673, 773 and 873 K to 7–10 dpa [41]. 5–8% of vanadium was transmuted into chromium in this experiment. Fig. 5 summarizes the void swelling observed in V–Cr alloys as a function of chromium content derived from neutron irradiations in FFTF [38], JOYO [40] and HFIR [41]. The data from HFIR are plotted vs. chromium content before and after irradiation. The figure also shows the predicted possible behavior of swelling at 10 dpa based on the fast reactor (FFTF and JOYO) data. It should be noted that the HFIR data agrees well with the prediction when the chromium content after irradiation is used as a parameter. This can be explained as follows. The swelling of V and V–10Cr alloys irradiated in FFTF to high exposure levels showed that the incubation of swelling is around 10 dpa and is roughly independent of alloy composition [42]. Thus, in the case of the HFIR irradiation to 10 dpa, any difference in swelling behavior would only appear near the end of the irradiation, when the composition was already close to that after irradiation.

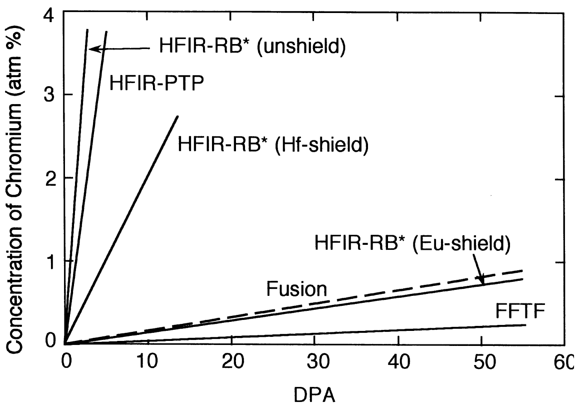

Fig. 4. Chromium production calculated for vanadium in FFTF (midplane), HFIR (PTP) and fusion first wall (STARFIRE) [1]. Also included are in HFIR-RB* with Hf shield [1] and with Eu shield [37].

2.4. Remarks on the status of solid transmutation studies

The studies discussed above demonstrated that alloying with expected transmutant elements before irradiation is sometimes a useful technique to elucidate the transmutation effects. However, more detailed studies

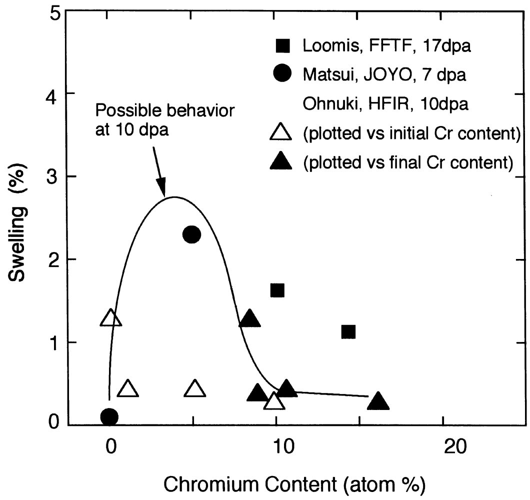

Fig. 5. Collection of swelling data for V–Cr alloys at ~873 K and 7–17 dpa from FFTF [38], JOYO [40] and HFIR-PTP [41]. The data from HFIR-PTP are plotted vs. Cr content both before and after irradiation. Prediction of the swelling behavior at 10 dpa is also shown.

are necessary. Fig. 5 shows a good example that lack of data at lower chromium levels by fast reactors results in an increased ambiguity of the prediction.

Tailoring of the transmutation rate to realize the fusion-relevant condition is particularly important. The generation rates of nickel/zinc in copper and chromium in vanadium during irradiation can be changed in the HFIR-RB* position using a thermal neutron shield. However, the generation rate is still rather high when a Hf shield is used [1]. In the JUPITER program, irradiation in HFIR-RB* with an Eu shield is now in progress. With the Eu shield, the transmutation rate is calculated to be reduced further and to be close to the fusion-relevant condition as shown in Fig. 4 [37]. A comparative HFIR-RB* irradiation without a shield is also planned.

3. Effect of temperature history

3.1. Role of varying temperature irradiation

Several results have been obtained from the periodic temperature change experiments in JMTR [8–10]. Varying temperature irradiation experiments were also carried out with ions and electrons [12–14]. The impact of varying temperatures on the irradiated behavior of fusion materials is discussed in the following sections.

3.1.1. Evaluation of the temperature history effect in materials test reactors

There are a number of experimental results suggesting that the microstructure after high fluence fission reactor neutron irradiation can be significantly influenced by low-temperature, low-fluence irradiations associated with the start-up and the shut-down procedures of typical materials test reactors. In the Japan–USA collaboration using FFTF/MOTA irradiation, formation of additional defect clusters during shut-down procedures of the reactor was observed in several materials [43–45].

Fig. 6 compares microstructures of Fe–16Cr–17Ni–0.024P at 873 K after irradiation in FFTF/MOTA during cycles 10, 11 and 12. In all cases, small defect clusters were observed in the matrix. However, the natures of these clusters were different in each irradiation cycle. After cycle 10, the clusters were a mixture of interstitial loops and stacking fault tetrahedra (SFT), as seen by their circular and triangular shapes, respectively. After cycle 11 and cycle 12, however, the clusters were dominated by SFT and loops, respectively. Because such clusters are typical components of microstructures at much lower temperatures than 873 K, it is expected that they were produced during the shutdown procedure of the last irradiation cycle.

Fig. 7 compares the shutdown histories of the reactor and the specimen canisters designed to operate at 873 K [46]. The cooling was done most swiftly in cycle 11 and most slowly in cycle 12. The difference in the cooling speed is consistent with the types of clusters observed. In the case of swift and slow cooling, vacancy clusters (SFT) and interstitial clusters (loops) were formed in the matrix, respectively. The loops observed were interpreted to be formed at 573–673 K and <10% power during the shutdown procedure, based on the loop density data derived from irradiations with fission neutrons, fusion neutrons and electrons [45,47]. The formation of the stacking fault tetrahedra was explained using rate theory, assuming a quick temperature decrease during irradiation [45]. These studies demonstrate that misleading results regarding the fine scale microstructure (loops and SFTs) can be obtained for materials irradiated at some "nominally constant" temperature if low-temperature transients during the reactor shutdown are not taken into account.

A potentially even more important effect of low-temperature transients exists when the transient occurs at the beginning or intermediate of the irradiation, since this could alter the microstructural evolution pathway. Nucleation of voids, loops and other radiation-induced defects typically occurs at doses $\ll$0.1 dpa [35]. Therefore, enhanced nucleation of voids and/or loops during a low-temperature transient at the beginning of the irradiation may significantly alter the sink strength of the evolving microstructure. This could produce either improved or degraded radiation resistance of the material compared to a steady-temperature irradiation, depending on the detailed experimental conditions.

A comparison of radiation-induced microstructures in pure metals and Fe–Cr–Ni alloys was made in JMTR

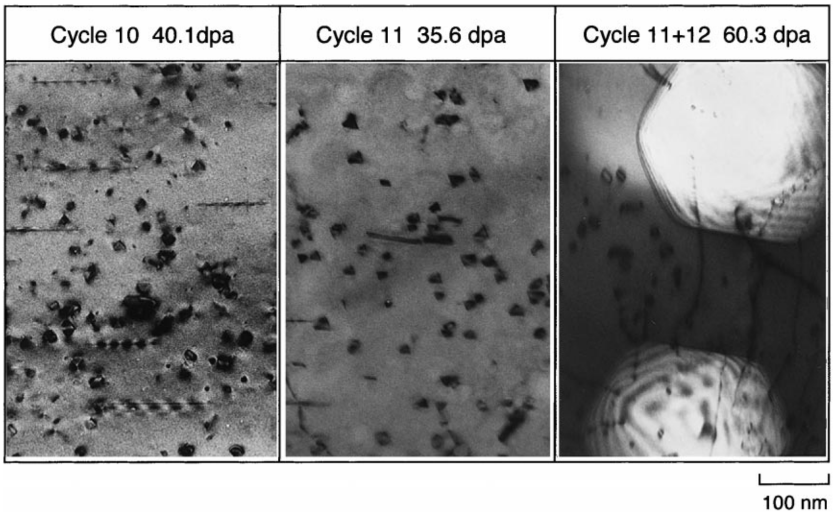

Fig. 6. Microstructures of Fe–16Cr–17Ni–0.024P at 873 K after irradiation in FFTF/MOTA in cycle 10, cycle 11 and cycles 11 and 12. Clusters observed in the matrix are a mixture of interstitial type loops and stacking fault tetrahedra after cycle 10, predominantly SFT after cycle 11 and predominantly interstitial loops after cycles 11 and 12, respectively.

with conventional (gamma-heating) and improved (electrical heater) temperature control methods. In the latter case, the specimen temperature was maintained independently of the reactor power. The results showed that enhanced nucleation of defect clusters occurred during the transient period in the conventional control case [5,7]. A similar enhancement of void nucleation was observed in vanadium alloys [10].

Reexamination of the previously obtained neutron irradiation data may also be needed, taking the temperature transient effects into account. It has also been shown that many earlier experiments were subject to unrecognized transients of temperature during irradiation [44]. Varying temperature irradiation experiments would be useful both for verifying past irradiation data and for seeking the mechanisms involved in the temperature history effects. It is anticipated that the impact of the varying temperature effect will be dependent on the precise values of the temperature excursion relative to key materials parameters such as recovery

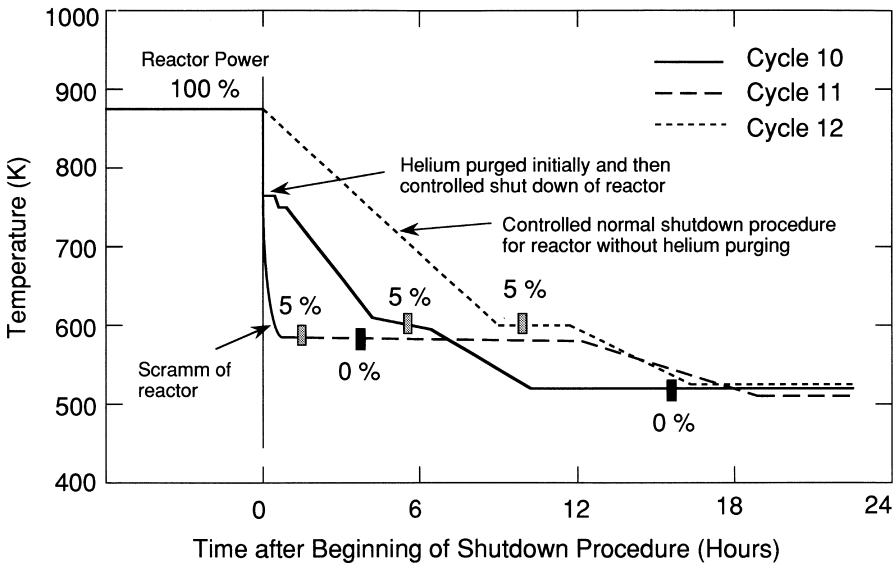

Fig. 7. Comparison of temperature histories of the specimen canisters designed at 873 K during the shutdown procedures of cycle 10, 11 and 12 of FFTF/MOTA [46]. The percentages included on the figure show the fraction of the original reactor power level at that point.

stages III and V (corresponding to vacancy migration and dissolution of small vacancy clusters, respectively).

3.1.2. Simulation of the temperature history in DEMO structural materials

DEMO reactors are designed to operate mostly as quasi-steady state machines, where the burn time will be weeks to months long. Periodic burning and intermittent cooling would result in repeated low temperature (transient) irradiation followed by high temperature irradiation of structural components. An irradiation experiment simulating such temperature history would allow a more realistic prediction of a certain material's behavior under DEMO conditions.

Temperature transients are also expected in ITER. Calculations were recently performed on expected temperature transients for the ITER shielding blanket [48]. Fig. 8 shows examples of the calculations. The figure shows that, after the initiation of the burn, the temperature reaches steady state in a few seconds to hundreds of seconds, depending on the position in the blanket. Since the objective of the temperature history calculation in the ITER calculation was to analyze stress changes during operation, the temperature history effect on the radiation response of materials was not considered. It should be noted that, in contrast to the transient during the start-up of fission reactors where the reactor power slowly increases, the fusion reactor will reach full power quickly, typically after a few seconds, although the temperature of the structural components continue to increase. This means that the neutron exposure level of structural components during the temperature transient for fusion reactors would be much higher than that for fission reactors.

3.1.3. Fundamental aspects

Change of temperature during irradiation is generally a useful technique when employed in charged particle irradiation studies as a probe into mechanistic understanding of the temperature-dependent microstructural processes operating during irradiation. Electron irradiation with variable temperature has been applied to the determination of the type of dislocation loops and detection of submicroscopic defect clusters [49]. The change of temperature during in-situ observation of ion irradiation-induced microstructures suggested mechanisms of vacancy dominance induced by cascades [50].

3.2. Insight into factors controlling the temperature transient effect

Earlier, there were studies of the impact of unavoidable temperature changes occurring during irradiation of stainless steels in fast reactors [51,52]. Generally, voids, once formed at lower temperatures, persisted during the subsequent irradiation at higher temperatures. Additional void nucleation could occur following decreases in temperature. Most of these phenomena could be explained based on the available understanding on defect mobility and stability. However, the recent temperature variant irradiation experiments have observed several new, unpredicted, and sometimes puzzling effects on microstructural evolution. The factors controlling the microstructural processes may be summarized as follows.

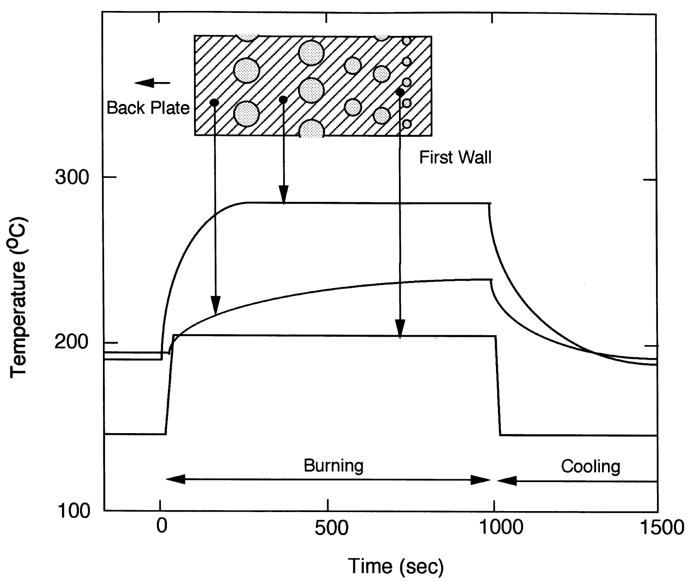

Fig. 8. An example of the temperature history analysis at the ITER shielding blanket during a repeated operation [48].

3.2.1. Submicroscopic vacancy clusters

One of the most interesting results obtained in the periodic temperature change experiments carried out in JMTR was that the dislocation evolution can be strongly suppressed by the periodic temperature variation. Fig. 9 shows the microstructure of Fe–16Cr–17Ni and Fe–16Cr–17Ni–0.25Ti irradiated in JMTR both at a constant temperature of 673 K and at a periodically-variable temperature of 473 and 673 K [8]. In this figure, the ion irradiation data on Fe–16Cr–17Ni with a stepwise temperature change are also shown [13]. In the JMTR experiment, dislocations almost disappeared by the temperature change. High density of cavities were formed in the Ti modified alloy. Since the change was so drastic, one would immediately suspect that a failure of the temperature control system caused annealing of defects during irradiation.

Such suspicion was dismissed by the ion irradiation experiment. Loops which were initially formed by the pre-irradiation at 473 K disappeared during the succeeding irradiation at 773 K as also shown in Fig. 9 [13]. A rate theory analysis of the varying temperature irradiation conditions indicated that the accumulation of vacancy clusters during the lower temperature irradiation suppressed the dislocation evolution during the subsequent higher temperature irradiation [8,13]. A similar suppression of dislocation evolution by the cyclic temperature change was reported for Ni and Cu [9].

3.2.2. Redistribution of solute and impurities

In the case where nucleation or resolution of precipitates takes place at low doses, a short low-temperature transient could change the precipitate evolution during irradiation. For example, a low temperature pre-irradiation caused finely dispersed phosphide formation in the matrix of Fe–Cr–17Ni–P alloys during subsequent irradiation at high temperature [53]. In vanadium, precipitates formed at high temperature were dissolved by very low dose additional irradiation at lower temperature [54].

3.2.3. Burgers vector of dislocations

A recent electron-irradiation study in Fe–10Cr ferritic alloys showed that the initial negative temperature excursion during irradiation may alter the Burgers vector fraction of loops formed during subsequent irradiation [14]. The study showed that the pre-irradiation at 573 K for a short time (e.g. 15 s, 0.002 dpa) changes the Burgers vector of loops formed during irradiation at 673 K from a mixture of $a\langle 1\ 0\ 0\rangle$ and $a/3\langle 1\ 1\ 1\rangle$ to a predominance of $a/3\langle 1\ 1\ 1\rangle$.

The Burgers vector of dislocations has been thought to be related to the swelling resistance in BCC metals and alloys. The $a/3\langle 1\ 1\ 1\rangle$ type dislocations were thought to suppress swelling because of its weak bias toward interstitials [55]. Thus it is possible that the radiation response of materials may be changed by the temperature transient through the change in the Burgers vector of dislocations.

3.3. Varying temperature irradiation experiment in HFIR

Based on the JMTR irradiation studies and the supporting charged particle irradiation studies, the mechanistic understanding of the temperature history effect at low dose has been largely enhanced. As the next step, it is important to estimate the temperature history effect at high neutron irradiation exposure levels. Some

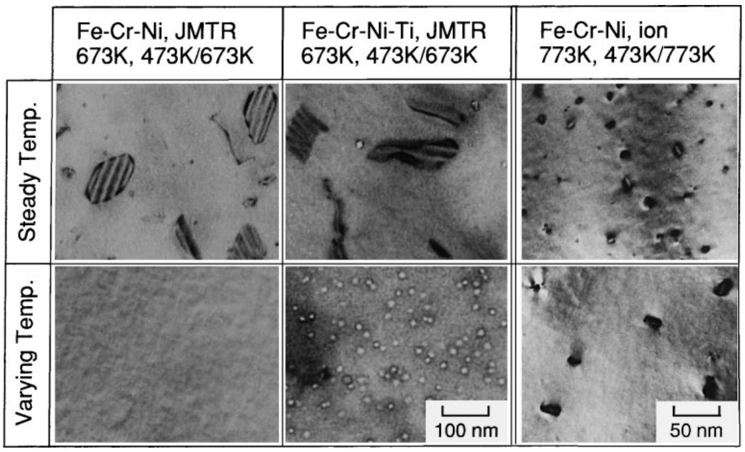

Fig. 9. Microstructure of Fe–16Cr–17Ni and Fe–16Cr–17Ni–0.25Ti irradiated in JMTR in constant and periodically-varying temperature conditions [8]. In the case of the variable condition, the temperature was periodically changed six times during the irradiation. The total dose was about 0.13 dpa. Also shown are microstructures of Fe–16Cr–17Ni irradiated with 4-MeV Ni ions at 773 K to 0.18 dpa and that irradiated in the same condition but pre-irradiated at 473 K to 0.2 dpa [13].

predictions could be made based on the mechanisms derived from the low dose JMTR irradiations, charged particle irradiations and the model-based calculations. However, such predictions need to be verified by well-controlled experiments.

For this purpose, a varying temperature irradiation experiment has been designed and will soon begin in JUPITER project [56]. This experiment is an unprecedented controlled irradiation experiment, in which symmetrical comparison of isothermal (623 and 773 K) and temperature-variant (623 K/473 K and 773 K/573 K) neutron irradiation effects will be made in HFIR to the dose level of ~10 dpa. In the temperature-variant zones, 10% of the exposure at the start of each reactor cycle will occur at reduced temperature (473 and 573 K). The temperature control will be carried out independently of the reactor power using electrical heating to supplement the heating caused by gamma rays.

4. Conclusions

Solid transmutant generation effects and temperature history effects have been recognized to be important issues for the study of neutron radiation damage under fusion conditions. These issues complicate the methodology for applying fission neutron irradiation data to fusion materials development. Advanced use of fission reactors is therefore being performed to research into these subjects. Charged particle irradiations are also useful for supporting the neutron irradiation studies.

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