Microstructure and Property Evolution in Advanced Cladding and Duct Materials Under Long-Term Irradiation at Elevated Temperature: Critical Experiments

Reactor Concepts RD&D

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Project Objective: To extend the range of operation of nuclear fuel cladding and structural materials in advanced nuclear energy and transmutation systems to that required for the fast reactor, the irradiation-induced evolution of the microstructure, microchemistry, and the associated mechanical properties at relevant temperatures and doses must be understood. This project performs a set of critical experiments to improve the understanding of these radiation effects.

Project Goals: 1) Identify the formation mechanism and evolution for dislocation loops with Burgers vector of $a\langle 100 \rangle$ and determine whether the defect microstructure saturates at high dose and identify whether a threshold irradiation temperature or dose exists for the nucleation of growing voids that mark the beginning of irradiation-induced swelling, and begin to probe the limits of thermal stability of the tempered martensitic structure under irradiation, 2) evaluate the stability of nanometer sized YTi- O based ODS particles at high fluence/temperature, 3) evaluate the extent to which precipitates form and/or dissolve as a function of irradiation temperature and dose, 4) how these changes are driven by radiation induced segregation, neighboring microchemical evolution, and initial microstructure, and 5) identify the mechanisms of irradiation creep in ferritic-martensitic steels.

1. Executive Summary

The major accomplishments of this project are as follows:

- Dislocation loops in ion irradiated F-M alloys are dominated by $\langle 100 \rangle$ type loops at high doses and at low doses, defect clusters and small loops (both $\langle 100 \rangle$ and $1/2\langle 111 \rangle$ type) exist.
- There is a clear dependence of dislocations on irradiation temperature with dislocation loops dominant at low temperature and dislocation segments and network dominant at high temperature. Dislocation loops saturate at ~ 100 dpa for the 9Cr model alloy. Dislocation loops appear to be larger and with a number lower density at 500 dpa for T91 and HCM12A.
- Ni/Si/Mn-rich, Cu-rich and Cr-rich precipitates are three commonly observed radiation-induced or enhanced precipitates in ion-irradiated F-M alloys. The type of precipitates is heavily dependent on alloy composition.
- Irradiation temperature has a significant effect on precipitates. Precipitates are larger and at lower number density in high temperature irradiations.
- There is an incubation dose for precipitate nucleation in ion irradiated F-M alloys. Precipitate coarsening occurred at high doses. A high volume fraction of radiation-induced carbides observed in F-M alloys occurs high dose and high temperature.
- The stability of Yt-Y-O nanoclusters 14YWT ferritic ODS steel under 5MeV Ni^{2+} irradiation to 100dpa and at -75°C , 100°C , 300°C , 450°C , 600°C , and 700°C was examined. Heavy ion Irradiation under 300°C leads to a substantial decrease in size and number density of nanoclusters in 14YWT. There are minimal changes in size and number density of nanoclusters in 14YWT irradiated at 450°C to 600°C compared to the unirradiated case.
- Irradiation at -50°C and 200°C leads to an apparent decrease in the average size and the number density of nanoclusters in the 9Cr-ODS. Change in size and density is much less for samples irradiated at 600°C . A slight coarsening of nanoclusters in the 9Cr-ODS was observed as a result of proton irradiation at 400°C to 3.7 dpa.
- The behavior of Cr (and Fe) RIS in F-M alloys was found to be largely consistent with the IK mechanism, supporting IK as the dominant mechanism of Cr RIS in F-M alloys.
- Dislocation loops nucleate anisotropically within a single grain depending on the angle between its habit plane and the tensile axis.
- Dislocation loop sizes correlate with stress and dose.
- SIPN contribution to strain accounts for 6-37% of the total strain measured. The longer the irradiation, the less SIPN contributes to the total strain.
- Stress dependence of irradiation creep is linear at stress below 160MPa and greater than linear at stress above 160MPa, suggesting a switch from dislocation climb to dislocation glide at higher stress.

2. Major Accomplishments

The following sections describe the major accomplishments or outcomes in this program. They are presented in a form that focuses on the key results and conclusions. Details of the experiments and full set of results are available in the quarterly reports and are referenced herein rather than repeated.

2.1 Dislocation Microstructure

Dislocation structures in irradiated Ferritic/Martensitic alloys were fully characterized in terms of dose and temperature. The studied alloys included T91, NF616, HCM12A, 9Cr and 12 Cr model alloys. The heavy ion irradiation experiments were conducted using the 1.7MV Tandatron accelerator in the Michigan Ion Beam Laboratory (MIBL) at the University of Michigan. Samples were irradiated over a range of 7-500 dpa and at temperatures of 400-500°C using 5 MeV Fe^{++} . The temperature variation was monitored using a thermal imager and was kept within $\pm 10^\circ\text{C}$ during the course of irradiation. The dose rate was $\sim 10^{-3}$ dpa/s. The irradiation depth for 5 MeV Fe^{++} is $\sim 1.2 \mu\text{m}$ according to SRIM calculation (full cascade mode). Low dose proton irradiations (1-3 dpa) were conducted at the University of Wisconsin's ion beam lab with 2.0 MeV protons, and the dose rate was approximate 1.3×10^{-5} dpa/s. The radiation damage peak is about 18 μm below the surface, and the TEM specimens were prepared at the depth of 15 μm . The characterization of dislocation loops was performed using two beam bright field/dark field TEM images or STEM bright field images with the incident beam along the direction of [001] zone axis.

Dislocation loops in ion irradiated F-M alloys are dominated by $\langle 100 \rangle$ type loops at high doses and at low doses, defect clusters and small loops (both $\langle 100 \rangle$ and $1/2\langle 111 \rangle$ type) exist.

For the irradiated T91 and HCM12A at 400°C or above to doses in the range 7-500 dpa, both the two beam condition TEM, and [001] zone axis STEM images show that the $\langle 100 \rangle$ type dislocations loops are dominating while the $1/2\langle 111 \rangle$ type dislocation loops are negligible, Figure 1. For the irradiated 9 and 12 Cr model alloys, both $\langle 100 \rangle$ and the $1/2\langle 111 \rangle$ type dislocation loops are identified as the $1/2\langle 111 \rangle$ type dislocation loops appear as ellipsoidal loops in a TEM image using g_{011} near the zone axis of [100]. However, the exact ratio between the $1/2\langle 111 \rangle$ and $\langle 100 \rangle$ type dislocation loops was not determined due to the fact that some dislocation loops are too small to be identified for their types. For NF616 irradiated at 500 and 700°C, it was assumed that all the dislocation loops should be $\langle 100 \rangle$ type since at such high temperature, the $1/2\langle 111 \rangle$ type dislocation loops can easily migrate and annihilate at line dislocations, grain boundaries or other defect sinks. For the NF616 irradiated at 500°C, the irradiated microstructures consisted of small clusters ($< 5 \text{ nm}$) and identifiable dislocation loops. However, for NF616 irradiated at 700°C, the dislocations were mainly segments or networks.

There is a clear dependence of dislocations on irradiation temperature with dislocation loops dominant at low temperature and dislocation segments and network dominant at high temperature.

Figure 2 shows dislocation evolution in T91 with increasing irradiation temperature. At 400°C, the dominant feature in the irradiated microstructure is small dislocation loops (Figure 2a). The dislocation microstructure of T91 irradiated to 30 dpa at 440°C contains a combination of dislocation loops and line dislocations (Figure 2b). Dislocation loops are absent in T91 irradiated to 100 dpa at 500°C and line dislocations become dominating feature (Figure 2c). The effect of temperature on dislocation microstructure of T91 is very distinct with three different regimes: dislocation loops dominant (400°C and probably below), a combination of dislocation loops and dislocation segments (440°C), and dislocation segments dominant (500°C and probably above). In HCM12A, dislocation loops are also dominant at 400°C (Figure 3a) and dislocation segments appear at 500°C (Figure 3b).

The transition of dislocation structures from loops to lines in irradiated NF616 is also very distinct as shown in Figure 4. The sample irradiated at 500°C contains a mixture of dislocation segment and dislocation loops, while at 700°C the dislocation network becomes a dominating feature.

For irradiated 9 and 12 Cr model alloys, since the irradiation was only conducted at 400°C, no temperature dependence can be deduced. However, it is confirmed again that the dislocation loops are the dominant dislocation structure in the materials irradiated at such temperature, as shown in Figure 5.

Dislocation loops saturate at ~100 dpa for the 9Cr model alloy. Dislocation loops appear larger at lower density at 500 dpa for T91 and HCM12A.

Dose dependence of dislocation loop is summarized in Figure 6. For T91 (Figure 6a), the number density of dislocation loops doubled between 30 dpa ($0.6 \times 10^{22} \text{ m}^{-3}$) and 100 dpa ($1.3 \times 10^{22} \text{ m}^{-3}$), but the average loop size was nearly the same at around 15 nm. The average loop size increased to 27 nm and the number density decreased to $0.7 \times 10^{22} \text{ m}^{-3}$ at 500 dpa. Although both size and number density changed significantly, dislocation loops are still the dominant microstructure at 500 dpa. Similar dose dependence holds for HCM12A irradiated at 400°C (Figure 6b). Density increased and the size was nearly unchanged at 100 dpa compared to 30 dpa. The size increased from 13 nm at 100 dpa to 22 nm at 500 dpa, while the number density decreased slightly from $1.5 \times 10^{22} \text{ m}^{-3}$ to $1.4 \times 10^{22} \text{ m}^{-3}$. In proton irradiated NF616 at 500°C, both the average dislocation loop size and number density start to saturate at 2 dpa (Figure 6c). For the Fe^{++} irradiated 9Cr, the similar saturation behavior of dislocation loop structure was observed. The changes of loop density and average dislocation loop size are minimal after 30 dpa (Figure 6d).

Table 1 Summary of size and density of dislocation loops in irradiated alloys.

Alloy	Temp. (°C)	Dose (dpa)	# of dislocation loops	Size (nm)	Density (10 ²² m ⁻³)	Note
T91	400	7	Defect clusters			
		30	222	15.6±0.4	0.6	100> type dislocation loops dominate
		100	399	14.7±0.2	1.3	
		500	294	27.2±0.5	0.7	
	500	100	Dislocation segments			
HCM12A	400	30	81	13.5±0.3	0.5	100> type dislocation loops dominate
		100	113	12.7±0.3	1.5	
		500	205	21.6±0.6	1.4	
	500	100	Dislocation segments			
		500	Dislocation segments			
9Cr Model	400	7	201	11.6±2.1	1.0	<100> and ½<111> dislocation loops dominate
		30	178	12.9±2.3	2.5	
		500	165	14.1±3.5	3.6	
12Cr Model	400	500	177	11.2±2.2	3.5	
NF616	500	1	-	8.07	0.08	Clusters and dislocation loops
		2	-	9.61	0.309	
		3	-	9.72	0.32	
	700	1-3	Dislocation segments or networks

Figure 1. a) $\langle 100 \rangle$ type of dislocation loops in T91 following irradiation to 100 dpa at 400°C as revealed using bright field TEM image with $b=[001]$ and $g=020$ (b) and STEM bright field image under multi-beam condition along $b=[001]$.

Figure 2. Dislocation loop evolution in T91 following Fe^{++} irradiation at various doses and temperatures: (a) 400°C: 100 dpa (b) 440°C: 30 dpa, and (c) 500°C: 100 dpa.

Figure 3. Dislocation microstructure in HCM12A following Fe^{++} irradiation to 500 dpa at (a) 400°C, and (b) 500°C.

Figure 4. Irradiated microstructures of NF616 to 3dpa: (a) 500°C and (b) 700°C.

Figure 5. Dislocation microstructures of 9 and 12 Cr model alloys irradiated at 400°C to 500 dpa.

Figure 6. Dislocation evolution vs. dose in (a) T91 (Fe⁺⁺ irradiated at 400°C), (b) HCM12A (Fe⁺⁺ irradiated at 400°C), (c) NF616 (proton irradiated at 500°C) and (d) 9 Cr model alloy (Fe⁺⁺ irradiated at 400°C).

2.2 Radiation-Induced Precipitates

Irradiation-induced precipitates were examined in ion irradiated F-M alloys including T91, HCM12A and HT9 using atom probe tomography (APT). 3D reconstruction and data analysis was conducted using IVAS 3.4 software from the CAMECA.

Ni/Si/Mn-rich, Cu-rich and Cr-rich precipitates are three commonly observed radiation-induced or radiation-enhanced precipitates in ion-irradiated F-M alloys. The type of precipitates is heavily dependent on alloy composition.

As Ni, Si and Mn are the common elements in F-M alloys, Ni/Si/Mn-rich clusters/precipitates are observed in all three alloys (T91, HCM12A and HT9). The observation is consistent for both ion irradiations (both proton and self-ion irradiations) and neutron irradiations. Therefore, Ni/Si-rich precipitates are probably the most common radiation-induced precipitates in F-M alloys containing Ni and Si. The Ni/Si/Mn-rich clusters/precipitates can be G-phase ($M_6Ni_{16}Si_7$) particles or their precursors depending on the irradiation conditions. Cu-rich precipitates only appear in HCM12A and T91 under both proton and Fe^{++} irradiations. No apparent Cu-rich precipitates are observed in HT9 as its bulk Cu content is only ~0.04 wt%. The volume fraction of Cu-rich precipitates are apparently dependent on bulk Cu content and it is much higher in HCM12A (1.02 wt% bulk Cu content) than in T91 (0.17 wt% bulk Cu content) under the same irradiation condition. Cu-rich precipitates are radiation enhanced and follow a similar nucleation, growth and coarsening (NGC) pattern as those observed in RPV steels. Cr-rich precipitates are slightly complex in F-M alloys. They are α' -phase or its precursors in proton irradiated samples. In Fe^{++} irradiated F-M alloys, radiation-induced Cr-rich precipitates normally contain a few percent of carbon and in some cases they are η carbides (M_6C). More details on the three types of precipitates from the research in this project can be found in publications [1] and [2].

Figure 7 shows examples of the three types of precipitates formed in F-M alloys following Fe^{++} irradiation to 500 dpa at 400°C. The proxigrams on the right show the composition of the three precipitates as indicated by arrows in T91. Note that the composition of the Ni/Si/Mn-rich precipitates is very close to the G-phase. Cu-rich precipitates tend to form near Ni/Si/Mn-rich precipitates. This phenomenon has been confirmed in both ion and neutron irradiated HCM12A.

There is an incubation dose for precipitate nucleation in ion irradiated F-M alloys. Precipitate coarsening occurred at high doses.

An incubation dose exists for Ni/Si/Mn-rich precipitates for both proton and heavy ion irradiations. In proton irradiation the incubation dose appears to be greater than 1 dpa while in Fe^{++} irradiation, the incubation dose is >7 dpa. The difference in incubation dose is probably due to dose rate, which is about two orders of magnitude higher for heavy ion irradiations compared to proton irradiations. As shown in Figure 8 for both T91 and HCM12A following Fe^{++} irradiation at 400°C, no Ni/Si/Mn-rich clusters form at 7 dpa. Ni/Si/Mn-rich precipitates appear at 30 dpa and there is virtually no change up to 100 dpa. Slight coarsening occurs at very high dose of 500 dpa. Quantitative analysis is shown in Figure 9. It can be seen that coarsening of the

Ni/Si/Mn-rich precipitates occurs at 500 dpa for both HCM12A and T91. Incubation dose for Cu-rich precipitates apparently is dependent on bulk composition. They appear at 7 dpa for HCM12A with high Cu content and 30 dpa for T91 with low Cu content. Also the coarsening process for HCM12A occurs at a low dose of <30 dpa (Figure 9) while the coarsening process for T91 is not obvious even at 500 dpa. The density and Cr-rich precipitates is generally low and a clear trend of evolution with dose was not seen. The nucleation sites of Cr-rich precipitates are independent of the other two types of precipitates.

Irradiation temperature has an appreciable effect on precipitates. Precipitates are larger and at lower density at high irradiation temperature.

The effect of irradiation temperature on precipitate size and density is very predictable with larger precipitates at lower density at higher temperature. An example of the temperature effect on the Cu-rich precipitates following Fe^{++} irradiation to 100 dpa in HCM12A and T91 is shown in Figure 10. Precipitates are evidently larger and at lower density at 500°C compared to those at 400°C. A similar temperature effect on Ni/Si/Mn-rich precipitates was also observed. The size and density of Ni/Si/Mn-rich and Cu-rich precipitates in HCM12A irradiated at 500°C are compared to those at 400°C in Figure 9. The substantial difference in size and density with temperature indicates the much lower nucleation rate at 500°C compared to that at 400°C.

A high volume fraction of radiation-induced carbides forms in F-M alloys after irradiation to high dose and high temperature.

Irradiation at high temperature to high dose (500°C: 500dpa) results in the formation of Cr_6C as shown in **Figure 11** from the APT atom maps, and from TEM/EDS and diffraction patterns in HCM12A. Formation of Cr_6C is probably due to the dissolution of pre-existing Cr_{23}C_6 as suggested by Maziasz [3]. At this extreme condition, partial dissolution of Cu-rich precipitates may occur leading to the smaller volume fraction of Cu-rich precipitates at 500 dpa than that at 100 dpa [2]. Further coarsening of Ni/Si/Mn-rich precipitates at 500 dpa leads to a very low number density that may be missed by the limited examined area. Figure 11 shows large Ni/Si/Mn-rich precipitates attached to a carbide in HT9 irradiated at 500°C:500 dpa. Significant segregation of Si to the carbide-matrix interface is also observed.

Figure 7. Formation of Ni/Si/Mn-rich, Cu-rich and Cr-rich precipitates in T91 and HCM12A following Fe^{++} irradiation to 500 dpa at 400°C. Graphs on the right are proxigrams of the three precipitates in T91 indicated by arrows in the left.

Figure 8. Evolution of Ni/Si/Mn-rich and Cu-rich precipitates with dose in HCM12A and T91 following Fe^{++} irradiation at 400°C. The Cr-rich precipitates are shown using Cr isosurface at 25 at% in HCM12A.

Figure 9. Evolution of size (average Guinier radius) and number density of Ni/Si/Mn-rich and Cu-rich precipitates with dose in HCM12A and T91.

Figure 10. Temperature effect on Cu-rich precipitates in HCM12A and T91 following Fe^{++} irradiation to 100 dpa.

Figure 11. Formation of M_6C carbides in HCM12A and large Ni/Si/Mn-rich precipitates (G-phase) in HT9 following Fe^{++} irradiation to 500 dpa at 500°C.

2.3 Nanoclusters in ODS alloys

Heavy ion irradiation at 300°C leads to a substantial decrease in size and number density of nanoclusters in 14YWT. There are minimal changes in size and number density of nanoclusters in 14YWT irradiated at 450°C to 600°C compared to the unirradiated case.

The 14YWT oxide dispersion strengthened (ODS) ferritic steel (nominal composition: Fe-14at%Cr- 3at%W-0.4at%Ti-0.25at%Y₂ O₃) was provided by Oak Ridge National Laboratory (ORNL). Ion irradiations were conducted using Pelletron tandem accelerator at the Environmental and Molecular Science Laboratory (EMSL) at Pacific Northwest National Laboratory (PNNL) using 5 MeV Ni²⁺ ions at -75°C, 100°C, 300°C, 450°C, 600°C, and 700°C to damage levels of 100 dpa in the investigating area. 3-D atoms distribution maps of elements chromium, titanium, yttrium, oxygen in the unirradiated 14YWT ODS steel are shown in Figure 12. Nanoclusters are associated by the regions enriched with O, Ti and Y atoms.

The atom maps of elements of 14YWT collected by APT showed that the nanoclusters were largely eliminated during irradiation at -75°C and 100°C, and very few nanoclusters were observed to remain in the APT 3D map for the samples irradiated at 300°C to 100 dpa as shown in

Figure 12(b), (c) and (d). The nanoclusters that remaining were smaller than those in the unirradiated sample, indicating that Ti, Y and O from the nanoclusters had a tendency to dissolve under these irradiation conditions. In contrast, large number densities of nanoclusters were observed in samples irradiated to the same damage levels at 450°C, 600°C, and 700°C samples. For these samples, zones of Ti, Y and O enrichment were similar to the unirradiated samples, revealing a stable nanoclusters population, as shown in Figure 12(e), (f) and (g).

The nanocluster size distribution at different irradiation temperatures are shown in Figure 13. The average Guinier radius, $\langle R_g \rangle$, and number density, N_v , are also included. The peak of the nanocluster size distribution for the samples was about 0.8 nm. The average Guinier radii of the unirradiated sample and samples irradiated at 300°C, 450°C, 600°C, and 700°C samples were 1.1 ± 0.5 , 0.8 ± 0.2 , 1.2 ± 0.6 , 1.1 ± 0.5 nm and 1.1 ± 0.5 , respectively, and corresponding number densities were 12×10^{23} , 3.7×10^{23} , 9.1×10^{23} , 7.2×10^{23} and $6.0 \times 10^{23} \text{ m}^{-3}$. These values show that after irradiation at 300°C the average nanocluster size decreased by about 25% and the number density decreased by 70%. Less dramatic changes were observed for samples irradiated at 450°C, 600°C and 700°C samples.

Irradiation resulted in a variation in the nanocluster composition, as indicated in Table 2. The composition of the nanoclusters and matrix of 14YWT steel determined using the maximum separation method [4] in this study are also summarized in Table 2. Here the Ti/Y ratio approaches ≈ 4 in the nanoclusters of 450°C and 600°C irradiated samples that were similar to the unirradiated samples, but this ratio was 1.5 in the 300°C samples and 2.0 in the 700°C samples. That may indicates that the Ti/Y ratio was controlled by the size of nanoclusters and the irradiation temperature. The metal-to-oxygen ([Ti+Y]:O) ratio was approximately 1:1 in all samples.

Irradiation at -50°C and 200°C led to an apparent decrease in the average size and the number density of nanoclusters in the 9Cr-ODS. The change in size and density was much less for samples irradiated at 600°C.

The 9Cr-ODS ferritic steels were provided by KOBELCO research institute, INC, whose chemical composition is shown in Table 3. Ion irradiation were conducted by Pelletron® tandem accelerator at the Environmental and Molecular science laboratory at Pacific Northwest National Laboratory (PNNL) using 10 MeV Pt³⁺ ions at -50°C/100dpa, 200°C/100dpa , 600°C/100dpa.

Figure 14 shows the atom maps of elements collected by APT after Pt³⁺ irradiation at -50°C, 200°C and 600°C. Very few nanoclusters were observed to be present in the APT 3D maps for the samples irradiated at 200°C and 100dpa as shown in the Figure 14c. The nanoclusters were nearly eliminated in the -50°C:100dpa sample suggesting that the nanocluster is not stable during irradiation at very low temperature. In contrast, Figure 14d showed a large number densities of nanoclusters in samples irradiated at 600°C, even at a damage level of 200 dpa. The nanocluster size distributions at each condition are shown in Figure 15. The average Guinier radius, $\langle R_g \rangle$, and number density, N_v , were also included. The peak of the nanocluster size distribution for the samples was about 0.8nm in the -50°C and 200°C samples, and about 1.6nm and 1.2nm in the unirradiated and 600°C irradiated samples. The average Guinier radii of the unirradiated sample and those irradiated at -50 °C , 200°C up to 100dpa and 600°C samples with damage level up to 200dpa were 1.4 ± 0.5 nm, 0.8 ± 0.1 nm, 0.9 ± 0.5 nm, 1.3 ± 0.5 nm, respectively, and corresponding number densities were 9.8×10^{23} , 0.6×10^{23} , 6.1×10^{23} and $9.8 \times 10^{23} \text{m}^{-3}$. These values show that after irradiation at -50°C the average nanocluster size was reduced by about 45% and the number density decreased by 95%. In the 200°C samples, average nanocluster size was reduced by about 35% and the number density decreased by 50%. Less change in size and density was observed for samples irradiated at 600°C.

The composition of the nanoclusters and matrix of 9Cr-ODS steel in this study are summarized in Table 4. Here the Ti/Y ratio approaches ≈ 4.5 in the nanoclusters of 600°C irradiated samples that were similar to the unirradiated samples, but this ratio was 2 in the 200°C samples. The value of the metal-to-oxygen ([Ti+Y]:O) ratio was similar in all of the samples .

Stability of nanoclusters in ODS steels during irradiation is a complex phenomenon that can be affected by various intrinsic and extrinsic factors such as alloy composition and irradiation conditions (temperature, damage rate, etc) [5]. During irradiation, the nanoclusters could grow through diffusion of solute atoms from the matrix and surrounding nanoclusters or may shrink due to radiation-induced recoils or diffusion to other defect sinks (point defects, dislocation, and interface). In general, if the nanocluster is stable in the ODS steels during irradiation, the balance should be maintained ----the number of the outputting atoms is equal to the inputting atoms. Although heavy ion beam into matrix can change local chemistries and the associated local diffusion rates, radiation-enhanced diffusion by the increase of point defects has to be taken into account, which is in agreement with the existence of an effect of temperature.

A slight coarsening of nanoclusters in the 9Cr-ODS was observed as a result of proton irradiation at 400°C to 3.7 dpa.

The 9Cr-ODS ferritic steels were provided by the Japan Atomic Energy Agency, whose chemical composition is shown in Table 5. Proton irradiation was conducted with a 1.7 MV Tandatron accelerator at the MIBL (Michigan Ion Beam Laboratory) using 2 MeV protons at 400°C to a dose of 3.7 dpa.

Figure 16 shows atom maps for 9Cr-ODS before and after proton irradiation. After irradiation, the number density of the nanoclusters is up to $1.5 \times 10^{23} \text{ n/m}^3$ with the average Guinier radii about $2.2 \pm 1.0 \text{ nm}$, as shown in Table 6. The change of number density and size of nanoclusters is less than the uncertainty of the larger nanoclusters and is not likely a significant change. The value of (Ti+Y):O ratio does not change after irradiation. However, The Ti:Y ratio decreased from 3.0 to 1.5 and the concentration of Cr increased from 4% to 8% after irradiation, indicating that the segregation of Cr occurs in the nanoclusters during irradiation.

Table 2. Compositions (at. %) of the matrix and nanoclusters of 14YWT irradiated under various conditions as determined by atomic probe tomography (APT).

Elements	Unirradiated	300°C	450°C	600°C	700°C
Matrix					
Fe	82.8	86.76	83.61	83.64	83.40
Cr	15.2	11.78	14.39	15.06	15.10
W	0.4	0.67	0.46	0.52	0.58
O	0.4	0.16	0.38	0.20	0.39
Ti	0.19	0.07	0.18	0.11	0.13
Y	0.08	0.04	0.07	0.05	0.07
Nanoclusters					
Fe	55.09±8.29	57.98±15.15	51.45±8.27	56.85±7.14	56.34±7.74
Cr	20.21±1.65	12.29±3.1	21.94±1.27	20.77±1.71	20.42±1.68
W	0.30±0.06	0.12±0.08	0.27±0.04	0.47±0.08	0.52±0.05
O	10.08±4.11	10.65±6.21	11.58±4.24	9.38±3.74	9.89±3.89
Ti	8.34±3.98	8.21±5.71	8.68±3.8	7.75±3.6	7.65±3.63
Y	2.03±0.97	5.36±3.79	2.04±0.92	2.10±0.95	4.05±2.02
Ti:Y	≈4	≈1.5	≈4	≈4	≈2
[Ti+Y]:O	≈1	≈1	≈1	≈1	≈1

Table 3. Chemical composition of 9Cr-ODS Steel provided by KOBELCO research institute, Inc.

Chemical composition (at.%)													
C	Si	Mn	P	S	Ni	Cr	W	Ti	Y	O	N	Ar	Fe
0.15	0.03	0.02	<0.005	0.003	0.01	9.2	1.99	0.32	0.28	0.15	0.027	0.004	Bal.

Table 4. Compositions (at. %) of the matrix and nanoclusters of 9Cr-ODS steels irradiated under various conditions as determined by atomic probe tomography (APT).

Elements	Unirradiated	-50°C	200°C	600°C
Matrix				
Fe	87.89	88.22	88.06	88.50
Cr	10.00	9.74	10.10	9.79
W	0.25	0.37	0.27	0.30
O	1.20	0.73	0.89	1.02
Ti	0.36	0.52	0.31	0.24
Y	0.07	0.11	0.09	0.04
Nanoclusters				
Fe	30.74		29.74	30.19
Cr	6.01		3.71	6.59
W	0.10		0.23	0.12
O	24.56		22.60	25.55
Ti	27.64		21.64	27.18
Y	6.24		12.53	5.9
Ti:Y	≈4.3		≈2	≈4.5
[Ti+Y]:O)	≈1.3		≈1.5	≈1.2

Table 5. Chemical composition (wt.%) of 9Cr-ODS Steel provided by the Japan Atomic Energy Agency

Chemical composition (at.%)													
C	Si	Mn	P	S	Ni	Cr	W	Ti	Y	O	N	Ar	Fe
0.14	0.048	<0.05	<0.02	0.004	0.06	8.67	1.96	0.23	0.27	0.14	0.017	0.004	Bal.

Table 6. The size and composition (at.%) of the titanium-, oxygen- and yttrium-enriched nanoclusters with a size less than 5nm in diameter as estimated by APT from the maximum separation method with $d_{\max} = 0.66$ nm, $n_{\min} = 8$ atoms.

Unirradiated specimen					Irradiated specimen				
Matrix			Nanoclusters		Matrix			Nanoclusters	
Y	0.10±0.01		8.76±0.23		0.17±0.01			11.3±0.19	
Ti	0.19±0.01		26.31±0.34		0.26±0.01			16.32±0.18	
O	0.29±0.01		24.12±0.08		0.61±0.01			21.95±0.12	
Cr	8.86±0.02		4.34±0.03		9.64±0.03			8.26±0.30	
Fe	89.61±0.02		29.1±0.63		87.44±0.03			34.03±0.51	
W	0.51±0.07		0.45±0.09		0.54±0.007			0.21±0.04	
			(Ti+Y):O	Y: Ti			(Ti+Y):O	Y: Ti	
Ratio			1.4	0.33			1.4	0.69	
R			1.86±0.99nm				2.18±0.99nm		
N _v			1.66×10 ²³ n/m ³				1.47×10 ²³ n/m ³

Figure 12. APT 3D reconstruction of 14YWT samples irradiated with 5MeV Ni ions to 100 dpa at temperatures of: (a) unirradiated (b) -75°C (c) 100°C (d) 300°C (e) 450°C (f) 600°C, and (g) 700°C

Figure 13. Nanocluster size distribution in unirradiated samples and those irradiated at 300°C, 450°C, 600°C, and 700°C up to 100dpa with 5MeV Ni ions.

Figure 14. APT 3D reconstruction of 14YWT samples irradiated with 10 MeV Pt ions (a) unirradiated sample (b) up to 100 dpa at -50°C (c) 100 dpa at 200°C (d) 200 dpa at 600°C

Figure 15. Nanocluster size distribution in unirradiated sample and irradiated samples

Figure 16. APT elemental maps of 9CrODS steel (a) before irradiation and (b) after proton irradiation to 3.7 dpa at 400°C.

2.4 Radiation-Induced Segregation

Grain boundary Cr enrichment occurred in a bell-shaped temperature dependence between 300°C and 600°C. A “crossover” temperature exists between 600°C and 700°C at which Cr RIS changes from enrichment to depletion.

The temperature dependence of RIS was studied in alloy T91 irradiated with 2.0 MeV protons to 3 dpa at temperatures ranging from 300°C to 700°C. The change in grain boundary composition was averaged over all line scans collected from a given irradiation condition; these average RIS values are shown as a function of temperature in Figure 17. From this graph, it is clear that only small amounts of Cr RIS, never exceeding ~2 wt%, were observed. Cr enrichment and Fe depletion occurred in a bell-shaped temperature dependence between 300°C and 600°C, but at 700°C the behavior reversed to Cr depletion and Fe enrichment. Minor elements (Si, Ni, Cu) segregation was observed between 400°C and 500°C, enriching in a bell-shaped temperature dependence over this range. Chromium enrichment and Fe depletion were maximized at 450°C, while enrichment of the minor elements Si, Ni, and Cu were maximized at 400°C.

The bell-shaped temperature dependence can be attributed to point defect mobility. At low temperatures, defects are immobile and cannot diffuse to sinks, severely limiting the amount of segregation. At elevated temperatures, high point defect mobility induces back-diffusion, removing concentration gradients. Hence, the most segregation occurs at moderate temperatures. At the extreme temperatures of 300°C and $\geq 600^\circ\text{C}$, some Cr and Fe RIS persisted, while minor element RIS was totally suppressed. This observation suggests that the mechanism of RIS of the minor elements differs than that of Cr and Fe.

Figure 17. Average change in grain boundary concentration of Cr, Fe, Si, Cu, and Ni RIS as a function of temperature, in T91 irradiated to 3 dpa with 2.0 MeV protons.

One of the most notable behaviors of RIS in F-M alloys is the “crossover” from Cr enrichment to Cr depletion (and also Fe depletion to Fe enrichment) between 600°C and 700°C. This type of behavior has never been reported in any other alloy system. In this project, the crossover was also observed in alloy NF616, irradiated with 2.0 MeV protons to 3 dpa at 500°C and 700°C. Figure 18 shows representative Cr RIS profiles from NF616, demonstrating Cr enrichment at 500°C reverting to Cr depletion at 700°C.

Figure 18. Representative Cr RIS profiles in NF616 irradiated to 3 dpa.

The average full width at half maximum (FWHM) of the Cr RIS profiles and the average area under the Cr enrichment peaks are shown in Figure 19 for T91 at 3 dpa. Not surprisingly, the area under the Cr enrichment peaks exhibited a bell-shaped temperature dependence, much like the change in grain boundary Cr concentration does. The FWHM of the Cr peaks however, increased as a function of temperature due to back-diffusion, which caused broadening of the Cr concentration gradient at elevated temperatures.

Figure 19. Average FWHM of peak and area under Cr RIS peak as a function of temperature, in T91 irradiated to 3 dpa with 2.0 MeV protons.

For additional details about RIS Accomplishment #1, please view references [6-10].

The behavior of Cr (and Fe) RIS in F-M alloys was found to be largely consistent with the IK mechanism, supporting IK as the dominant mechanism of Cr RIS in F-M alloys.

This study used a one-dimensional inverse Kirkendall (IK) model to calculate Cr RIS in a bcc Fe-Cr binary alloy system. The model simultaneously solves a system of equations representing the concentrations of the major alloying elements and the point defects, as a function of space and time. These equations are solved across a plane foil; one surface of the plane foil is fixed to simulate the grain boundary and to act as an unbiased point defect sink. Symmetry is assumed across the grain boundary. A number of inputs are required, but the model is most sensitive to four of them: the vacancy and migration energies of both Cr and Fe. Appropriate values for the Fe and Cr vacancy and interstitial migration energies were taken from various *ab initio* calculations published in the open literature, since limited experimental studies of these migration barriers provided only qualitative results. Furthermore, since the measured composition profile is the convolution of the actual composition profile with the electron beam, the model results were convoluted so as to provide a fair comparison with experimental measurements.

The IK model was able to reproduce the experimentally-measured temperature dependence of Cr RIS, within the known uncertainty range of the model input parameters. The IK model calculated RIS magnitudes almost identical to those measured experimentally when the dislocation density of $7.5 \times 10^{14} \text{ m}^{-2}$ was used (Figure 20), which is consistent with the total sink strength of the material, as measured in reference [11]. Most importantly, the IK model predicted the crossover between Cr enrichment and Cr depletion between 600°C and 700°C .

Figure 20. Effect of sink density on temperature dependence of IK modeled Cr RIS for Fe-9Cr, compared with experimental measurements from T91, at 3 dpa, 10^{-5} dpa/sec.

The crossover can be explained by the diffusion coefficient ratios of Cr to Fe for both vacancies and interstitials. The crossover occurs because the ratio of the vacancy diffusion coefficient in Cr to that in Fe crosses the ratio for interstitials, resulting in a change in Cr RIS direction. When the interstitial and vacancy diffusion coefficient ratios are equal (i.e. at the crossover), the contribution of Cr enrichment by interstitials is balanced by the contribution of Cr depletion by vacancies. Below the crossover temperature, Cr enrichment by interstitials dominates Cr depletion by vacancies, resulting in a net Cr enrichment. Conversely, above the crossover temperature, Cr depletion by vacancies dominates Cr enrichment by interstitials, resulting in a net Cr depletion.

The IK model was also able to reproduce the measured dependence of Cr RIS on bulk Cr concentration. When concentration-dependent interstitial migration energies were input, the IK model predicted decreasing Cr enrichment with increasing Cr concentration, consistent with experiments (Figure 21). This observation can also be explained by the diffusion coefficient ratios of Cr to Fe for both vacancies and interstitials. Implementing composition-dependent interstitial migration energies caused the interstitial diffusion coefficient ratio of Cr to Fe, to change, as illustrated in Figure 22 for the 11-12 wt% Cr interstitial migration energies (solid line) compared to the original 9 wt% Cr interstitial migration energies (dashed line). The shift in the interstitial diffusion coefficient ratio caused two significant effects: (1) the crossover temperature decreased to $\sim 550^{\circ}\text{C}$, and (2) the difference between the vacancy and interstitial diffusion coefficient ratios at 400°C decreased. The latter of these effects explains the observed decrease in Cr enrichment as a function of increasing bulk Cr concentration.

Figure 21. Comparison of composition dependence of Cr RIS between IK model and experimental measurements at 400°C , 3 dpa, 10^{-5} dpa/sec.

Figure 22. Effect of composition-dependent interstitial migration energies on the Cr to Fe interstitial diffusion coefficient ratio for 11-12 wt% Cr (solid lines) compared to that for 9 wt% Cr (dashed line).

An alternative RIS mechanism, the solute drag mechanism, was also investigated. This mechanism considers both solute-interstitial and solute-vacancy complexes. Solute-interstitial complexes are considered as di-interstitial dumbbells. For solute-vacancy complexes, atomic species strongly bound to vacancies would be carried, or “dragged”, along with the vacancy flux to grain boundaries. In this work, the solute drag mechanism was modeled by adding the diffusion of solute-vacancy and solute-interstitial complexes to the existing IK model presented earlier.

The binding energies of Cr-interstitial and Cr-vacancy complexes are positive, suggesting that the solute drag mechanism will always produce Cr enrichment. Thus, it is clear that *the solute drag mechanism is unable to account for the crossover from Cr enrichment to Cr depletion*. Therefore, Cr depletion will not occur if solute drag is a dominant process. And indeed, the model that included the solute drag mechanism demonstrated that the IK mechanism cannot account for Cr-interstitial binding, confirming that attractive Cr-vacancy and Cr-interstitial binding cannot explain Cr depletion.

The RIS measurements from this study are consistent with the IK mechanism. A more rigorous test, however, is to determine whether RIS measurements from the literature can be explained by the IK mechanism. The IK model predicts a concentration-dependent crossover temperature; this trend is an effective way to compare the IK mechanism to literature measurements, as shown in Figure 23. Observations of Cr enrichment should fall below the crossover temperature line, while observations of Cr depletion should fall above the crossover. It is recognized that while the literature data are predominantly from commercial alloys containing a number of minor elements, the IK model does not account for elements other than Fe and Cr. The effect of minor

elements on the crossover temperature, and on Cr RIS behavior in general, is not well known. However, the good match between measurement and model for the temperature dependence of RIS in T91 using sink densities that are close to those calculated from the measured microstructure, indicate that the effect of the minor elements cannot be large. Thus, the crossover temperature for T91 is likely close to that for Fe-Cr.

The majority of RIS measurements from the open literature reported that the direction of Cr RIS is consistent with the IK model prediction for the given alloy composition (open triangles in Figure 23). Two studies observed no Cr segregation (open circles); these studies have bulk Cr compositions and temperatures that fall close to the IK-predicted crossover line, which is reasonable. Several experiments were conducted using techniques that make it difficult to determine the true direction of Cr RIS (open squares). However, in general, the *RIS measurements from the open literature fall into good agreement with the crossover temperature line calculated from the IK model in this study.*

Figure 23. Experimental measurements of the directions of Cr RIS in F-M alloys published in the literature (open symbols), as compared to the crossover temperature calculated in this work and experimental measurements of Cr RIS from (closed symbols).

2.5 Irradiation Creep

The stress dependence of the irradiation creep rate of alloy T91 is found to be linear below 160MPa and follows power law behavior above 160 MPa.

Irradiation creep of T91 was investigated using in-situ proton irradiations with 3.0 MeV protons at temperatures in the range 400-500°C, stresses of 100-200 MPa up to 1-2 dpa. The detailed experimental setup for proton irradiation creep are discussed by Xu et al. [13]. Experiments were designed to isolate the stress dependence of primary creep, and allow an in-depth analysis of anisotropy of the irradiation creep microstructure. The full table of experiments and strain rate results are tabulated in Table 7.

Results of measured irradiation creep rates as a function of stress are shown in Figure 24. The strain rate behavior can be empirically fit by a combination of a linear stress dependence and power law stress dependence. The linear stress behavior at low stress is consistent with irradiation creep driven by dislocation climb, while the power law stress behavior at high stress is consistent with creep behavior driven by dislocation glide [14]. The stress at which dislocation glide overtakes dislocation climb is observed to be 160MPa for irradiation creep. This value is similar to the 170MPa turnover stress observed by Haney et al [15] for thermal creep of T91 at 500°C. The measurements for the irradiation creep experiments suggest dislocations are responsible for the strain rate behavior of ferritic-martensitic steel T91.

Figure 24. Stress dependence of irradiation creep of T91 at 450°C.

Table 7. Sample conditions and strain rate of all irradiation creep experiments

Sample Name	Alloy	Temperature (°C)	Stress (MPa)	Dose Rate (dpa/s)	Dose (dpa)	Strain Rate (10^{-9}s^{-1})	Statistic Error (10^{-9}s^{-1})
IT400160-A	T91	400	160	2.6×10^{-6}	1	2.9	0.11
IT450000-A	T91	450	0	3.4×10^{-6}	1	N/A	N/A
IT450100-A	T91	450	100	3.4×10^{-6}	2	1.67	0.06
IT450120-A	T91	450	120	3.4×10^{-6}	1	1.9	0.025
IT450140-A	T91	450	140	3.4×10^{-6}	1	2.05	0.04
IT450160-A	T91	450	160	3.4×10^{-6}	1	2.7	0.035
IT450180-A	T91	450	180	3.4×10^{-6}	1	4.67	0.037
IT450180-B	T91	450	180	3.4×10^{-6}	1	5.00	0.03
IT450200-A	T91	450	200	3.4×10^{-6}	1	12.5	0.053
IT450200-B	T91	450	200	3.4×10^{-6}	1	11.5	0.032
IT500160-A	T91	500	160	$*3 \times 10^{-6}$	1.5	1.78	0.16
IT500160-A	T91	500	160	$*3.4 \times 10^{-6}$	0.8	2.33	0.096
IT500160-A	T91	500	160	$*4.8 \times 10^{-6}$	1.2	5.08	0.13
IT500180-A	T91	500	180	1×10^{-5}	1	5.78	0.25
IH450160-A	HT9	450	160	3.4×10^{-6}	1	3.4	0.07
IA450160-A	HCM12A	450	160	3.4×10^{-6}	1	5.85	0.02
TT450200-A	T91	450	200	0	0	1.4	0.002
TT500200-A	T91	500	200	0	0	12.8	0.032
TT500200-B	T91	500	200	0	0	11.5	0.042

The anisotropy in the dislocation loop density is observed to increase linearly with stress. Loop size distribution is correlated to both stress and irradiation time.

Four irradiation creep samples were analyzed by TEM for identification of unique irradiation creep microstructure. The method for quantifying localized dislocation loop anisotropy was developed so as to preserve the tensile axis direction throughout the analysis. TEM analysis of the irradiation creep microstructure revealed evidence of stress induced preferential nucleation (SIPN) as shown by the anisotropy in the dislocation loop density.

Figure 25 plots the local anisotropy of the dislocation loop density and dislocation loop size of irradiation creep samples. The x-axis in Figure 25 is defined by the angle between the loop normal and the tensile axis. The dislocation loop density is defined by the grain normalized loop count shown as blue data points in Figure 25. The dislocation loop density is found to have a linear dependence on the orientation to the tensile axis. The higher the applied tensile stress, the larger the effect of the anisotropy on the loop density. The observed anisotropy in dislocation loop density as a function of stress is evidence for preferred nucleation of dislocation loops, lending credibility to the operation of SIPN irradiation creep mechanism [16].

The same localized anisotropy found for dislocation loop density was not found for dislocation loop size as shown by the data points in red in Figure 25. However, the applied stress does act to increase the overall size of dislocation loops. Figure 26 illustrates the dislocation loop size distribution for all four irradiation creep conditions. The dislocation loop size for 200MPa condition is a significantly larger than those of 180MPa. The loop size of the 180MPa conditions are comparable to those found for 100MPa despite only having half of the total dose. The observations from the TEM analysis determined that dislocation loops is both a function of applied stress and the irradiation dose.

a)

b)

c)

d)

Figure 25. Loop anisotropy plot of the irradiation creep experiments for a) IT450100, b) IT450180, c) IT500180, d) IT450200.

a)

b)

c)

d)

Figure 26. Loop size spectrum of irradiation creep samples, a) IT450100, b) IT450180, c) IT500180, d) IT450200.

The SIPN contribution to irradiation creep strain is calculated based on observed anisotropy in the dislocation loop density.

The strain contribution of the dislocation loops are calculated based on TEM observations by carefully accounting for the loop density, loop size, and loop orientation to the tensile axis. Semi-empirical equations are developed to link experimental TEM observations with macroscopic strains that are consistent with the theoretical description. The semi-empirical approach is capable of determining SIPN strain in all three primary directions for a grain situated at any direction relative to the tensile axis is shown in Figure 27. The orientation of any grain relative to the tensile axis is uniquely defined by the angles θ and ϕ . A typical plot of the SIPN contribution as a function of grain orientation is shown in Figure 28.

The result of the SIPN calculations are compared with the total macroscopic strain tabulated in Table 8. The maximum SIPN contribution are found to account for 6-37% of the total strain measured. The longer the irradiation, the less SIPN contributes to the total strain. This observation confirms that SIPN is a transient phenomenon that mainly affects the primary regime of irradiation creep. SIPN is found to be insufficient to fully account for all the irradiation creep strain observed in the sample.

The remaining irradiation creep strain could be explained by preferential absorption of point defects. Although an accurate empirical treatment of the SIPA contribution requires detailed analysis of dislocation network Burgers vector that was not included in the scope of this project, there is indirect macroscopic and microscopic evidence that points to the operation of SIPA. The stress dependence of T91 irradiation creep is found to be linear under 160MPa and higher than linear above 160MPa. This is consistent with the theoretical understanding of creep by preferred absorption of point defects (SIPA) resulting in dislocation climb at low stress, and dislocation glide at high stress [14], [17]. In addition, the effect of applied stress on the dislocation loop size is further evidence for SIPA as the creep mechanism that is operating in addition to SIPN in T91. SIPN and SIPA are identified as the most likely dominant primary irradiation creep mechanism in T91 through irradiation experiments and microstructural analysis. The anisotropy in the microstructure of the material plays a significant importance in the irradiation creep behavior of ferritic-martensitic steels.

Table 8. TEM analysis irradiation creep sample conditions and SIPN strains.

Sample Name	Dose (dpa)	Dose Rate (dpa/s)	Temp (°C)	Applied Stress (MPa)	Irradiation Time (Hours)	Max Calculated SIPN Strain (%)	Measured Total Strain (%)	Percent of SIPN Strain (%)
IT450100-A	~2	3.4×10^{-6}	450	100	170	0.006	0.10	6
IT500180-A	1	1×10^{-5}	500	180	30	0.022	0.06	37
IT450180-A	1	3.4×10^{-6}	450	180	80	0.021	0.13	15
IT450200-A	1	3.4×10^{-6}	450	200	80	0.040	0.36	11

Figure 27. Orientation of a grain with dislocation loop to the tensile axis as defined by θ , and ϕ .

Figure 28. SIPN calculations as a function of the angles θ and ϕ dislocation loop make to the tensile axis for irradiation creep sample IT450200.

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* denotes publications resulting directly from this project

Degrees Awarded

Cheng Xu, Ph.D. 2014
Alicia Certain, Ph.D. 2012
Kevin Field, Ph. D. 2012
Janelle Wharry, Ph.D. 2012

Publications

J. Wharry, G. S. Was, “The Mechanism of Radiation-Induced Segregation in Ferritic-Martensitic Alloys,” *Acta. Mater.* (2104), in press.

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