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ALLOY DEVELOPMENT FOR IRRADIATION PERFORMANCE QUARTERLY PROGRESS REPORT FOR PERIOD ENDING JUNE 30, 1980

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from Contributions of Participating Laboratories

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	<p style="margin-left: 40px;"><i>The handbook activity is progressing slower than desired with inputs. The primary delay seems to center around conflicting priorities between preparing for reactor experiments and preparing data sheets. A portion of the data sheets, those relating to fatigue crack growth and irradiation creep, have been received and are being reviewed. To speed up the turn around time for these data sheets an alternate approach is being tried which involves placing more emphasis on the people working on projects since they have to be responsive to project needs in a timely manner.</i></p>	
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	<p style="margin-left: 40px;"><i>Three-dimensional neutronic calculations are under way to follow the irradiation environment of the MFE-4A experiment, which has recently been loaded into the Oak Ridge Research Reactor (ORR), and are in progress to determine the response of the ORR to multiple tungsten core pieces that will be used during later irradiations to reduce the thermal flux levels seen by the nickel-containing alloys. Preliminary results indicate that two core pieces of 50% W or four core pieces of 25% W can be used simultaneously in the reactor. However, since additional fuel is required to maintain criticality, the core fuel loading exceeded the standard 6 kg ²³⁵U by about 15%. In addition, one-dimensional neutronic calculations are being planned to determine the heating rates within these tungsten core pieces and within the experimental capsules.</i></p>	

2.3	ORR-MFE-4: A Spectral Tailoring Experiment to Simulate the He/dpa Ratio of a Fusion Reactor in Austenitic Stainless Steel (Oak Ridge National Laboratory)	10
	<i>This experiment consists of two capsules, each with two isothermal chambers. The first, ORR-MFE-4A, operates at 300 and 400°C; the second, ORR-MFE-4B, will operate at 500 and 600°C. The ORR-MFE-4A experiment is now in the reactor, operating successfully. It contains a total of 1326 specimens almost entirely of type 316 stainless steel and Path A PCA.</i>	
2.4	Neutronic Calculations for the Conceptual Design of an In-Reactor Solid Breeder Experiment, TRIO-01 (Oak Ridge National Laboratory)	24
	<i>Preliminary one-dimensional neutronic calculations have been carried out to determine the tritium production and heating rates within the solid fusion breeder material Li₂O, which is to be irradiated in the ORR. Where the amount of ⁶Li was reduced to 0.5% (the naturally occurring amount is 7.5%), the maximum values obtained for tritium production and heating rates are 3.3×10^{19} atoms/s m³ and 34 MW/m³.</i>	
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	<i>Sheet and rod stock of austenitic stainless steels have been exchanged between the U.S. and USSR fusion reactor materials irradiation effects programs. The United States supplied type 316 stainless steel, while the USSR submitted a low-nickel, high-manganese austenitic stainless steel.</i>	

- 3.2 Fatigue Crack Growth Testing of 316 SS for MFE-5 (Hanford Engineering Development Laboratory) 42

The last in a series of tests demonstrating the prototypic fatigue machine has been successfully completed. An eight-specimen chain test was conducted at 425°C with one specimen being continuously monitored using the electrical potential method. Results were used to finalize precracking and loading conditions for the in-reactor test.

- 3.3 The Microstructure and Mechanical Properties of 20%-Cold-Worked Types 316 Stainless Steel and 316 + 0.23 wt % Ti After HFIR Irradiation at 55 to 375°C (Oak Ridge National Laboratory) 48

The 20%-cold-worked types 316 and 316 + Ti stainless steel were irradiated in HFIR at temperatures of 55 to 375°C to neutron fluences producing up to 13 dpa and 740 at. ppm He. Both steels offer similar mechanical properties at these irradiation temperatures as determined by postirradiation tensile testing. Both alloys are strengthened by irradiation, but the ductility of cold-worked 316 decreases while that of cold-worked 316 + Ti increases. The failure mode was ductile-transgranular for all irradiated specimens. Microstructural examination reveals cavities and dislocation loops at 285 and 375°C in both alloys. Both alloys swell less than 0.5% and even less at higher irradiation temperature. The titanium modified alloy swells slightly less.

- 3.4 Composition and Microstructure of Precipitate Phases in Austenitic Stainless Steels (Oak Ridge National Laboratory) 75

Precipitation in thermally aged and neutron-irradiated samples of types 316 and 316 + Ti stainless steel is characterized by conventional transmission electron microscopy and by extraction and x-ray energy dispersive spectroscopy. Below 750°C, the major phases in the thermally aged alloys are eta, tau, Laves, sigma, and MC in 316 + Ti. Each phase has a unique and clearly distinguishable characteristic solubility for the alloy elements found in these steels. Particularly important to irradiation behavior is the fact that all the phase except MC and tau can concentrate Si, but only eta concentrates Ni. Relative to this baseline, these phases have nearly the same composition when produced by irradiation in HFIR. The phase MC is exceptional in that it is the only phase that is enhanced by irradiation and yet incorporates little or no Si or Ni.

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Machining of the specimens is complete. Calibration of equipment to monitor crack growth is complete. The environmental test chamber for fatigue testing has been set up.

5.2	Tensile Properties of Helium-Injected and Reactor-Irradiated V-20 Ti (Oak Ridge National Laboratory)	138
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Sheet tensile specimens of annealed V-20% Ti alloy have been preinjected with 90 and 200 at. ppm He by use of 60-MeV (9.6-pJ) alpha particles at the Oak Ridge Isochronous Cyclotron (ORIC). Some of the samples were then irradiated in row 7 of the Experimental Breeder Reactor (EBR)-II at temperatures of 400, 525, 575, 625, and 700°C to neutron fluences of 2×10^{26} to 4×10^{26} neutrons/m² (>0.1 MeV).

Tensile properties and fractography of the injected and the irradiated specimen have been compared with those of the control specimen. This comparison showed that the helium injection increased the strength at test temperatures beta, 500°C and decreased the elongation above 500°C. The reduction of the elongation depended on the amount of injected helium. The loss of ductility resulting from the neutron irradiation was much greater when the specimens were irradiated and tested at temperatures above 600°C.

5.3	Fatigue Behavior of Unirradiated Path C Alloys (Oak Ridge National Laboratory)	146
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Results of exploratory fatigue tests on annealed Nb-1% Zr showed that this alloy has about the same fatigue resistance at room temperature and at 650°C. It was also shown that the fatigue lifetime of Nb-1% Zr is somewhat superior in comparison with average values obtained for 20%-cold-worked type 316 stainless steel tested at low strain ranges by a factor of about 10.

Fatigue tests of the ADIP heat of V-15% Cr-5% Ti are presently under way. Test results obtained to date indicate that at room temperature this alloy has marginally better fatigue resistance than Nb-1% Zr. Only one fatigue test on V-15% Cr-5% Ti at 650°C has been completed. This data point fell beta, the average trend curve of Nb-1% Zr.

6.	PATH D ALLOY DEVELOPMENT — INNOVATIVE MATERIAL CONCEPTS	153
6.1	Development of Iron-Base Alloys with Long-Range-Ordered Crystal Structure (Oak Ridge National Laboratory)	154

The iron-base LRO alloys with compositions $(Fe,Ni)_3V$ are being developed for fusion energy applications. The alloys have excellent high-temperature strength and good ductility and fabricability in the ordered state. Studies of phase relations indicate that σ phase precipitates from the disordered solid solution (γ) at temperatures above the critical ordering temperature (T_c). Below T_c , atom ordering takes place in the alloys through the peritectoid reaction $\gamma + \sigma \rightarrow \gamma'$, where γ' is the cubic ordered phase ($L1_2$ -type) formed on the face-centered cubic (fcc) lattice. The σ phase region can be reduced or eliminated by adding small amounts of titanium. Tensile tests as a function of temperature indicate that the LRO alloys with base compositions show a ductility minimum around T_c . However, modifying the alloys with titanium significantly improves their ductility at elevated temperatures. The tensile properties of the base and titanium-modified alloys are not affected by long-term aging at 550°C. Preliminary evaluation of material prepared by using commercially produced ferrovanadium as feed material indicates this as a promising approach to lowering the alloy production cost.

6.2	The Effect of 4-MeV Nickel Ion Irradiation on the Micro-structure of $(Fe,Ni)_3V$ Long-Range-Ordered Alloys (Oak Ridge National Laboratory)	162
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The unirradiated microstructure of $(Fe_{61}Ni_{39})_3V$ contains a relatively uniform distribution of small MC-type precipitate particles and large, widely scattered islands of σ phase. Essentially the same alloy, except for a 0.4 wt % Ti addition, contained scattered inclusions with MC-type precipitate particles decorating the dislocations at the inclusion-matrix interfaces.

Irradiation of $(Fe_{61}Ni_{39})_3V$ at 525, 570, 625, and 680°C with 4-MeV (0.6-pJ) nickel ions to 70 dpa with the simultaneous injection of helium and hydrogen produced cavities, a high density of dislocation loops, and a redistribution of MC-type precipitate particles. The alloy remained ordered throughout the irradiation except at 680°C, which was above its critical ordering temperature of about 670°C. The irradiation also produced a high density of dislocations in the titanium-modified alloy but only a few small cavities at 525 and 570°C. No cavities were observed at either 625 or 680°C. The MC-type particles precipitated in the modified alloy at all

bombardment temperatures. As the bombardment temperature was increased, the precipitate particle size also increased. However, the number of these particles decreased. The mechanism by which titanium enhanced the resistance of $(Fe_{61}Ni_{39})_3V$ to ion damage is unclear.

6.3 Properties of Rapidly Solidified Nuclear Grade 316 Stainless Steel (Massachusetts Institute of Technology) 174

Nuclear grade 316 stainless steel was processed by rapid solidification. Mechanical testing (room temperature and high temperature tensile, Charpy impact, low cycle fatigue and stress rupture testing) showed that the rapidly solidified alloy in all instances either matches or exceeds to an important degree the mechanical properties of ingot material; notable exceptions include long term creep at 650°C due to the much finer grain size of the RS alloy.

6.4 Optimization of Structure and Properties of Prime Candidate Alloy (PCA) by Rapid Solidification (Massachusetts Institute of Technology) 183

A high density of heterogeneous nucleation sites for helium trapping was provided by reducing the grain size, by increasing the TiC content and surface area and by controlling the dislocation structure by rapid solidification and subsequent thermomechanical treatments. Thermomechanical treatments were developed which resulted in higher strength and elongation in stress rupture testing at 650°C than in a 20% cold worked reference state. While rapid solidification allowed for a controlled TiC precipitate size, density and distribution, coarsening may effectively limit the applications of TiC dispersion strengthened austenitic stainless steel at higher irradiation temperatures.

7. PATH E ALLOY DEVELOPMENT — FERRITIC STEELS 191

7.1 Procurement and Conversion of the National Fusion-Ferritic Steel Program 12Cr (HT-9) Heat (General Atomic Company) 192

A 3,830 kg (8,440 lb.) slab of 12Cr-1Mo-0.3V steel was fabricated into four different plate thicknesses for use by the National Fusion-Ferritic Steel Program. Details of the conversion are discussed in this progress report.

7.2 The Fracture Properties of a Temper Embrittled
 12Cr-1Mo-0.3V Steel (General Atomic Company) 201

Dynamic fracture toughness, as measured by precracked and instrumented Charpy V-notch specimens, and standard Charpy V-notch tests were performed at temperatures from -192° to 207°C a material in two conditions. Material was austenitized for 1 hour at 1000°C, air cooled, and tempered at 650°C for 1 hour; and austenitized and tempered for 1 hour at 1000°C and 650°C and air cooled, and subsequently aged at 550°C for 100 hours. The aging was done at a temperature known to cause temper-embrittlement in this class of steels (1,2,12). Optical metallography of the microstructure and X-ray analysis of extracted carbides are presented. Fractography to relate the mode of fracture with energy absorbed, and a more complete microstructural evaluation continues and will be presented in the next ADIP quarterly.

1.3 Tempering and Transformation Behavior of HT9 Weldments
 (Sandia National Laboratories) 216

Dilatometric measurements of HT9 weld and base material indicated that the on-heating transformation to austenite occurs at approximately 840°C and that the martensite start temperature (M_s) upon cooling from above the upper critical temperature (A_{c3}) occurs at 240°C. Postweld heat treatment of autogenous gas tungsten arc (GTA) welds results in a variety of composite microstructures consisting basically of tempered martensite and secondary carbides. The tempering response is relatively sluggish at tempering temperatures below 600°C. A one-hour heat treatment at 800°C reduced the martensitic hardness in the fusion zone and heat-affected zone (HAZ) to nearly base metal values. Tempering curves for both the fusion zone and HAZ are presented.

7.4 Fatigue Crack Growth in Path E and Path B Alloys (Hanford
 Engineering Development Laboratory) 226

Fatigue crack growth tests on unirradiated HT-9 in helium at 150, 300, 500 and 600°C and on 9Cr-1Mo at 25°C in air have been conducted. Room temperature air testing of solution treated and aged B1, B2, B3, B4 and B6 alloys has been completed and is compared to the 50% cold-worked B alloys tested under similar conditions. Comparisons of measured fatigue crack growth are made with 20% cold-worked 316 stainless steel.

7.5 Analysis of Single Specimen Tests on HT-9 for J_{1c} Determination (Hanford Engineering Development Laboratory) 236

Fracture toughness tests were performed on unirradiated 2.54 mm thick HT-9 specimens at 25, 149, 232, 315, 427 and 539°C. The electropotential technique was applied to develop single specimen method for J_{1c} determination. Based on experimental data, a semi-empirical expression was obtained for a calibration curve which was used to measure continuous crack extension. With this continuous Δa measurement, it is possible to generate J versus Δa curves from single specimens at various temperatures successfully. Large specimens were also tested at 232°C to study the thickness of HT-9. The upper shelf fracture toughness of HT-9 is evaluated at temperatures ranging from room temperature to 539°C.

7.6 Environmental Effects on Properties of Ferritic Steels (Argonne National Laboratory) 255

Calibration fatigue tests were conducted with HT-9 ferritic steel specimens at 755 K in a flowing lithium environment. The procedure for strain control and strain measurement has been established. Several continuous cycle fatigue tests have been performed with gauge specimens of HT-9 alloy. The results are being analyzed to determine the strain-life relationship. Exposure of corrosion specimens of HT-9 steel with solid Li_2O , $LiAlO_2$, and Li_2SiO_3 breeding materials at 873 K has been completed. Metallographic evaluation of the corrosion specimens is in progress.

7.1 Specimen Preparation and Loading for the AD-2 Ferritics Experiment (Hanford Engineering Development Laboratory) . . . 260

Experimental hardware has been built and specimens have been fabricated for the AD-2 experiment. The loading of the specimens into the six uninstrumented B-7C capsules has been completed and these capsules have been shipped to Idaho Falls for insertion into ERR-11, Cycle 109.

7.8 Characterization of Ferritic Steels for HFIR Irradiation (Oak Ridge National Laboratory) 294

Small heats of ferritic (martensitic) steels based on 12%Cr-1%Mo and 9%Cr-1%Mo were prepared containing about 0, 1, and 2%Ni. Additional heats were made with 2%Ni, in which the content of the ferrite-forming elements was adjusted to restore the net chromium equivalent to a value near that of the unmodified alloy. During irradiation in the High Flux Isotope Reactor (HFIR), transmutation of the ^{58}Ni will give helium concentrations approximating those

produced in such steels in fusion reactor service. Because the addition of nickel can affect the response to heat treatment, the microstructures of the alloys are being characterized.

After normalizing, the alloys without nickel and those containing 1 and 2% Ni (but with no chromium equivalent adjustments) had microstructures that were entirely martensite. The 2% Ni addition lowered the A_{c1} temperature, making it necessary to temper these alloys at temperatures no greater than 700°C. The normalized microstructure of the 2% Ni alloys with adjusted chromium equivalent contained large amounts of a phase in addition to the predominant martensite. Work is in progress to determine if this phase is δ -ferrite or retained austenite.

8. STATUS OF IRRADIATION EXPERIMENTS AND MATERIALS INVENTORY 309

8.1 Irradiation Experiment Status and Schedule 310

The following bar charts show the schedule for all ADIP reactor irradiation experiments. Experiments are presently under way in the Oak Ridge Research Reactor (ORR) and the High Flux Isotope Reactor (HFIR), which are mixed spectrum reactors, and in the Experimental Breeder Reactor (EBR)-II, which is a fast reactor.

During the reporting period irradiation was begun for two irradiation experiments: ORR-MFE-4A in the ORR and HFIR-CTR-33 in the HFIR.

8.2 ETM Research Materials Inventory (Oak Ridge National Laboratory and McDonnell Douglas Astronautics Company) 319

The Office of Fusion Energy has assigned program responsibility to ORNL for the establishment and operation of a central inventory of research materials to be used in the Fusion Reactor Materials research and development programs. The objective is to provide a common supply of materials for the Fusion Reactor Materials Program. This will minimize unintended materials variables and provide for economy in procurement and for centralized record-keeping. Initially this inventory is to focus on materials related to first-wall and structural applications and related research, but various special purpose materials may be added in the future.

9. CORROSION TESTING AND HYDROGEN PERMEATION STUDIES 325

9.1 Hydrogen Dissolution and Permeation Studies of ADIP Program Alloys (Argonne National Laboratory) 326

No report for this period. The next reporting of progress on this task will be at the end of the fourth quarter of FY-1980.

9.2 Vanadium Alloy/Lithium Pumped-Loop Studies (Argonne National Laboratory) 327

No report for this period. The next reporting of progress on this task will be at the end of the fourth quarter of FY-1980.

9.3 Compatibility of Static Lithium with a Long-Range-Ordered Fe-Ni-V Alloy and 2 1/4 Cr-1 Mo Steel (Oak Ridge National Laboratory) 328

Tests of 2 1/4 Cr-1 Mo steel in static lithium at 400, 500, and 600°C for exposures up to 3000 h have been completed. Analysis of the post-test lithium from the 500- and 1000-h experiments at 500°C indicated a decarburization of the 2 1/4 Cr-1 Mo steel, which probably caused the observed decreases in yield and ultimate tensile strengths of the steel when exposed to lithium at 600°C for 3000 h. A long-range-ordered Fe-31.8 Ni-22.5 V-0.4 Ti (wt %) alloy with a critical ordering temperature of 680°C was compatible with static lithium after 2000 h at 650 and 710°C.

9.4 Mass Transfer of Type 316 Stainless Steel in Lithium Thermal-Convection Loops (Oak Ridge National Laboratory) 337

The possible effects of nitrogen in lithium-type 316 stainless steel thermal-convection systems are discussed. Variations in the normally low nitrogen concentrations of the lithium did not significantly alter the short-time dissolution rate. However, the presence of nickel in lithium may influence the distribution of the dissolved elements around the loops through nickel interactions with other elements (especially chromium) in the lithium.