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Status of FeCrAl ODS Irradiations in the High Flux Isotope Reactor

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OAK RIDGE NATIONAL LABORATORY
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Fuel Cycle Research and Development (FCRD)

Status of FeCrAl ODS Irradiation in the High Flux Isotope Reactor

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ABSTRACT

FeCrAl oxide-dispersion strengthened (ODS) alloys are an attractive sub-set alloy class of the more global FeCrAl material class for nuclear applications due to their high temperature steam oxidation resistance and hypothesized enhanced radiation tolerance. A need currently exists to determine the radiation tolerance of these newly developed alloys. To address this need, a preliminary study was conducted using the High Flux Isotope Reactor (HFIR) to irradiate an early generation FeCrAl ODS alloy, 125YF. Preliminary post-irradiation examination (PIE) on these irradiated specimens have shown good radiation tolerance at elevated temperatures (≥330°C) but possible radiation-induced hardening and embrittlement at irradiations of 200°C to a damage level of 1.9 displacement per atom (dpa). Building on this experience, a new series of irradiations are currently being conceptualized. This irradiation series called the FCAD irradiation program will irradiate the latest generation FeCrAl ODS and FeCr ODS alloys to significantly higher doses. These experiments will provide the necessary information to determine the mechanical performance of irradiated FeCrAl ODS alloys at light water reactor and fast reactor conditions.

1. INTRODUCTION

A recent interest has grown on developing novel materials deemed to have enhanced accident tolerance in light water reactor (LWR) designs. At the core of this development, are materials with increased high temperature oxidation resistance in steam environments compared to the currently deployed Zr-based components. FeCrAl-based alloys with minor Y additions are promising near-term materials for this application as they can form passivating Al_2O_3 oxide scale in steam environments up to 1450 °C while limiting the heat and hydrogen production that can exacerbate accident conditions [1,2]. Within the more global FeCrAl material class exists a sub-set of alloys known as oxide dispersion strengthened (ODS) FeCrAl alloys. These FeCrAl ODS alloys are characterized as having been produced via powder metallurgy techniques and contains a fine dispersion of oxide nanoclusters in the typically ferritic grain structure. FeCrAl ODS alloys are of interest as they can exhibit similar steam oxidation resistance compared to wrought FeCrAl alloys but have superior high temperature mechanical strength.

FeCrAl ODS alloys could also be attractive materials as their small grain sizes and fine dispersions of nanoclusters leads to increased defect density. Zinkle and Snead have shown that high sink strength materials (nominally $>10^{16}$ m^{-2}) can exhibit increased radiation tolerance compared to lower sink strength counterparts [3]. Initial investigations into the radiation tolerance of wrought FeCrAl alloys, which have lower sink densities than FeCrAl ODS alloys, have focused on examining the compositional effects and cold-working effects on the formation of radiation-induced defect structures. These studies have shown that radiation leads to the formation of dislocation loops and radiation-enhanced precipitation of the Cr-rich α' phase [4–6]. These radiation-induced/enhanced microstructural features affect the general mechanical properties of the alloys after irradiation and could be the limiting factor of the component made from a wrought FeCrAl alloy.

It is hypothesized that similar results to those presented by Zinkle and Snead can be achieved using FeCrAl ODS alloys, and the radiation tolerance, and hence the operation lifetime can be greatly increased for this material class by using ODS material. A series of neutron radiation exposures followed by post-irradiation examination (PIE) is currently on-going to test this hypothesis. Preliminary work included the irradiation of 125YF, an FeCrAl ODS variant, alongside wrought FeCrAl alloys using the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). This irradiation series is deemed the FCAT (Fe-Cr-Al-Tensile) irradiation program. The irradiation program includes the irradiation of sub-sized SS-J2 tensile specimens to doses of 2 dpa, 8 dpa, and 16 dpa at temperatures of 200°C, 330°C, and 550°C. The lowest doses irradiation capsules have recently become available for PIE while the higher doses samples are awaiting insertion in the HFIR for irradiation.

A new series of irradiation capsules are currently being designed and will be fabricated to test a greater range of FeCrAl ODS and FeCr ODS alloys at elevated temperatures and doses. This irradiation series is deemed the FCAD irradiation series. It is anticipated that these irradiation capsules will be irradiated to 2 dpa, 16 dpa, and 50+ dpa at temperatures of 330°C and 550°C within the HFIR at ORNL. The objective of this report is to summarize the initial findings based on preliminary PIE of the FCAT irradiation program in respect to the ODS alloy and provide insight into the test matrix and designs of the FCAD irradiation program.

2. FCAT IRRADIATION PROGRAM

2.1 Materials

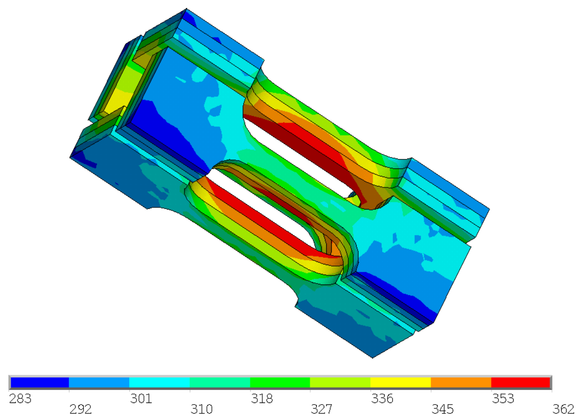
The FCAT irradiation program [7] included irradiation of 125YF, a first generation FeCrAl ODS alloy developed by ORNL. The alloy was produced from 12 wt.% Cr, 5 wt.% Al and balance Fe gas atomized powder. The powder size ranged from 45 µm to 150 µm before ball milling for 40h in an Ar atmosphere in a Zoz CM08 Simoloyer. Additionally, Y_2O_3 and FeO powder were added to the ball mill. A 10:1 ball-to-charge ratio was used. Ball milled powder was degassed at 300°C for 24h in a mild steel extrusion can. After degassing, the powder and can was sealed, heated for 1h at 950°C and then extruded at 950°C.

The resulting consolidated alloy had a composition of 83 wt.% Fe, 11.67 wt.% Cr, 4.9 wt.% Al, 0.01 wt.% Ti, 0.19 wt.% Y, 200 ppm C, 202 ppm N, 1920 ppm O, and 30 ppm S. Grain sizes were maintained near 0.4 – 0.5 µm with an average grain aspect ratio of 1.08. Full details on the alloy can be found elsewhere [8]. SS-J2 tensile specimens were manufactured from the extruded feedstock using electric discharge machining (EDM). EDM burn layers were mechanically removed when possible to limit the effect of these layers on subsequent mechanical tests. Individual samples were laser engraved with unique identifiers allowing tracking through the build, irradiation, and PIE processes.

2.2 Irradiation capsule design

Irradiation capsules which house both SS-J2 type and newly developed SS-2E type tensile specimens were developed for use within ORNL's HFIR [7]. Two SS-J2 specimens of the 125YF alloy were loaded in each irradiation capsule. The remaining positions within the capsule were filled with wrought FeCrAl alloys. Capsules also include passive SiC thermometry. These SiC specimens are used as they can validate the modeled target irradiation temperatures have been met by using post-irradiation dilatometry techniques [9]. Specimen temperatures were controlled by changing the radial gas gap between the sub-assembly and outer housing of the irradiation capsule. He gas was used as the fill gas, no temperature control was completed using varying gas compositions.

Determination of the radial gas gap and the irradiation capsule operating temperature was calculated using an ANSYS finite element analysis (FEA) design. A 3D model was completed as it provides the greatest insight into the thermal gradients within the design during irradiation. An example of the thermal gradients in 4 stacks of 3 SS-J2 tensile specimens within the FCAT irradiation design is given in Figure 1. Figure 2 shows the predicted temperature contours of the SiC passive thermometry in the same capsule. Note the SiC bulk temperatures are markedly higher than the specimens, which is due to the thermometry being oriented within the specimens and radially closer to the assembly center. Capsule outer housing material varied depending on nominal target irradiation temperature. Lower irradiation temperatures (typically <450°C) used Al 6061-T6 while higher irradiation temperature capsules used Mo-based alloys.



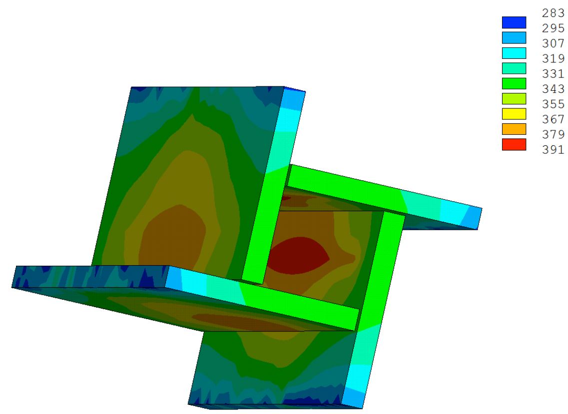


Figure 1: Predicted specimen temperature contours for a 330°C irradiation in HFIR.

Figure 2: Predicted thermometry temperature contours for a 330°C irradiation in HFIR.

2.3 Irradiation test matrix

The FCAT irradiations were designed to investigate the contributions of composition, microstructure, irradiation dose, and irradiation temperature on the radiation tolerance of wrought and ODS FeCrAl alloys. To accomplish this task, 3 damage dose levels at 2 dpa, 8 dpa, and 16 dpa and temperatures at 200°C, 330°C and 550°C were selected. This resulted in a total of nine different irradiation capsules being designed, built, and irradiated. Central axial positions were selected for each irradiation capsule to minimize axial thermal gradients which could lead to difficulties in interrupting PIE results. A summary of the FCAT irradiation test matrix is provided in Table 1.

Table 1: Summary of FeCrAl capsule irradiation conditions.

Capsule ID	Exposure Time (hrs)	Neutron Flux (n/cm^2s) E > 0.1 MeV	Neutron Fluence (n/cm^2) E > 0.1 MeV	Dose Rate (dpa/s)	Dose (dpa)	Irradiation Temperature (°C)
FCAT-01	548	1.10×10^{15}	2.17×10^{21}	9.8×10^{-7}	1.9	200*
FCAT-02	548	1.04×10^{15}	2.05×10^{21}	9.3×10^{-7}	1.8	363.6 ± 23.1
FCAT-03	548	1.10×10^{15}	2.17×10^{21}	9.8×10^{-7}	1.9	559.4 ± 28.1
FCAT-04	2304*	1.10×10^{15}	8.68×10^{21}*	9.8×10^{-7}*	7.6*	200*
FCAT-05	2304*	1.04×10^{15}	8.20×10^{21}*	9.3×10^{-7}*	7.2*	330*
FCAT-06	2304*	1.10×10^{15}	8.68×10^{21}*	9.8×10^{-7}*	7.6*	550*
FCAT-07	4608*	1.10×10^{15}	1.74×10^{22}*	9.8×10^{-7}*	15.2*	200*
FCAT-08	4608*	1.04×10^{15}	1.64×10^{22}*	9.3×10^{-7}*	14.4*	330*
FCAT-09	4608*	1.10×10^{15}	1.74×10^{22}*	9.8×10^{-7}*	15.2*	550*

*Target values represented, nominal values are currently being determined

2.4 Preliminary PIE

The low dose (<2 dpa) samples have recently become available for PIE. Currently, the passive SiC thermometry is being analyzed to determine if the target irradiation temperatures have been met. Dilatometric analysis was conducted up to a maximum temperature of 600°C at a constant ramp rate of 1°C/min and a cooling rate of 2.5°C/min using a Netzsch 402 CD dilameter. A standardized algorithm was used to determine the nominal irradiation temperatures [9]. To date, the FCAT-02 irradiation capsule has fully completed SiC dilaometric analysis, the FCAT-03 is partially completed and the FCAT-01 is awaiting analysis. Table 2 summarizes the completed analysis. Table 2 shows that the nominal average irradiation temperatures appear within ~35°C of the specimen design temperatures for both the 330°C and 550°C irradiation capsules. As stated earlier, the SiC resides within in central portion of the capsule which is modeled to be the highest temperature regime. This would suggest actual irradiation specimen temperatures are much closer to the design temperatures.

Table 2: Results of dilatometric analysis of SiC thermometry bars contained within irradiated capsules.

Capsule ID	Specimen ID	Minimum Temperature (°C)	Median Temperature (°C)	Maximum Temperature (°C)	Average Median Temperature (°C)
FCAT-02	14 TOP	346.2	358.1	372.5	370.6 ± 17.6
	16 TOP	359.9	383	405.7	
	18 MID	336.4	345.2	368.2	364.9 ± 27.9
	20 MID	394.1	384.6	402.1	
	22 BOT	337.9	329.5	342.4	355.5 ± 36.7
	24 BOT	369.9	381.4	403.1	
	Average	**363.6 ± 23.1**			
FCAT-03	26 TOP	450.1	524.3	571.5	556.7 ± 45.8
	28 TOP	635.0	589.1	601.1	
	30 MID	517.7	551.0	574.5	551.0
	34 BOT	488.4	573.1	597.6	573.1
	Average	**559.4 ± 28.1**			

A single tensile specimen of 125YF from FCAT-01, FCAT-02, and FCAT-03 have been mechanically tested using uniaxial tensile tests to evaluate the mechanical performance of the alloy after irradiation. Tensile tests were completed using an Instron universal test machine with a crosshead speed of 0.0055 mm/s resulting in a nominal strain rate of $\sim 10^{-3}$ s^{-1}. No contact or non-contact extensometery was completed at the time of the test. All tests were performed at room temperature in air. The resulting stress-strain curves from these tests are shown in Figure 3. Yield strengths were found to be between 1037 and 1108 MPa. The 330°C, 1.8 dpa and the 550°C, 1.9 dpa specimens showed reasonable uniform elongations and total elongations with values at 5-6% and 9-12%, respectively. Low temperature irradiations at 220°C resulted in higher embrittlement with an almost complete loss in work hardening and a total elongation of 5%. The results shown in Figure 3 would suggest some degree of irradiation temperature dependence on the mechanical response of irradiated ODS FeCrAl alloys. Further PIE efforts, including advanced microstructural characterization, are needed to provide more insight on the radiation tolerance of this alloy class.

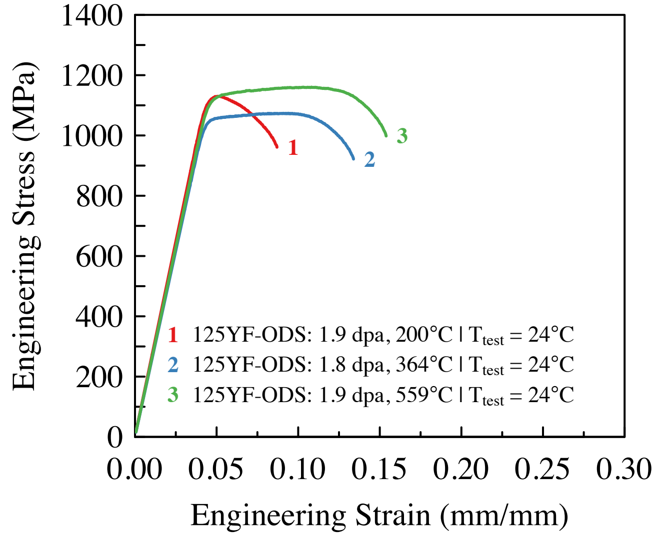


Figure 3: Plots showing representative stress-strain curves for the 125YF alloy after irradiation in the HFIR.

3. FCAD IRRADIATION PROGRAM

3.1 Irradiation Capsule Design

The same irradiation capsule design will be used for the FCAD series as the FCAT series in an effort to minimize cost and time to deployment. This capsule design is a very flexible design and can be loaded solely with SS-J2 tensile specimens, SS-2E tensile specimens, or a mixture of both. Here, it is planned to fill a capsule with solely SS-J2 sample geometries, resulting in 36 total specimens per irradiation condition. As discussed, the capsule also provides positions for passive SiC thermometry allowing for verification of irradiation temperature. The sub-assembly design, shown in Figure 4, has proven to be an effective design allowing for easy disassembly while in hot cell.

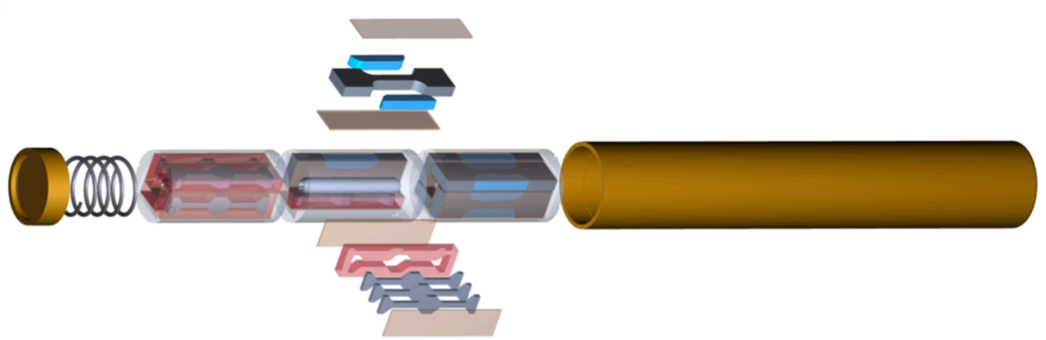


Figure 4: Finalized HFIR irradiation capsule design for FCAD irradiation program. Design is identical to the FCAT irradiation capsules.

3.1.1 Irradiation Test Matrix

The test matrix has been developed to provide cross compatibility with irradiation conditions of the FCAY and FCAT irradiation programs [10] while also providing the high dose radiation response of these alloys. High dose responses are of interest as FeCrAl ODS alloys could have potential applications in other nuclear reactor designs such as fast reactors being developed by TerraPower, LLC. The proposed FCAD test matrix is provided in Table 3.

Table 3: Summary of FCAD irradiation test matrix.

Capsule ID	Exposure Time (hrs)	Neutron Flux (n/cm^2s) E > 0.1 MeV	Neutron Fluence (n/cm^2) E > 0.1 MeV	Dose Rate (dpa/s)	Dose (dpa)	Irradiation Temperature (°C)
FCAD-01	548	1.10×10^{15}	2.17×10^{21}	9.8×10^{-7}	1.9	330
FCAD-02	548	1.10×10^{15}	2.17×10^{21}	9.8×10^{-7}	1.9	550
FCAD-03	4384	1.10×10^{15}	1.74×10^{22}	9.8×10^{-7}	15.2	330
FCAD-04	4384	1.10×10^{15}	1.74×10^{22}	9.8×10^{-7}	15.2	550
FCAD-05	13152	1.10×10^{15}	5.21×10^{22}	9.8×10^{-7}	45.6	330
FCAD-06	13152	1.10×10^{15}	5.21×10^{22}	9.8×10^{-7}	45.6	550

The alloy loading list is still in development. Currently, it is planned to use 14YWT as a reference material to the newly developed FeCrAl ODS alloys. It is also planned to include several Fe-12Cr ODS alloy variants to test the influence of Al and other alloying elements on the radiation tolerance of the alloys currently in development. Finally, several wrought FeCrAl alloys will be fielded to provide a contrast between the ODS and wrought alloy variants. At least 3 tensile specimens per alloy will be loaded per irradiation conditions resulting in 12 different alloys to be tested.

3.1.2 Schedule

The FCAD irradiation capsules are still undergoing final design and development. It is planned in U.S. fiscal year 2017 that samples and capsule internals will begin fabrication followed by inspection and final assembly. The lowest dose irradiation capsules will run for 1 cycle in the HFIR, while the 15.2 dpa will run for 8 cycles and the 45.6 dpa will run for 24 cycles. Insertion of capsules late in fiscal year 2017 would result in low dose capsules being ready for PIE in late 2017, early 2018 while the higher dose specimens will take an additional year and 3 years before PIE can begin. PIE for all capsules is planned to be similar to PIE already completed on the FCAY and FCAT irradiation capsules.

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