**ANALYSIS OF STRESS-INDUCED BURGERS VECTOR ANISOTROPY IN PRESSURIZED TUBE SPECIMENS OF IRRADIATED FERRITIC-MARTENSITIC STEEL: JLF-1** - D. S. Gelles (Pacific Northwest National Laboratory)* and T. Shibayama (University of Hokkaido, Japan)

## OBJECTIVE

The objective of this effort is to provide understanding of microstructural evolution in irradiated ferritic/martensitic steels for first wall applications in a fusion reactor.


## SUMMARY

A procedure for determining the Burgers vector anisotropy in irradiated ferritic steels allowing identification of all a<100> and all $\frac{a}{2}$<111> dislocations in a region of interest is applied to a pressurized tube specimen of JLF-1 irradiated at 430°C to 14.3 x $10^{22}$ n/$cm^2$ (E > 0.1 MeV) or 61 dpa. Analysis of micrographs indicates large anisotropy in Burgers vector populations develop during irradiation creep.


## PROGRESS AND STATUS

### Introduction

In a previous report,[1] it was shown that irradiation creep induced Burgers vector anisotropy for perfect dislocations could be determined in pressurized creep tubes of the Japanese duplex ferritic steel JFMS irradiated in the FFTF/MOTA. The procedure used a process of ellimination to identify each of the a<100> and $\frac{a}{2}$<111> Burgers vector in a field of view.

More recently, pressurized tube specimens of JLF-1 were made available for microstructural examination and it was possible to again examine Burgers vector anisotropy.[2] JLF-1 is the first of a series of low activation Fe-9Cr-2W-0.2V steels which were prepared to exclude nickel and molybdenum additions. Three sets of up to four tubes were irradiated, three tubes at 430°C, four tubes at 460°C and four tubes at 520°C. All sets contained unstressed conditions. Microstructural examination of these pressurized tubes was a part of the Monbusho experimental objectives.

### Experimental Procedure

Pressurized tubes of JLF-1 steel were made from tubing fabricated from rod stock. The chemical composition of the rod stock is given in Table 1. Tube segments had dimensions of 0.57 mm outside diameter, 0.20 mm wall thickness, and were 19.8 mm in length. Endcaps of the same material were electron beam welded to both ends of the tubing segments. One endcap had a small (0.13 mm diameter) hole for pressurization to obtain the desired hoop stress at the design irradiation temperature. The pressurization gas was helium. Diameter measurements were made using a non-contacting laser measurement system before and after irradiation. Three specimens were irradiated in level 2 of FFTF/MOTA 2A in canister 4D-2 and 2B in canister 4D-1 at 430°C for an accumulated exposure time of 503.4 equivalent full power

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days, over 12082 h. The estimated accumulated damage in these creep specimens during irradiation was 14.3 x $10^{22}$ n/$cm^2$ (E > 0.1 MeV) or 61 dpa.[2]

Table 1. Nominal Composition of JLF-1 (wt%) with balance Fe

| Cr | C | Mo | W | Mn | Nb | V |
|---|---|---|---|---|---|---|
| 9.04 | .097 | >.01 | 1.97 | 0.46 | na | 0.19 |
| Si | P | S | Ni | N | Ta | Ti |
| >0.1 | na | na | na | na | 0.07 | .001 |

na - not available

Following irradiation and diametral measurement, one JLF-1 specimen, comprising the maximum stress condition following irradiation at the lowest temperature of 430°C (VA08), was selected for microstructural examination and Burgers vector anisotropy measurement.

The specimen was sectioned with a slow speed saw by first removing the end caps and then splitting the tube longitudinally. Curved disks 3 mm in diameter were then punched from the central region of the split tube using a punch designed for tubing specimens. The disks were mechanically ground flat and then thinned for transmission electron microscopy using standard procedures. Examinations were performed using a JEOL 1200EX transmission electron microscope with a double tilting goniometer stage. Column realignment was found to be necessary after each change in specimen tilt.

## Results

### *Diameter change and swelling measurements*

Diameter change measures for the pressurized tube specimens of JLF-1 from the FFTF/MOTA are given in Table 2, and the diametral creep strains are plotted using closed diamonds, as a function of hoop stress in Figure 1, with measurements for HT-9 and JFMS, a duplex steel, for comparison.[3]

Table 2. Creep Data on JLF-1 Pressurized Tubes Irradiated in FFTF

| ID | VA02 | VA05 | VA08 | VA01 | VA04 | VA07 | VA10 |
|---|---|---|---|---|---|---|---|
| Temperature (°C) | 430 | | | 459 | | | |
| Hoop Stress (MPa) | 0 | 30 | 60 | 0 | 30 | 60 | 100 |
| Diametral Strain (%) | 0.04 | 0.13 | 0.27 | 0.03 | 0.08 | 0.16 | 0.23 |

| ID | VA00 | VA03 | VA06 | VA09 |
|---|---|---|---|---|
| Temperature (°C) | 519 | | | |
| Hoop Stress (MPa) | 0 | 30 | 60 | 100 |
| Diametral Strain (%) | na | 0.23 | 0.61 | 0.60 |

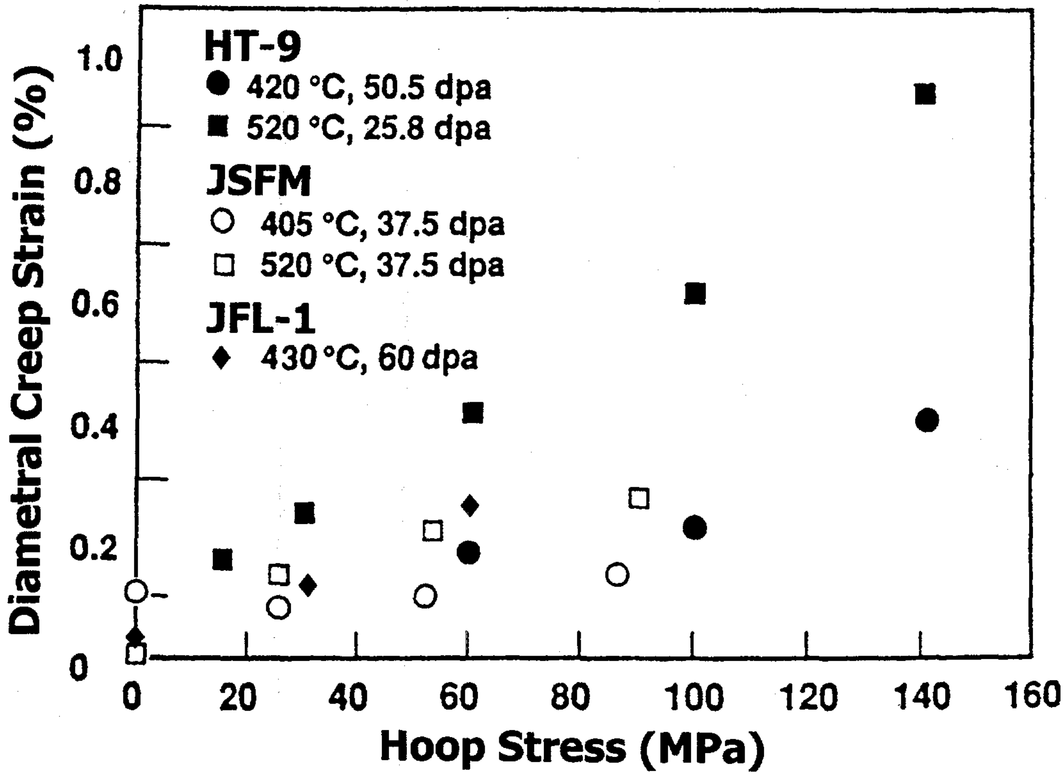


Figure 1. Irradiation Creep Response of JFMS and JLF-1 in Comparison with HT-9 as a Function of Hoop Stress.

From Figure 1, it can be shown that allowing for differences in dose, JLF-1 has creep strengths similar to HT-9 and JFMS. JLF-1 shows non-linear behavior with greater curvature than that for HT-9 indicative of complex behavior such as precipitation. A more detailed description of creep response in ferritic steels has recently been published by Toloczko et al.[4]

Swelling as determined from density change was measured only for specimens VA02 and VA08 of JLF-1. Results are provided in Table 3. Table 3 indicates that swelling was 0.658% for unstressed JLF-1 irradiated at 430°C to 61 dpa whereas under a hoop stress of 60 MPA the swelling increased to 0.885%.

Table 3. Swelling in JLF-1 specimens irradiated at 430°C as a function of stress

| Specimen ID | Stress | Swelling |
|---|---|---|
| VA02 | 0 MPa | 0.658 |
| VA08 | 60 MPa | 0.886 |

Microstructural examination

Microstructural examination of JLF1 was limited to specimen VA08 which had been pressurized to the largest hoop stress of 60 MPa. Furthermore, only dislocation anisotropy was studied in detail. However, irradiated was found to have caused no major changes in microstructure; no void swelling was found and no evidence for further precipitation could be identified. These findings appear contradictory to swelling measurements given in Table 3. A possible explanation lies in the anticipated precipitation of tungsten in the form of $M_{23}C_6$.

An area was selected for analysis of dislocation anisotropy with orientation near (013) and with the tube length effectively parallel to $[01\bar{1}]$. A sequence of six micrographs was taken using $\vec{g}$ = [200], $[01\bar{1}]$ and $[\bar{1}10]$. Three of the images are provided in Figure 2 to show the dislocation structure found within a region bounded by subgrain boundaries. The area shown in Figure 2 has also been analyzed to provide a quantitative estimate for Burgers vector anisotropy. The foil is again approximately 67 nm thick, but the field of view for measurement is similar to that shown in order to avoid subgrain boundary effects. From Table 4, it can be shown that dislocation densities vary between 0.9 and $9.5 \times 10^9$ $cm^{-2}$, corresponding to a factor of about ten variation. Anisotropy develops in both the $\frac{a}{2}$<111> and a<100> populations.

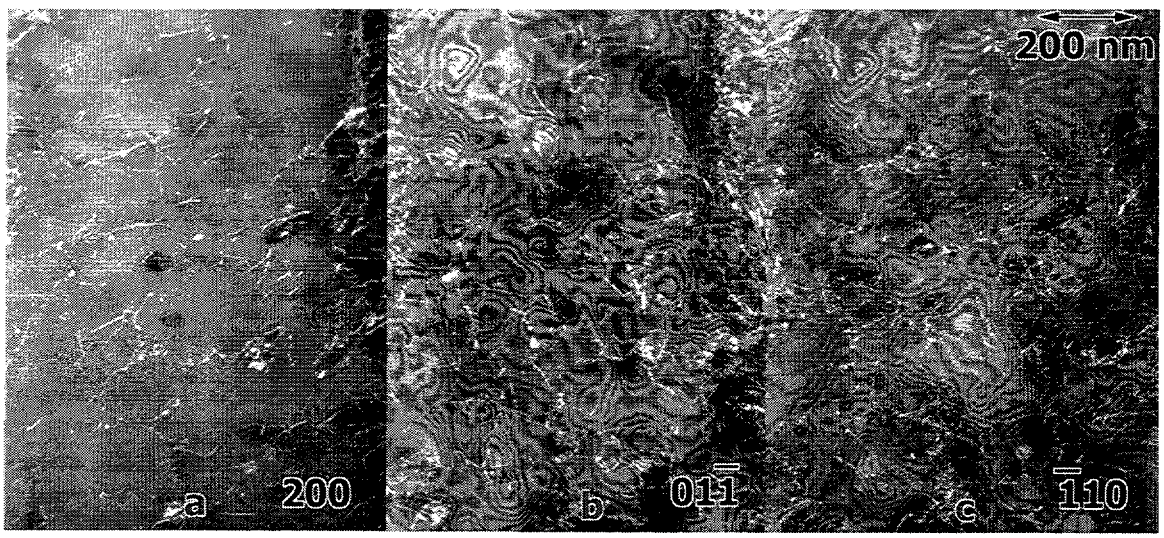


Figure 2. Weak Beam Dark Field Micrographs of an Area in Specimen VA08 Arranged for Comparison of Dislocations using 200 contrast in a), $01\bar{1}$ contrast in b), $\bar{1}10$ contrast in c).

Table 4. Dislocation Density Measurements, Showing Burgers Vector Anisotropy as a function of Burgers vector for specimen VA08 of JLF-1 irradiated at 430°C with a hoop stress of 60 MPa and compared to Specimen RR03 of JFMS Irradiated at 407°C with a Hoop Stress of 86 MPa.[1]

| Burgers vector | | $\frac{a}{2}[111]$ | $\frac{a}{2}[\bar{1}11]$ | $\frac{a}{2}[1\bar{1}1]$ | $\frac{a}{2}[11\bar{1}]$ | a[100] | a[010] | a[001] |
|---|---|---|---|---|---|---|---|---|
| $10^9 cm^{-2}$ | VA08 | 9.47 | 1.38 | 3.03 | 2.65 | 0.89 | 2.27 | 4.17 |
| | RR03 | 1.94 | 1.02 | 0.62 | 4.50 | 1.77 | 4.28 | 4.72 |

## Discussion

This work has provided an opportunity to extend Burgers vector anisotropy determination in the ferritic alloy class. Procedures have been tested, and results have been generated showing that a significant anisotropy can be generated in ferritic/martensitic steels at 400-430°C for stress levels of 60-86 MPa. This discussion section is intended to cover two topics: limitations of the procedure and significance of the results.

The analysis given in Table 4 is based on measurements that may not be statistically significant. The specimen thicknesses were only 67 nm, and only about 40 dislocations were analyzed in the field of view. Dislocation motion may have occurred in such thin foils, although evidence for such motion was not observed. Few dislocation nodes could be found in the analyzed regions to show that the Burgers vector identification did not violated energy balances. Also, dislocation density measurements have not yet been completed on an unstressed specimen.

The results that anisotropy as large as a factor of 8 in JFMS and 10 in JLF-1 between different Burgers vector populations is surprising, given the limited amount of total strain found in the specimens. The strain measured in specimen RR03 was 0.15% and in VA08, it was 0.27%, whereas the unstressed specimen RR00 deformed 0.11% presumably due to precipitation and VA02 deformed 0.04 despite swelling of 0.658%. Therefore, the strain due to irradiation creep may have been as low as 0.04% in RR03 but 0.24% possibly influenced by precipitation in VA08. In comparison, results on austenitic steels provided similar levels of anisotropy in irradiated pressurized tubes, with deformations ranging between -0.02 and 1.255%.[5] The lower value corresponded to a situation where the perfect dislocation population had only just been generated, and therefore the average dislocation velocity was very low. Therefore, it appears that the present results indicate that dislocation motion may have been very limited in the JFMS specimen for the dose level achieved, about 40 dpa possibly caused by the high precipitate density found in the specimen.

## CONCLUSIONS

Procedures have been developed to determine Burgers vector anisotropy in ferritic/martensitic steels containing both $\frac{a}{2}\langle 111\rangle$ and $a\langle 100\rangle$ Burgers vectors. The procedures have been tried on two irradiated pressurized tube specimens of JFMS steel irradiated in the FFTF/MOTA. Analysis of results for the specimens irradiated at stresses of 60 to 86 MPa indicates differences in Burgers vector anisotropy as large as a factor of 10.

## AKNOWLEDGEMENT

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