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Program Annual Progress Report for Period
Ending June 30, 1977**

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MAGNETIC FUSION ENERGY MATERIALS TECHNOLOGY PROGRAM ANNUAL
PROGRESS REPORT FOR PERIOD ENDING JUNE 30, 1977

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FOREWORD

The objectives of the Magnetic Fusion Energy (MFE) Materials Technology Program, which is described in this report, are to continue to solve the materials problems of the Fusion Energy Division of ORNL and to meet needs of the national MFE program, directed by the ERDA Division of Magnetic Fusion Energy (DMFE). This work is a continuation of the program described in previous annual progress reports.^{1,2} The principal areas of work include radiation effects, compatibility studies, materials studies related to the plasma-materials interaction, materials engineering, radiation behavior of superconducting magnet insulation, and mechanical properties of superconducting composites. The level of effort and schedules are consistent with Logic II of the DMFE Program Plan.³

In addition to the experimental work described in this report, an intense effort has been directed toward the development of a National materials program plan. Four task groups have been convened by the Materials and Radiation Effects Branch of DMFE. These task groups and chairmen are as follows:

- Alloy Development for Irradiation Performance — J. O. Stiegler, ORNL
- Plasma Materials Interactions — W. Bauer, SLL
- Damage Analysis and Fundamental Studies — D. G. Doran, HEDL
- Special Purpose Materials — J. L. Scott, ORNL

The attention of the first three task groups is directed mainly at the blanket structural material and plasma-materials interactions. The Special Purpose Materials Task Group is concerned with other crucial generic materials problems related to coolants, breeding materials, tritium barriers, graphite, ceramics, heat-sink materials, and magnet materials. The results of the deliberations of the various task groups should become available within the next year.

¹J. L. Scott, comp., *Controlled Thermonuclear Materials Technology Program Annu. Prog. Rep. June 30, 1975*, ORNL-5082.

²J. L. Scott, comp., *Magnetic Fusion Energy Materials Technology Program Annu. Prog. Rep. June 30, 1976*, ORNL 5189.

³DMFE Staff, *Fusion Power By Magnetic Confinement Program Plan*. ERDA-76/110/0-4 (July 1976).

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Design studies in The Next Step⁴ (TNS) and the Demonstration Reactor⁵ (DEMO) provide important guidance to the materials program by identifying viable systems and operating conditions. Since a major goal of the materials program is to provide information needed for the successful design and operation of the DEMO reactor, the conclusions of the DEMO study relative to the technological requirements for commercial tokamak fusion power are especially relevant. These conclusions are listed below.⁵

1. The blanket structural material. It is our judgment that some type of austenitic stainless steel will be capable of achieving the desired 10 to 20 MW-year/m² integral wall loading. Moreover, the unique helium production reactions associated with nickel-bearing alloys in thermal neutron fluxes allow an excellent simulation of fusion reactor neutron radiation effects in existing fission reactors. Although much additional work will be required to qualify an austenitic stainless steel for use in commercial fusion power plants, we are very enthusiastic about the prospects for such an alloy. At the same time we recommend continued work on refractory metals, especially niobium- and vanadium-base alloys.

2. The blanket coolant. In pursuing the choice of coolant, major emphasis was placed on the coolant circuit being capable of operating at low pressure. On this basis, it appears that sodium-potassium salts (of which nitrate-nitrites, hydroxides, and chlorides are candidates) offer the most promise. The nitrate-nitrite salt mixtures have been used most extensively in industrial applications, and therefore they have been recommended for primary consideration. These salt mixtures exhibit relatively low melting points (~150°C), and also they are relatively inexpensive (~\$1/kg). The salt thermal stability and compatibility with iron-base alloys seem acceptable to temperatures of ~500°C. A development program will be required to determine the

⁴M. Roberts, *Conclusions from the Oak Ridge EPR Studies and Initiation of the TNS Program - A Brief Summary*, ORNL-5278 (March 1977).

⁵D. Steiner et al., *ORNL Fusion Power Demonstration Study: Interim Report*, ORNL/TM-5813 (March 1977).

ultimate acceptability of these salts in a fusion reactor environment. We recommend that helium and liquid lithium be pursued concurrently as potential blanket coolants.

3. The breeding material. It appears that liquid lithium offers the greatest potential for the breeding material. Lithium yields the highest flexibility with regard to tritium breeding. It has good thermal properties and acceptable chemical compatibility. Much further work is still required in the areas of tritium recovery and MHD effects.

4. The power conversion system. The recommended power conversion system would consist of a primary and intermediate salt loop coupled to a conventional steam cycle. The primary purpose of the intermediate heat transport loop is to prevent pressurization of the low-pressure blanket by high-pressure steam should there be a tube leak in the steam generator. Assuming a primary loop salt exit temperature of about 450°C, a steam cycle thermodynamic efficiency of about 35% can be achieved with this power conversion system. The tokamak operates in a cyclic mode (e.g., a 20-min burn time followed by a 1-min down time, yielding a duty cycle factor of 95%). Salt storage tanks coupled to the intermediate heat transport loop would provide the necessary thermal storage to ensure continuous power to the line during the entire tokamak operating cycle.

5. The coil system. It appears that superconducting coil requirements for the tokamak power plant can be satisfied with Nb-Ti as the superconducting material. Moreover, normal conducting coils may also be feasible and desirable under certain conditions. For example, the application of normal conducting coils is not feasible for the toroidal field system of tokamak reactors that operate at low power density ($\sim 1 \text{ MW/m}^3$ in the plasma) because, at low power density, the fusion power output would be comparable to the ohmic power losses in the coil system. In order to get an acceptable power balance, the thermal fusion power release should be at least an order of magnitude greater than the ohmic heating losses in the coil system. If tokamaks can operate at high power density ($5\text{--}10 \text{ MW/m}^3$ in the plasma) and if the required field on axis is in the range 3.0–4.5 T, then it appears that normal conducting toroidal coils may be practical in some applications

for reactor systems. Thus, our conclusion is that the major thrust of a superconducting magnet development program should be directed to Nb-Ti and that serious consideration should be given to the application and development of normal conducting coils for use in tokamak reactor systems. Work should, of course, continue on advanced superconducting materials.

6. The pulsed electrical system. It appears that the primary energy storage requirements of the poloidal field driving system and the plasma heating system can be satisfied with motor-generator flywheel sets similar to those being proposed for the Tokamak Fusion Test Reactor (TFTR). Advanced energy storage concepts, such as homopolar generators and superconducting energy storage devices, have been proposed for the energy storage needs of tokamak fusion reactors. However, our studies indicate that the more conventional and currently available technology of motor-generator flywheel sets will be acceptable on the basis of both engineering and economics considerations. The advanced energy storage concepts should be pursued, but they do not appear to be necessary for commercial feasibility.

As a result of the DEMO design input, the emphasis in the Radiation Effects and Materials Engineering Programs is placed on stainless steels and nickel-base alloys. The ability of mixed-spectrum fission reactors to effectively simulate the fusion reactor environment also influences these priorities. Since it is clearly too early to select a reference material for the DEMO reactor, we do not neglect refractory metals in our program. Samples that have been preinjected with helium are being tested together with noninjected controls.

Since lithium is the preferred breeding material and the Compatibility Program has a limited budget, almost all the work is directed at lithium compatibility with various structural alloys. Because of the advantages of a low-pressure coolant, some effort is directed toward molten-salt coolants. There is also great interest in helium as a coolant, and we have a sizeable program that is a part of our High-Temperature Gas-Cooled Reactor Program. Since this work is not funded by DMFE, it is not included in this report.

The efforts relative to plasma-materials interactions and magnet development are necessarily much more near term than are the other materials programs. Here the emphasis is on *in situ* diagnostics for the Impurities Studies Experiment (ISX) and on laboratory simulations of the phenomena observed in ISX, and its predecessor tokamak, ORMAK. The magnet-related activities are closely coupled with the ORNL Magnetics and Superconductivity Program.⁶

This report contains a summary description of the materials work done in the last year for the MFE program.

⁶Staff of ORNL, *Fusion Energy Division Annu. Prog. Rep.* Dec. 31, 1976, ORNL-5275, pp. 199-222.

SUMMARY

1. RADIATION EFFECTS

The Radiation Effects Program is concerned with the irradiation performance of materials that are candidates for application in the first wall and high-flux regions of fusion reactor blankets. The largest effort of this program is the evaluation of radiation effects in austenitic stainless steels. Experimental work on nickel-base alloys, refractory metal alloys, and sintered aluminum product (SAP) is also continuing. The program goals are to develop improved alloys tailored to optimum service in fusion reactors and to define the reactor performance limits imposed by these structural materials.

Examination of 20%-cold-worked type 316 stainless steel irradiated in the HFIR in the temperature range 580 to 600°C shows that recrystallization is occurring for fluences producing about 4 dpa. Recrystallization is not observable at 1.5 dpa and complete at 40 to 60 dpa. Swelling due to cavity formation in this temperature range is approximately linear with transmutation-produced helium content, but in going through the fluence range where recrystallization occurs the cavity concentration decreases by almost an order of magnitude. Titanium added to type 316 stainless steel reduces the amount of swelling during irradiation and enhances the postirradiation tensile ductility. The improved properties result from the better accommodation of the helium produced by the irradiation. The mechanism is nucleation of clusters of small bubbles on a fine distribution of TiC particles.

New phases of the work on austenitic stainless steel initiated in the past year include the irradiation of samples preinjected with helium, evaluation of irradiation effects in weldments, and the study of fatigue properties of irradiated samples. Samples of type 316 stainless steel cyclotron implanted with helium are under irradiation in EBR-II at temperatures in the range 400 to 700°C. Samples are either small sheet tensile specimens for postirradiation tensile tests or pressurized tubes for the determination of helium effects on irradiation creep. The

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weldment evaluation program has been initiated with welds in type 316 stainless steel made by the gas-shielded metal-arc process. All initial irradiations are on samples taken transverse to the welds, with weld metal in the central portion of the gage section. Irradiation in EBR-II and HFIR span the irradiation temperature range from about 80 to 620°C. The fatigue testing equipment has been assembled and is under test outside the hot cells to develop operating procedures. The first experiment containing ten hourglass fatigue samples is under irradiation at 300°C in a HFIR peripheral target position.

Irradiated samples of Inconel 600 and PE-16 have been tensile tested after immersion density measurements to determine swelling. Swelling in the Inconel 600 ranged from 0 to almost 4%, with the swelling weakly temperature dependent between 300 and 600°C but markedly greater for irradiation at 650 and 700°C. Swelling is generally less in PE-16, with significant swelling found only for irradiation temperatures above 600°C. Tensile tests show strength increases for irradiations at 300 to 400°C and strength decreases for irradiation at 600 to 700°C. Tensile elongation are lowest at the extremes of this temperature range, with greatest ductility for irradiation and tests at 400 to 500°C. Irradiation experiments currently in HFIR will expand the range of fluence on PE-16 specimens to both higher and lower values.

The microstructures of molybdenum alloys irradiated at temperatures between 425 and 1000°C have been examined to evaluate the swelling. The swelling and microstructures are qualitatively similar in Mo, Mo-0.5% Ti, and TZM. The defect concentration in the alloys is higher than in the pure molybdenum. The suppression of voids in TZM irradiated at lower fluences did not persist to the higher fluence of this experiment (2.5×10^{26} n/m²).

A review of the available experimental data from irradiations using neutrons with higher energies than found in fission reactors shows that property changes can be quantitatively related to fission reactor results. The results scale roughly with damage energy, but the scale factor probably depends on the specific property being measured.

This success in correlating damage produced in different neutron spectra justifies the extensive use of HFIR and EBR-II and the beginning use of the ORR in partially simulating fusion reactor operation conditions. Details of irradiation experiments in these reactors are given in appropriate sections of the report. To quantify further the comparisons of materials and facilities, the damage parameters of a number of elements have been calculated for a fusion reactor first-wall neutron spectrum. The results are useful in synthesizing the response of any alloy or in evaluating the sensitivity of this response to changes in alloy composition.

Immersion density and tensile property measurements of irradiated SAP containing He and ^3H indicated little change in the properties. The remaining samples have been postirradiation annealed and are awaiting tests to further determine the effect of gas distribution on the properties.

Two new techniques are being evaluated for their usefulness as probes of the defect microstructure produced by irradiation. These are small-angle scattering of neutrons or x rays and positron annihilation. The small-angle scattering techniques are responsive to cavities and also to other defect clusters. Positron annihilation is sensitive to any defect cluster of vacancy nature. Both techniques are being evaluated against electron microscopy results, and will be judged on the ability to be more thorough or more economical than TEM.

The highlights of other papers published in the reporting period are given in the final section of the Radiation Effects chapter.

2. SURFACE STUDIES

The *in situ* SXAPS study in ORMAK was terminated at the time the tokamak experiment was permanently shut down at the end of 1976. The study pointed out the severity and extent of the hydrocarbon contamination in ORMAK and showed the relative effectiveness of hydrogen and oxygen discharge cleaning as well as the rapidity of recontamination following cleaning by the glow discharge.

The development of the surface analysis station for ISX is proceeding rapidly, with the phase I system being essentially completed. This will provide an operational Auger electron spectrometer and an ultrahigh-vacuum sample transfer apparatus to be used in monitoring the cleanup and other surface phenomena at the first wall as ISX begins operation.

Development of the Wall Material Test Facility has continued with the construction of a multielement hot-cathode gas discharge apparatus, which provides a high degree of operating flexibility. Initial studies of hydrogen adsorption and desorption phenomena at the surface of oxidized stainless steel samples have shown that greater than 10^{20} atoms/m² may be easily sorbed in the surface region during a glow discharge and then rapidly desorbed over a period of a few seconds when the discharge is terminated. The reservoir of hydrogen in the surface region is much larger than that in the plasma, and exchange with the plasma is rapid. Hydrogen recycle is thus likely to strongly influence plasma behavior.

3. COMPATIBILITY STUDIES

Corrosion rates of type 316 stainless steel were measured in lithium at 500 to 650°C and described by an Arrhenius-type equation. Additions of Si, Mg, Co, Al, and C to lithium were tested in capsules to determine their effectiveness as corrosion inhibitors for stainless steels. Solubility of Li₂O in Li was measured from 195 to 734°C in a cooperative study with ANL. Corrosion rates of type 316 stainless steel were determined in molten Li₂BeF₄ at 650°C and in a nitrate-nitrite heat-treating salt at 430 and 505°C. The rates were much lower than those found for type 316 stainless steel and Hastelloy N in molten NaOH-KOH at 365 to 430°C.

4. MATERIALS ENGINEERING

A program is outlined to identify and solve the materials engineering problems associated with the design of fusion reactors.

5. MAGNET DEVELOPMENT

A cooperative program has been initiated with AERE-Harwell, IGC, and Sumitomo Electric Inc., Ltd., Japan, to provide superconducting composite samples to ORNL for evaluation. Harwell and IGC are supplying Nb₃Sn multifilamentary (MF) conductors and Sumitomo has supplied V₃Ga conductors. The Harwell material has been heat-treated at ORNL to provide varying thicknesses of Nb₃Sn layers on niobium cores. The thickness of this layer has been found to be the most important parameter determining the mechanical properties of the conductor. Metallographic examination of multifilamentary Nb₃Sn conductors strained to 0.8% at 4.2 K showed intermittent cracking of the Nb₃Sn layer, which would result in irreversible degradation in superconducting properties. Stress-induced heating produced substantial amounts (10^3 – 10^5 GJ/m³) of heat during initial deformation and cyclic stressing.

The effect of mixed (neutron and gamma) radiation on organic and organic-inorganic composite electrical insulators at liquid helium temperatures is being studied in support of the development of superconducting magnets. Electrical and mechanical properties of a variety of insulators proposed for use in magnets are being measured as a function of radiation dose. Equipment for testing and for irradiation is in various stages of design, construction, or use.

6. NEUTRONICS CALCULATIONS FOR RADIATION DAMAGE STUDIES

During the past year the primary knock-on atom (PKA) data base RECOIL, derived from ENDF/B-IV data and composed of most elements of interest to the magnetic fusion energy (MFE) community, has been used extensively to calculate radiation damage in metals (i.e., gas production and atom displacements) for many different neutron environments. Some of the information generated includes radiation damage of materials by a fusion-reactor first-wall neutron spectrum. Also, information generated by RECOIL was used to obtain the expected increase in radiation damage in the superconducting magnets due to penetrations (injector tubes, diagnostic ports, etc.) in the shield of a Tokamak fusion reactor. Sensitivity calculations on some of the data contained in the RECOIL data base are in progress to ascertain what data should

be improved. This analysis includes the sensitivity of the group-average cross sections (damage and gas) to different in-group weighting schemes and the sensitivity of some of the damage energy partial cross sections to different angular distributions.

1. RADIATION EFFECTS

F. W. Wiffen and J. O. Stiegler

The performance of early fusion reactors will be limited to some extent by the properties of materials. The Radiation Effects Program is directed at defining the irradiation performance limits of materials having potential application in the first wall and high-flux regions of the blanket. This work is complicated by lack of a high-flux irradiation facility with a fusion reactor neutron spectrum. Until such facilities are developed, the program must be based on simulation techniques. Fission reactor irradiations are used to investigate materials for fusion reactor application in our program. These irradiations will establish acceptable operating ranges and performance limits for classes of materials that are candidates for fusion reactor applications. Our attention is divided between stainless steels, nickel-base alloys, and refractory-base alloys.

The experimental work is being restructured to bring new experiments in line with the program plan developed by DMFE's Alloy Development for Irradiation Performance task group. As most of the results reported here are from experiments under way before the formation of this task group, there has been no attempt to connect these results with the program plan.

1.1 THE IRRADIATION RESPONSE OF AUSTENITIC STAINLESS STEELS

1.1.1 Microstructures of HFIR-Irradiated Type 316 Stainless Steel - P. J. Maziasz

Irradiation in the HFIR produces large amounts of displacement damage and helium in nickel-bearing alloys and can thus help develop an understanding of the response of type 316 stainless steel in a

fusion reactor environment. Previous work^{1,2} has shown that the large amounts of helium produced during these irradiations result in different microstructures than would irradiation that produces displacement damage alone in annealed and cold-worked type 316 stainless steel. The helium affects both the nature and magnitude of the response. The HFIR irradiations produced equilibrium helium bubbles for almost all cases examined, while irradiation that produces only displacement damage with very little helium leads to voids: i.e., cavities with little or no helium. The size and concentration of the cavities determines the swelling. Both size and concentration are affected differently in annealed and cold-worked material, since each of these has a different distribution of sinks that collect the helium produced. In both annealed and cold-worked material, the primary effect of helium is to increase the cavity concentration. In annealed material irradiated without producing large helium concentration, below about 550 or 600°C nucleation of cavities is heterogeneous and primarily on precipitates at the low fluences. It is somewhat more homogeneous in the matrix at high fluences. In HFIR irradiations with high helium levels, the size of the homogeneously nucleated cavities is little affected by high helium levels, but the concentration is increased. The net result is increased swelling in the irradiations that produce helium. In addition, the high helium levels result in grain boundaries that are decorated with cavities, which also contribute to swelling. In cold-worked material, only occasional cavities are observed in recovered areas after irradiation that produces low helium levels. At the high helium levels of HFIR irradiations, cavities are smaller but orders of magnitude more numerous than for low helium concentrations. Nucleation occurs preferentially at or near dislocations.

¹F. W. Wiffen and E. E. Bloom, "Effect of High Helium Content on Stainless Steel Swelling," *Nucl. Technol.* 25: 113-23 (January 1975).

²P. J. Maziasz, F. W. Wiffen, and E. E. Bloom, "Swelling and Microstructural Changes in Type 316 Stainless Steel Irradiated Under Simulated CTR Conditions," pp. I-259-88 in *Radiation Effects and Tritium Technology for Fusion Reactors*, CONF-750989 (March 1976).

We have established the effect of fluence during irradiation at 580 to 600°C on the recrystallization of 20%-cold-worked type 316 stainless steel. We had previously reported² that the microstructure was recrystallized after irradiation to 40-60 dpa, 3000-4000 at. ppm He. At lower fluences (1.5-3.0 dpa, 30-80 at. ppm), the microstructure is partially recrystallized at fluences producing 60 at. ppm He or greater, and unrecrystallized below 60 at. ppm He. Tensile tests showed the recrystallized samples to have very low total elongation values (~0.5% or less), whereas the unrecrystallized sample has a total elongation of 2.75%.

The swelling of 20%-cold-worked type 316 stainless steel irradiated at 600°C is plotted as a function of helium content in Fig. 1.1. It is nearly linear. The microstructure shows very much

Fig. 1.1. Swelling of 20%-Cold-Worked Type 316 Stainless Steel as a Function of Helium Content. Irradiation at about 600°C to fluences producing 3.0 to 60.0 dpa.

smaller cavities and almost an order of magnitude higher cavity concentration at 80 at. ppm He than at 4000 at. ppm He. (See Table 1.1 and Fig. 1.2.) This can be explained by similar behavior of the cold-worked and annealed materials after recrystallization. During recrystallization cavity coalescence is aided by dislocation rearrangement, and these cavities subsequently grow as irradiation continues.

Table 1.1. Cavity Statistics for 20%-Cold-Worked Type 316 Stainless Steel Irradiated at 600°C in HFIR

Property Observed	Low-Fluence Sample	High-Fluence Sample
Helium content, at. ppm	86.0	4070
Displacement damage, dpa	3.3	60.0
Immersion density decrease, %	0.47	3.3
Matrix cavity fraction, vol %	0.06 ± 0.03	3.0 ± 0.5
Cavity concentration, number/m ³	$(2.4 \pm 0.7) \times 10^{21}$	$(3.3 \pm 3.0) \times 10^{20}$
Average diameter, nm	7.1 ± 0.6	65 ± 14
Microstructural observations	Partially recrystallized	Recrystallized with grain growth occurring

In summary, helium has been shown to affect the microstructures of both annealed and cold-worked type 316 stainless steel. It thereby affects such important properties as swelling and total tensile elongation. Levels of helium expected to be produced during fusion reactor operation increase swelling and decrease ductility in both annealed and cold-worked material relative to results from breeder reactor irradiation conditions. Temperature dependence studies at a given helium level (3000–4000 at. ppm) have shown that the loss of cold-worked microstructure results in a loss of irradiation resistance (in terms of swelling and elongation). Fluence dependence studies show that recrystallization occurs at a low fluence (30–80 at. ppm He) during irradiation at 600°C, leading to an early loss of irradiation resistance at this temperature.

Fig. 1.2. Microstructures in 20%-Cold-Worked Type 316 Stainless Steel. Irradiated at about 600°C to (a) 3.0 dpa and 80 at. ppm He and (b) 60 dpa and 4100 at. ppm He. Note the very small (~ 10 -nm-diam) cavities in the recrystallized region and along the cell walls of the low-fluence sample in (a).

1.1.2 Comparison of the Response of Type 316 and Titanium-Modified Type 316 Stainless Steel to Simulated Fusion Reactor Irradiation — P. J. Maziasz and E. E. Bloom

Adequate ductility and swelling resistance are material properties required for successful performance of fusion reactor structures. Ductility is important because fracture of a fusion first wall will shut down the reactor by impurity quenching of the plasma. Swelling can be a major contributor to the stress causing failure, so swelling resistance is important through its coupling to the mechanical properties. Neutrons from the deuterium-tritium plasma will produce both helium and displacement damage. These fusion neutron effects are simulated in HFIR irradiation.

Previous studies¹⁻³ of type 316 stainless steel have shown that cold working can improve the irradiation resistance over annealed material. The addition of 0.23 wt. % Ti has been shown⁴ to further improve the irradiation resistance. Better postirradiation creep-rupture life after simulated fusion irradiation resulted from the titanium addition in type 316 stainless steel.

Solution annealed (1 hr at 1050°C) and 20%-cold-worked samples of both type 316 and titanium-modified type 316 (0.23 wt. % Ti) were irradiated in HFIR. The cold-worked material was irradiated at about 600°C to 30-80 at. ppm He and 1.5-3.0 dpa. The annealed material was irradiated at 550 to 800°C to 2000-4000 at. ppm He and 30-60 dpa. Displacement damage is produced by the fast neutrons, while the helium is produced via a two-step reaction of ⁵⁸Ni with thermal neutrons. Elevated temperatures were produced by nuclear heating of the specimens and a helium gas gap barrier to heat flow to the reactor coolant. The samples were tensile tested at a strain rate of 0.0028/min at temperatures near the irradiation temperatures, from 350 to 800°C.

³E. E. Bloom and F. W. Wiffen, "The Effects of Large Concentrations of Helium on the Mechanical Properties of Neutron-Irradiated Stainless Steel," *J. Nucl. Mater.* 58: 171-84 (1975).

⁴P. J. Maziasz and E. E. Bloom, "Comparison of Type 316 and Titanium-Modified Type 316 Stainless Steels Irradiated in HFIR," *Magnetic Fusion Energy Materials Technology Program Annu. Prog. Rep.* June 30, 1976, ORNL-5189, pp. 5-8.

Limited data for irradiations above 550°C show increased ductility in titanium-modified type 316 stainless irradiated in the annealed condition as compared with annealed type 316 (Fig. 1.3).

Fig. 1.3. A Comparison of Total Tensile Elongation of Titanium-Modified Type 316 and Type 316 Stainless Steel as a Function of Test Temperature for Test Temperatures Close to the Irradiation Temperature. Both alloys were solution annealed 1 hr at 1050°C before irradiation.

Samples irradiated to low fluences in the 20%-cold-worked condition at 600°C also show improvement of titanium-modified type 316 relative to type 316 stainless steel (Fig. 1.4). Also included are results on a cold-worked sample of the same heat of type 316 stainless irradiated in EBR-II at the same temperature to over twice the fluence. This result of EBR-II irradiation, where helium content is only a few atomic parts per million, demonstrates the severity of irradiation producing both helium and displacement damage compared with irradiation producing only displacement damage.

Swelling of these same samples has been measured by immersion density and transmission electron microscopy. It had previously

Fig. 1.4. A Comparison of Total Tensile Elongation of Titanium-Modified Type 316 and Type 316 Stainless Steel as a Function of Helium Content. Both alloys were cold worked 20% before irradiation.

been shown^{1,2} that swelling is much greater for HFIR-irradiated type 316 than EBR-II-irradiated type 316 (Fig. 1.5) and that swelling is reduced by cold working type 316 stainless steel. Swelling is reduced by cold work because cavity size decreases, more than compensating for the cavity concentration increase. Also, irradiation produces less swelling in annealed titanium-modified type 316 than in annealed type 316 stainless steel. This results from the matrix cavity size remaining unaffected while the cavity concentration is less in the titanium-containing material than in type 316 stainless steel. The grain boundary cavity size is also less by a factor of 5 to 7 in the titanium-modified alloy. In 20%-cold-worked type 316 swelling is also reduced by the addition of titanium. This occurs by reduction in the cavity concentration despite a slight increase in the cavity size.

Fig. 1.5. A Comparison of Swelling Versus Temperature for Annealed Type 316 and Titanium-Modified Type 316 Stainless Steel.

Cavity nucleation sites are also affected by chemical composition. Annealed titanium-modified type 316 stainless steel shows cavities nucleating preferentially at or near small precipitate particles. The matrix is denuded of cavities in the zone surrounding the precipitates, similar to grain boundary denuded zones (Fig. 1.6). This effect may also indicate that the boundary between the matrix and the small precipitate rods is the preferred helium collection site.

The precipitation response of annealed titanium-modified type 316 is different from that of type 316 stainless steel. The response may be considered in terms of matrix and grain boundary response, similar to the treatment of cavities for swelling. Chi phase is the primary intragranular phase in type 316 irradiated in the temperature range 480 to 600°C. It forms as small thin plates, 0.1 to 0.3 μm across. Lesser amounts of M_{23}C_6 occur in the form of large plates. The grain boundaries contain M_{23}C_6 particles at 480 to 600°C and

Fig. 1.6. Irradiated Annealed Alloys. (a) Type 316 stainless steel irradiated at about 580°C to 1800 at. ppm He and 29 dpa. (b) Titanium-modified type 316 stainless steel irradiated at about 600°C to 2000 at. ppm He and 28 dpa. The average cavity/diameter (a) is about 53 nm, whereas in (b) it is about 22 nm, with the cavities at the precipitate particles being from 8 to 15 nm in diameter. The large, blocky precipitate particles are believed to be the same in both samples. The cavities in (b) show a strong tendency to cluster on TiC particles.

very large globular sigma phase particles, a few micrometers in diameter, above 600°C. Helium bubbles are usually found along the precipitate-free portions of the boundary.

Titanium carbide is the primary matrix precipitate phase in annealed titanium-modified type 316 stainless steel irradiated between 580 and 680°C. It forms as small rods (0.1–0.3 μm long), which are generally surrounded by clusters of cavities. Large plates of M_{23}C_6 are also formed, and occasional particles of Laves phase are observed. The grain boundaries contain sigma phase above 600°C. The most striking feature in the titanium-modified alloy is the interaction between the cavities and the TiC precipitate, as shown in Fig. 1.6(b).

Both preirradiation microstructure and titanium addition can affect the irradiation response of type 316 stainless steel. A key difference is in the accommodation of a fixed amount of helium in the different alloy microstructures. This is reflected in different macroscopic responses, especially in measured ductility and swelling. The titanium-modified alloy has smaller cavities on the grain boundaries, less swelling, and greater ductility. The fine precipitate of TiC in titanium-modified type 316 appears to be an important factor. It is inferred to be a powerful collection site for helium, resulting in clustering of small helium bubbles around the precipitate particles.

The conclusions are summarized as follows:

1. Titanium-modified type 316 stainless steel shows greater total elongation and lower swelling than does type 316 in both the annealed and cold-worked conditions after irradiation simulating fusion conditions.
2. Titanium-modified type 316 stainless steel accommodates helium better than does type 316 because helium bubbles cluster on a very fine scale on TiC particles.
3. The improved accommodation of helium in the modified alloy results in lower swelling and greater postirradiation ductility.

1.1.3 The Response of Welds to Irradiation - F. W. Wiffen, J. A. Horak, and J. F. King

Weldments in the first wall and front sections of the blanket of a fusion reactor will be exposed to approximately the same operating conditions as will the base metal. The solution used in fission reactors, where closure welds on fuel pins extend well out of the high flux region, probably cannot be adapted to fusion reactor design. The result of these considerations is that the irradiation response of weld metal, of the weld-affected zones in the base metal, and of the base metal are all of equal concern.

Austenitic stainless steels will most likely be joined by a gas-shielded arc welding process, and experiments for the evaluation of irradiation effects in welds have been initiated using welds made in this way. Welds have been made between sections of 6-mm-thick (0.25 in.) base plate of type 316 stainless steel in the 20%-cold-worked condition. Two different filler metals were used, either type 316 stainless steel or 16-8-2. Initial experiments are using specimens taken through the welds, containing weld metal in the central gage portion of the specimens. The weld section and location of the tensile specimens that were made from the welded sections are shown schematically in Fig. 1.7.

Weld-containing tensile specimens of type 316 stainless steel have been included in EBR-II and HFIR irradiation experiments. The EBR-II, row 7 experiment (X-287) contains samples at 400, 525, and 575°C. Specimens in HFIR are under irradiation at 55°C and throughout the temperature range 280 to 620°C. On completion of the irradiation, the specimens will be tensile tested at various temperatures. Supporting fractography, metallography, and microscopy will be used as required.

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Fig. 1.7. Schematic Representation of Weld Design, Sample Location, and Sample Orientation for Irradiation Effects Study on Weldments in 20%-Cold-Worked Type 316 Stainless Steel. Dimensions: thickness, 6 mm; land, 3 mm; specimen length, 39.37 mm; gage diameter, 2.03 mm; button, 4.06 mm.

1.1.4 Fatigue Properties of Fusion Reactor First-Wall Materials — M. L. Grossbeck and K. Liu

The pulsed operation of Tokamak fusion reactors will cause cyclic stresses in structural components. The first wall will also be subjected to helium formation and displacement damage. We are developing an experimental program to evaluate the effect of cyclic stresses on irradiated materials containing large quantities of helium. In an ideal program specimens would be exposed in a fusion reactor neutron spectrum to stress and thermal cycles expected during reactor operation. Since suitable irradiation facilities do not exist and stress cycles are only approximately known, initial studies will employ postirradiation tests of materials irradiated in fission reactors.

A recent materials engineering study⁵ indicates that for a wall life goal of 10 MW years/m² the fatigue life of stainless steel may

⁵B. Cramer, McDonnell Douglas Astronautics Corporation, private communication, 1977.

not be adequate. However, alloy modification and microstructural control based upon an understanding of the effects of irradiation damage and fatigue may significantly extend wall life.

1.1.4.1 Summary of Previous Research

Previous experiments have examined the effects of irradiation on the fatigue properties of austenitic stainless steels in specimens irradiated in a fast reactor neutron spectrum (moderate displacement levels but helium concentrations below 10 ppm) and in specimens containing cyclotron-injected helium (no displacement damage). The data obtained lead to contradictory conclusions, and the combined effects of displacement damage and high concentrations of helium (>100 at. ppm) have not been investigated. Since neutron irradiation reduces ductility,⁶ and since ductility is the most important index of resistance to low-cycle fatigue,⁷ irradiation probably reduces low-cycle fatigue life. However, irradiation hardening may result in slower progression of fatigue damage. The temperature-dependent competition between these two effects will determine the effect of irradiation on fatigue life.

Helium can also have opposing effects on fatigue behavior. Helium bubbles or clusters may result in lattice strengthening.^{8,9} An opposing effect results from sweeping helium bubbles into grain boundaries¹⁰

⁶E. E. Bloom, "Correlation of Structure and Ductility of Irradiated Austenitic Stainless Steel," pp. 93-102 in *Irradiation Embrittlement and Creep in Fuel Cladding and Core Components*, The British Nuclear Energy Society, London, 1973.

⁷R. E. Peterson "Fatigue of Metals: Engineering and Design Aspects," *Mater. Res. Stand.* 3: 122 (1963).

⁸C. F. Serpan, Jr., et al., "Microstructural Effects of Implanted Helium on Fatigue of 316 Stainless Steel," *Trans. Am. Nucl. Soc.* 16: 71-73 (1973).

⁹D. J. Michel, C. F. Serpan, Jr., and A. G. Pieper, "Effect of Helium on the Fatigue Behavior of the Molybdenum-Base Alloy TZM at 900°C," *Nucl. Technol.* 22: 79-87 (1974).

¹⁰D. S. Whalenell and R. S. Nelson, "The Thermal Release of Helium Injected into Nickel, Stainless Steels and Nimonic PE-16," *Radiat. Eff.* 14: 249-55 (1972).

and thus reducing ductility. The dominant mechanism at any given temperature remains to be determined.

1.1.4.2 Objectives of Present Research

This program has the following objectives:

1. determine the direction of the effect of irradiation on fatigue properties at various temperatures (A sufficiently high fluence and helium content will be employed to emphasize the effect and make an unambiguous determination of its direction.),
2. determine the mechanisms of helium effects in irradiated materials under cyclic stress (Before suggestions for improvement of fusion reactor materials can be made, the mechanism of failure must be understood.),
3. propose improvement of alloys for fusion reactor use.

1.1.4.3 Duty Cycle Simulation

The burn times for various types of fusion devices vary by several orders of magnitude. Tokamak designs indicate burn times ranging from about 100 to 5000 s.

During operation, heat is deposited in the first wall primarily by neutrons, alpha particles, and electromagnetic radiation. The heat is transferred to coolant in contact with the opposite side of the wall. Consequently, the plasma side of the wall will experience compressive stresses, the opposite side, tension. Upon shutdown, the entire wall will experience a tensile stress due to the coolant pressure.

As an example, the UWMAK-I Tokamak wall stresses are considered.¹¹ Rough calculations show these stresses to be the following:

	<u>Operation</u>	<u>Shutdown</u>
Plasma Side	-35 MPa (-5 ksi)	+35 MPa
Coolant Side	100 MPa (14 ksi)	+35 MPa

¹¹B. Badger et al., *UWMAK-I, A Wisconsin Torroidal Fusion Reactor Design*, UWFD-68, University of Wisconsin (November 1973).

A more recent calculation⁵ using finite element analysis and considering irradiation-produced swelling indicates that compressive stresses as high as 210 MPa may exist during transients.

The stress cycle chosen to test irradiated materials is fully reversed stress of a level higher than will be experienced in actual use. The first series of tests will use a standard fully reversed ramp function. This series of tests will be performed on approximately 40 specimens irradiated and tested at 300, 400, and 500°C, the same temperatures at which the other mechanical tests on irradiated fusion reactor materials will be conducted. Later tests will be conducted to determine the effect of hold time.

Because the effect of hold time in fatigue tests appears to saturate,¹² a test cycle considerably shorter than the actual duty cycle can be justified and allows testing in a reasonable time. A short series of tests may be conducted to determine if the effect of hold time does saturate in the materials considered. Such information may be useful in determining the mechanisms of helium interaction. Strain levels in the plastic region are proposed in consideration of the recent finite element analysis.

As fusion reactor designs become more firm and calculations of wall stresses become available, the test matrices will be modified. In addition, specimen geometry will be modified to more nearly reflect true wall loading. Hollow tube specimens designed to model the first-wall segments can be cyclically stressed by internal pressurization. Such specimens may also be miniaturized to permit *in-situ* testing in fission reactors.

1.1.4.4 Materials for Examination

Type 316 stainless steel will receive initial emphasis in the test program. Annealed and 20%-cold-worked titanium-modified stainless

¹²C. R. Brinkman, G. E. Korth, and R. R. Hobbins, "Estimates of Creep-Fatigue Interaction in Irradiated and Unirradiated Austenitic Stainless Steels," *Nucl. Technol.* 16: 297-307 (1972).

steel will be included in order to develop information on an alloy that has inherently higher ductility. Other alloys will be added as guidance is received from the Alloy Development for Irradiation Performance Task Group.

1.1.4.5 Irradiation

The High Flux Isotope Reactor (HFIR) will be used for initial irradiations in this program. Exposures will attain damage and helium levels of 10 dpa and 500 at. ppm. In subsequent experiments to examine effects of microstructure the Oak Ridge Research Reactor (ORR) will be used to allow large numbers of samples to be irradiated in a controlled environment with actively instrumented temperature control. Similar helium and displacement levels will be obtained.

1.1.4.6 Experimental Apparatus

A servo-hydraulic fatigue testing system will be used in a hot cell for the investigation, with the control and monitoring electronics outside the cell.

Since the atmosphere has a significant influence on fatigue properties at high temperatures,¹³ materials for use in the oxygen-free environment of a fusion reactor must be tested in a vacuum. A vacuum chamber having a large access door and capable of remote operation has been fabricated for use with the testing system.

This system will be the only one of its kind capable of testing irradiated materials in a vacuum or controlled atmosphere.

1.1.4.7 Status of the Project

A servo-hydraulic fatigue testing system has been procured and installed in the operating area outside the hot cell for preliminary testing. Special grips that allow easy remote operation and are compatible with a vacuum environment are now in use. A

¹³H. H. Smith, P. Shahinian, and M. R. Achter, "Fatigue Crack Growth Rates in Type 316 Stainless Steel at Elevated Temperature as a Function of Oxygen Pressure," *Trans. Metall. Soc. AIME* 245: 947-53 (1969).

diametral extensometer similar to that used by Slot et al.¹⁴ has been installed on the load frame. The extensometer was designed to permit remote installation on the specimen. Further testing of this capability will be necessary when the unit is installed in the hot cell. An induction heater is to be used for elevated-temperature testing. It is now in use for out-of-cell testing.

A vacuum chamber has been designed for use with the special grips and for remote operation with manipulators. It has been constructed by a commercial vacuum products firm and is now ready for installation. The chamber is to be pumped by a turbomolecular pump capable of pressures below 10 μ Pa.

The hot cell selected has a very low background radiation level, allowing easy entry for equipment service.

1.2 IRRADIATION EFFECTS IN NICKEL-BASE ALLOYS — F. W. Wiffen

Among the large number of alloys that have been considered as candidate structural materials for fusion reactor first wall and other components in the high-flux regions are a number of nickel-base alloys. This interest in nickel alloys was focused by the Princeton Plasma Physics Laboratory conceptual reactor study,¹⁵ which specified PE-16 as the structural material. In this design the PE-16 would operate in the temperature range 250 to 660°C, under neutron fluxes producing 27 to 44 dpa per year and 390 to 650 at. ppm He per year. That alloy was specified because of its resistance to swelling, relative to austenitic stainless steels, under conditions simulating LMFBR irradiation.

Swelling and mechanical properties after irradiation that closely simulates fusion reactor conditions can be quite different from the property changes that occur under fission reactor irradiation,

¹⁴T. Slot, R. H. Stentz, and J. T. Berling, "Controlled-Strain Testing Procedures," p. 100 in *Manual on Low Cycle Fatigue Testing*, ASTM Spec. Tech. Publ. 465, American Society for Testing and Materials, Philadelphia, 1969.

¹⁵R. G. Mills, ed., *A Fusion Power Plant*, MATT-1050, Princeton University (August 1974).

because of the greater helium production rates. A suitable simulation of fusion conditions is achieved in HFIR irradiation of nickel-containing alloys. The mixed neutron energy spectrum of HFIR results in displacement rates equivalent to about 3 MW/m^2 (due to HFIR's fast neutrons) and very high helium production rates due to the two-step capture reaction sequence of nickel with thermal neutrons. Two classes of nickel-base alloys are being evaluated under conditions of high helium production rates accompanying the production of displacement damage. The alloys are PE-16 (typical of the precipitation-strengthened nickel alloys) and Inconel 600 (typical of the solid-solution-strengthened nickel alloys). Both these alloys are reported to resist swelling on the basis of limited ion bombardment results. The goals of these experiments are: (1) investigate swelling under conditions of concurrent displacement damage and transmutation helium production, and (2) examine tensile properties of these irradiated materials as a function of irradiation and test temperatures.

1.2.1 Experiment

Rod tensile samples of PE-16 and Inconel 600 were irradiated in three identical experiments in the peripheral target positions of the HFIR flux trap. Design irradiation temperatures ranged from 300 to 700°C and the irradiation-produced helium contents in the range of 300 to 2000 at. ppm and displacement levels from 4 to 9 dpa.

Postirradiation examination of swelling involved micrometer measurements of length increases and changes in sample density. Density determinations were made by the immersion technique, using water with Photoflow added to reduce surface tension.

Tensile tests at various temperatures were run at a strain rate of 0.0028/min ($4.6 \times 10^{-5}/\text{s}$).

1.2.2 Swelling

Swelling in Inconel 600 ranged from 0 to almost 4% over the range of irradiation conditions examined. The results are shown as a

function of irradiation temperature in Fig. 1.8. Inspection of these data shows the following:

Fig. 1.8. Swelling in Inconel 600.

1. Swelling values are very high for the dpa levels achieved.
2. Swelling in Inconel 600 increases as the helium content increases, for a fixed irradiation temperatures.
3. Cold working Inconel 600 is not very effective in suppressing swelling during irradiation in the presence of high helium production rates.
4. Swelling under these irradiation conditions is weakly temperature dependent for irradiations between 300 and 600°C but increases markedly for irradiations at 650 and 700°C.

The swelling of PE-16 irradiated over the same range of irradiation temperatures fell within $\pm 0.5\%$ of zero. Helium levels generated in the PE-16 were lower than in the Inconel 600, because of the lower nickel content of the alloy. The densification observed in most of the irradiated samples of PE-16 is relative to the density of control

samples solution annealed 2 hr at 1120°C and then aged 16 hr at 700°C. It is suspected that the densification results from changes in the precipitate content of the samples during irradiation. Examination of the PE-16 swelling data suggests the following tentative conclusions:

1. Swelling in irradiated PE-16 is nearly identical for solution-annealed or annealed-plus-aged material.
2. The alloy is less affected by helium levels near 1000 at. ppm than is Inconel 600 for the same irradiation temperatures.
3. When irradiation-produced densification is taken into account, it appears that the swelling of PE-16 increases with increasing temperature through the range 300 to 700°C.
4. For the conditions examined, the swelling of PE-16 is significant only for irradiation temperatures above 600°C.

1.2.3 Tensile Properties

The tensile properties of the 20% cold-worked Inconel 600 are shown as a function of test temperature in Fig. 1.9. The properties of Inconel 600 irradiated in the solution annealed condition were very similar. The tensile properties of irradiated PE-16, irradiated in both the solution-annealed and solution-annealed-plus-aged conditions, are shown in Fig. 1.10. Control values for PE-16 are from handbook data.

These tensile property measurements lead to the following conclusions:

1. Strength values are increased for irradiation at 300 to 400°C.
2. Strength values decreased below unirradiated values for irradiations at 600 to 700°C.
3. Elongation values are lowest at the temperature extremes, with values at 400 and 500°C greater than at 300 or 700°C.
4. Helium clearly dominates the fracture process at 700°C.

1.2.4 Direction of Future Work

On the basis of these results a decision has been made to focus future efforts on the PE-16 alloy. New irradiation experiments will

Fig. 1.9. Tensile Properties of Irradiated 20%-Cold-Worked Inconel 600. 10^3 psi = 6.895 MPa.

Fig. 1.10. Tensile Properties of Irradiated Nimonic PE-16. 10^3 psi = 6.895 MPa.

expand the matrix of irradiation conditions to higher and lower fluences. In addition to immersion density and tensile tests, examination by electron microscopy, fractography, and metallography will be used to identify irradiation effects mechanisms. When all results are available, they will be evaluated in the framework of fusion reactor conceptual designs that specify nickel-base alloys as structural materials. Limitations imposed on reactor design by property changes will be considered, and future experiments necessary to evaluate the potential of these alloys in fusion reactors will be designed.

1.3 NEUTRON-IRRADIATION EFFECTS IN MOLYBDENUM AND MOLYBDENUM ALLOYS¹⁶ — J. Bentley and F. W. Wiffen

The high strength of molybdenum alloys at elevated temperatures makes these alloys attractive candidates for structures in advanced fusion power systems. This paper reports on microstructural examination following neutron irradiation at temperatures in the range of interest for fusion application.

1.3.1 Experimental

Sheet specimens of molybdenum, Mo-0.5% Ti, and TZM alloy (Mo-0.5% Ti-0.08% Zr) were irradiated in the X-034 structural materials experiment in row 2 of EBR-II at temperatures of 425, 585, and 790°C to a fluence of 2.5×10^{26} n/m² (>0.1 MeV) and at approximately 1000°C to a fluence of 4.4×10^{26} n/m². The measured¹⁷ helium content of one of the irradiated (2.5×10^{26} n/m²) molybdenum specimens was 0.4 at. ppm. Following electropolishing the specimens were examined by TEM.

1.3.2 Results

The now familiar behavior of decreasing dislocation density with increasing irradiation temperature is followed. The dislocation

¹⁶ Full paper published in *The Technology of Controlled Nuclear Fusion*, CONF-760935-P1, pp. 209-18 (1976).

¹⁷ Measured by H. Farrar IV, Atomics International.

densities in the alloys are higher than in the unalloyed molybdenum. At irradiation temperatures of 790 and 1000°C, the dislocations generally take the form of coarse networks, but some large interstitial loops are also present. Following irradiation at 585°C, the structure is on a considerably finer scale, with high concentrations of "black spots," (assumed to be small dislocation loops) and "rafts" of black spots, as well as dislocation segments. Concentrations of small dislocation loops were higher than the average for the sample near the dislocation segments, particularly in the molybdenum. At the lowest irradiation temperature, 425°C, the dislocation structure consists almost entirely of high concentrations of small dislocation loops either randomly dispersed or in linear arrays of three to ten loops. These clustered loops all possess a common Burgers vector, as do all the loops constituting a "raft," but these clusters are linear arrays, not planar arrays as in the case of rafts.

In TZM irradiated at 585°C, the concentration of small dislocation loops is much higher than in the molybdenum or Mo-0.5% Ti, and careful imaging near low-index poles revealed that the loops were ordered on a very imperfect bcc superlattice with a lattice parameter of approximately 30 nm.

High concentrations of small voids were observed in all specimens. Figure 1.11 illustrates the qualitative behavior of the distributions. With the exception of the lowest irradiation temperature (425°C), the void concentrations decrease with increasing irradiation temperature and are larger in the alloys than in the unalloyed molybdenum. The mean void diameter also increases with irradiation temperature and is slightly larger in the molybdenum than in the alloys. These trends are confirmed by the quantitative data in Table 1.2. This table also shows the void swelling calculated from the void size distributions and reveals that at irradiation temperatures of 425 and 585°C the swelling is approximately the same in all materials, but at irradiation temperatures of 790 and 1000°C the swelling is significantly higher in the alloy than in the molybdenum.

The specimens at 1000°C were irradiated to a higher fluence. Values of swelling normalized to 2.5×10^{26} n/m² are also shown in

Fig. 1.11. Electron Micrographs Showing Void Structures in Irradiated Mo, Mo-0.5% Ti, and TZM. Diffracting conditions: $z = [023]$, $g = 600$, $w_g > 0$, overfocus $\approx 1 \mu\text{m}$. Foil thicknesses = 100 nm.

Table 1.2. Quantitative Void Substructure Data for Molybdenum-Base Alloys Irradiated in EBR-II

Material	Irradiation Temperature		Fluence, >0.1 MeV (n/m ²)	Void Concentration (m ⁻³)	Mean Void Diameter ^a (nm)	Void Swelling (%)	Void-Lattice Parameter (nm)
	(°C)	(K)					
Mo	425	698	2.5×10^{26}	6.3×10^{22}	2.4	0.05	b
Mo	585	858	2.5	8.2×10^{22}	5.1	0.57	26
Mo	790	1063	2.5	4.5×10^{22}	5.7	0.45	34
Mo	1000	1273	4.4	5.0×10^{21}	13.1	0.59 (0.39) ^c	59
Mo-0.5% Ti	425	698	2.5	1.1×10^{23}	2.1	0.05	b
Mo-0.5% Ti	585	858	2.5	1.6×10^{23}	4.0	0.54	23
Mo-0.5% Ti	790	1063	2.5	6.5×10^{22}	6.1	0.79	35
Mo-0.5% Ti	1000	1273	4.4	1.7×10^{22}	10.2	0.33 (0.61) ^c	52
TZM	425	698	2.5	1.0×10^{23}	2.0	0.04	b
TZM	585	858	2.5	2.0×10^{23}	3.7	0.53	22
TZM	790	1063	2.5	8.0×10^{22}	5.7	0.78	32
TZM	1000	1273	4.4	1.6×10^{22}	10.9	1.1 (0.72) ^c	53

^aDiameter of void of mean volume.

^bVoid not ordered.

^cNormalized to a fluence of 2.5×10^{26} n/m² assuming swelling proportional to fluence to the power 0.75. Reference Bullough and Perrin.¹⁹

Table 1.2. This normalization suggests that peak swelling occurs at irradiation temperatures near 585°C in molybdenum and near 790°C in the alloys.

At irradiation temperatures from 585 to 1000°C the voids are ordered on an imperfect bcc superlattice. The void lattice parameter was calculated both from {110} d -spacings measured on micrographs taken at low-index poles and also from small-angle electron diffraction patterns using the method of Sass and Eyre.¹⁸ The quantitative results are shown in Table 1.2; the void-lattice parameter increases with increasing irradiation temperature, but there is little effect of material purity.

1.3.3 Discussion and Conclusions

Alloying molybdenum generally produced only quantitative differences in the damage microstructures, with the type of damage structure being qualitatively similar in all three materials. This observation is in contrast to many other alloy systems, where alloying profoundly affects the structure. In the present work the main quantitative difference is higher concentrations of defect clusters in the alloys. For the case of voids this results at 790 and 1000°C in increased swelling in the alloys; an unusual effect since generally pure metals show much greater swelling than alloys based on the same element. The observations have shown that there is a large imbalance in sink strengths, with the voids dominating. Under these conditions,¹⁹ any increase in dislocation density, such as occurs in the alloys, will result in more interstitials being absorbed at the biased sinks with less recombination at neutral sinks (voids) and a consequent increase in swelling. The higher dislocation

¹⁸S. L. Sass and B. L. Eyre, "Diffraction from void and bubble arrays in irradiated molybdenum," *Phil. Mag.* 27: 1447-1453 (1973).

¹⁹R. Bullough and R. C. Perrin, "Theory of Void Formation and Growth in Irradiated Materials," pp. 769-97 in *Radiation-Induced Voids in Metals*, J. W. Corbett and L. C. Ianniello, eds., AEC Symp. Ser. 26, CONF-710601 (April 1972).

densities in the alloys may be due to both increased nucleation of interstitial loops and to less coalescence of loops due to a higher loop glide stress.

The suppression of void formation in TZM observed²⁰ at lower fluences ($\sim 10^{24}$ n/m²) was not observed at the present higher fluences ($> 10^{26}$ n/m²) more typical of projected fusion reactor operation. Results and discussion on this important effect were reported previously.²¹

From the present work and other results on irradiated molybdenum,²²⁻²⁶ a general picture emerges of the development of the damage structure. In molybdenum, it appears that void nucleation is easy and void concentrations saturate at low damage levels.²³ Voids grow rapidly when the dislocation density is still high and the dislocations have the form of loops, but because the loops are

²⁰J. Bentley, B. L. Eyre, and M. H. Loretto, "Suppression of Void Formation in Neutron Irradiated TZM," pp. 1-297-311 in *Radiation Effects and Tritium Technology for Fusion Reactors*, J. S. Watson and F. W. Wiffen, eds., CONF-750989 (March 1976).

²¹J. Bentley and F. W. Wiffen, "Neutron-Irradiation Effects in Molybdenum and Molybdenum Alloys," *Magnetic Fusion Energy Materials Technology Program Annu. Prog. Rep. June 30, 1976*, ORNL-5189, pp. 11-13.

²²F. W. Wiffen, "The Effect of Alloying and Purity on the Formation and Ordering of Voids in BCC Metals," pp. 385-96 in *Radiation-Induced Voids in Metals*, W. Corbett and L. C. Iannelli, eds., AEC Symp. Ser. 26, CONF-720601 (April 1972).

²³J. Bentley, B. L. Eyre, and M. H. Loretto, "High Temperature Neutron Irradiation Damage in Molybdenum," pp. 923-31 in *Fundamental Aspects of Radiation Damage in Metals*, Vol. II, M. T. Robinson and F. W. Young, Jr., eds., CONF-751006-P2 (1976).

²⁴J. L. Brimhall, E. P. Simonen, and H. E. Kissinger, "Fluence and Temperature Dependence of Swelling in Irradiated Molybdenum," *J. Nucl. Mater.* 48: 339-50 (1973).

²⁵V. K. Sikka and J. Moteff, "Damage in Neutron Irradiated Molybdenum (I) Characterization of As-Irradiated Microstructure," *J. Nucl. Mater.* 54: 325-45 (1974).

²⁶B. L. Eyre, "A Review of the Contribution Made by Microscopy to the Study of Point Defects in BCC Metals," pp. 311-50 in *Defects in Refractory Metals*, (Proc. Int. Discussion Meeting, Mol, September 1971), R. de Batist, J. Nibbel, and L. Stals, eds., S.C.K./C.E.N., Mol, Belgium, 1972.

glissile they rapidly coalesce by a glide and self-climb mechanism, and the dislocation density is reduced. This results in a sink imbalance with voids as the dominant sink; thus, the void growth rate decreases markedly.¹⁹ With increased damage level, the voids order on a superlattice and this, as well as the sink imbalance, may limit the swelling rate.

The helium/dpa ratio in the present irradiations is much lower than that in a fusion reactor first-wall environment. It is considered that these expected higher helium generation rates will be less important in molybdenum alloys than in some other materials; first because void nucleation is already easy in molybdenum alloys, and second because even at modest fluences the high concentration of voids provides an adequate sink for the helium produced. Fission reactor irradiations probably therefore provide a reasonable simulation of fusion reactor irradiation response of molybdenum-base alloys.

1.4 A REVIEW OF "RECENT PROGRESS IN CTR BULK RADIATION EFFECTS STUDIES"²⁷ - F. W. Wiffen and J. O. Stiegler

One of the major reservations raised regarding the application of fission reactor irradiation data to the prediction of fusion reactor operation is the difference in the primary recoil spectra. The effect of this on some important physical and mechanical properties can be examined for low fluences attainable with existing *d-t* and *d-Be* neutron sources. Measurable results require pure metals, ambient-temperature irradiations, and maximum fluences practical with these sources.

We have compared the reported results of low-fluences, ambient-temperature irradiations conducted with 14-MeV neutrons and with fission reactor neutrons. These experiments are based on changes in several different physical properties in a number of different materials. Microstructural comparisons, which yield a qualitative

²⁷Extracted from pp. 135-47 in the *Proceedings of the Second Topical Meeting on the Technology of Controlled Nuclear Fusion*, CONF-760935-P1 (1976).

evaluation of the damage effectiveness of the bombarding species, are summarized in Table 1.3. These results are from low-fluence

Table 1.3. Qualitative Evaluation of the Damage Effectiveness of 14 MeV Neutrons by Transmission Electron Microscopy Observation

Experimenter ^a and Reference	Material	Irradiations Compared ^b	Comparative Observations ^c
Merkle 1-7	Au, Ag	14 MeV and Self-Ions	Observe expected subcascades. Most damage produced by inelastic scattering.
Mitchell et al. 8	Cu	14 MeV and Fission Reactor	Nature of damage essentially identical for both irradiations. Significantly more damage from high-energy neutrons than predicted.
Mitchell et al. 9, 10	Nb, Cu	14 MeV and Fission Reactor	Nature of damage identical. Significant difference in size and distribution of clusters, with 14 MeV producing the more complex damage state.
Roberto et al. 11, 12	Nb, Cu	~15 MeV and Fission Spectrum	Detailed agreement in cluster size, distribution, and total defect content. Quantitative agreement between predicted and observed amount of retained damage.
Narayan and Ohr 13	Nb	~15 MeV and Fission Spectrum	Loops 2/3 interstitial, 1/3 vacancy in both cases. Multiple clusters ~2% at high energy. Detailed nature of damage identical for the two irradiations.
Brimhall et al. 14	Mo	14 MeV and Fission Reactor	More retained damage from 14-MeV neutron irradiation than predicted. No unique damage structures.

^aReferences given in Table 1.5

^bAll for irradiation at the neutron source coolant temperature (20-65°C).

^cNo account is taken of the usually higher irradiation temperature during the fission reactor irradiation.

^dSamples examined after plastic deformation.

irradiations conducted at room temperature. For the pure metals used the damage is in the form of small dislocation loops, and comparisons are made on the basis of their size, concentration, and spatial distribution.

Comparisons of damage produced by irradiations in various neutron spectra have been made quantitative for measurements of a number of properties in several different materials. The results of these quantitative comparisons of the effect of neutron energy are collected in Table 1.4, and the picture that has developed from this review can be summarized as follows:

Table 1.4. Quantitative Comparison of Property Changes Produced by Fission-Energy and Fusion-Energy Neutron Irradiation

Experimenter and Reference (Table 1.5)	Material	Property Measured	Property Sensitivity	Reference Irradiation ^d Fission Reactor	Reference Irradiation ^d Maximum Reference Fluence n/m ² (>0.1 MeV)		High-Energy Irradiation ^b High Energy Neutron Source		Ratio: Response per HFE Neutron Response per Ref. Neutron		Ratio: Experiment Calculation
									Calculated	Experiment	
Chen, 15	MgO	Optical absorption	Anion vacancies	ORR	1×10^{23}		RTNS ^c	1.2×10^{23}	1.94	2	1.03
Bunch, 16	Al ₂ O ₃	Optical absorption	Point defects	Fission ^d	5×10^{23}		RTNS	10^{23}	1	4	1
Snead, 17	Nb ₃ Sn	SC transition temperature	Changes long-range order	HFBR	1.5×10^{23}		RTNS	1.8×10^{23}	1.0	$4.0^{+0.5}_{-0.7}$	1.3
Borg, 18	Au-Fe	Mössbauer Magnetic Ordering	Mobile defects	LPTR	9×10^{22}		RTNS	2×10^{23}	5.7	40	1.4
Damask, 19	Cu-Zn	Resistivity, short-range order	Free vacancies	LPTR	1.5×10^{23}		RTNS	8×10^{22}	8	10 ± 2	1
Merkle, 1, 6	Au	TEM	Defect clusters	CP-5	6×10^{23}		RTNS	2×10^{23}	5.0	6.0	1.2
Mitchell, 10	Nb	Yield stress	Defect clusters	LPTR	10^{23}		RTNS	10^{23}	10.6	17	1.6
Mitchell, 10	Cu	Yield stress	Defect clusters	LPTR	10^{23}		RTNS	4×10^{22}	8.7	20	2.3
Roberto, 11, 12	Nb	TEM	Defect clusters	BSR	5×10^{23}		d-Be ^e	2×10^{23}	3.1	2.5	0.81
Roberto, 11, 12	Cu	TEM	Defect clusters	BSR	1×10^{22}		d-Be	2×10^{23}	1.4	2.0	1.4
		x ray	Defect clusters	BSR	1×10^{22}		d-Be	2×10^{23}	1.5	3.3	0.46
Brimhall, 14	Mo	Lattice parameter	Interstitials	Hanford	1×10^{23}		RTNS	1.6×10^{23}	5	22	4.4
		Resistivity	All defects	Hanford	1×10^{23}		RTNS	1.6×10^{23}	5	5	1.0

^aAll irradiations at reactor ambient temperature, variously quoted from 40 to 65°C.

^bAll irradiations at room temperature, quoted values 23 to 27°C.

^cRTNS: 10¹² neutron source at Lawrence Livermore Laboratory.

^dGraphite reactor at Brookhaven National Laboratory.

^eTotal neutrons.

^fResult stated to be "in rough agreement with Robinson calculations of damage effectiveness."

^g1.0 MeV.

^hNot given.

ⁱ0.5 eV.

^jNeutrons from breakup of 40-MeV F^+ on Be target.

Table 1.5. References for Tables 1.3 and 1.4

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2. K. Merkle, L. R. Singer, and R. K. Hart, *J. Appl. Phys.* 34: 2800 (1963).
3. K. L. Merkle, *Phys. Status Solidi* 18: 1973 (1966).
4. K. L. Merkle, "Displacement Cascades in Gold by Means of Transmission Electron Microscopy," pp. 159-70 in *Radiation Damage in Reactor Materials*, Vol. 1, International Atomic Energy Agency, Vienna, 1969.
5. K. L. Merkle, L. R. Singer, and J. R. Wrcbel, *Appl. Phys. Letters* 17: 6 (1970).
6. K. L. Merkle, *Nucl. Technol.* 22: 66 (1974).
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8. J. B. Mitchell, C. N. Logan, and C. J. Echer, *J. Nucl. Mater.* 48: 139 (1973).
9. J. B. Mitchell et al., *Phil. Mag.* 31: 919 (1975).
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11. J. Roberto, J. Narayan, and M. J. Saltmarsh, "15 MeV Neutron Damage in Cu and Nb," pp. II-159-71 in *Radiation Effects and Tritium Technology for Fusion Reactors*, CONF-750989 (1976).
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14. J. L. Brinhall, L. A. Charlot, and H. E. Kissinger, *Radiation Effects*, 28: 115 (1976).
15. Y. Chen et al., "Production of Point Defects in 14.8 MeV Neutron-Irradiated MgO," pp. II-492-97 in *Radiation Effects and Tritium Technology for Fusion Reactors*, CONF-750989 (1976).
16. J. M. Bunch and F. W. Clinard, Jr., *J. Am. Ceram. Soc.* 57: 279 (1974).
17. C. L. Snead, Jr., et al., "Determination of the Damage-Energy Cross Section of 14-MeV Neutrons from Critical-Property Changes in Irradiated Nb₃Sn," pp. 229-37 in *Proceedings of the Second Topical Meeting on the Technology of Controlled Nuclear Fusion*, CONF-760935-P1, (1976).
18. R. J. Borg and G. J. Dienes, *J. Appl. Phys.* 46: 99 (1975).
19. A. C. Damask et al. *Radiation Effects* (in press).

1. The qualitative response to 14-MeV neutron irradiation is in agreement with predictions based on experience with irradiation in other facilities.

2. Comparisons of the response of a number of material properties to irradiation show that any given property varies similarly with fluence in different neutron spectra. The effect of the neutron spectrum on the variation is a translation on the fluence axis.

3. The quantitative response of a property change to 14-MeV neutron irradiation depends on the sensitivity of that property to various defect configurations. Properties dependent on the total number of defects scale directly with damage energy. Properties that depend on the type and distribution of clustered defects require more complex analysis of the damage distribution.

4. The scaling factor between irradiations in different facilities may depend on the property being measured. Different scale factors may have to be developed for each property of concern.

5. Nothing unexpected has yet been observed.

6. The physics of damage production is proving adequate.

7. Fluences at which transmutation effects become important have not yet been reached by 14-MeV neutron irradiations.

8. Nascent defect distributions within the damage cascade may become unimportant at projected reactor temperatures and fluences. These distribution differences will likely be of far less importance than variations due to kinetic differences between various facilities and differences due to transmutation products accumulated at the high fluences.

Fission reactor neutron irradiation effects have been compared with 14-MeV neutron-irradiation effects in experiments covering a wide range of materials and property measurements. These comparisons have so far been restricted to irradiations at or near room temperature and at relatively low fluences at which neither damage overlap events nor accumulated transmutation products would be expected to contribute to the damage state. The result of these comparisons is to show that property changes can be related quantitatively. The relationship

is in terms of calculated damage energies and our understanding of the dependence of the specific property change on the types of defects and defect clusters produced by the irradiation. This success in correlating damage produced in the different neutron spectra is in our judgment excellent. It suggests that fission reactor neutron irradiation provides a very useful and accurate simulation of the displacement damage that will be encountered in fusion reactor irradiations. It must be emphasized that in the general case the fission reactor irradiations will simulate only the displacement damage and that special attention must continue to be paid to the accompanying transmutation products, especially the helium that results from (n,α) reactions.

1.5 THE PROPERTIES OF SAP IRRADIATED TO PRODUCE HIGH GAS CONTENTS — P. J. Maziasz and K. Farrell

Sintered aluminum product (SAP) has been proposed as a material for use as the first wall of a fusion reactor. To examine the feasibility of this proposal, we are determining the effects of gas contents expected during fusion reactor operation on the mechanical properties of SAP. HFIR irradiation of SAP doped with ^6Li has been chosen to simulate fusion reactor irradiation. Displacement damage is produced by the fast-neutron component of the HFIR spectrum, and gas production (both tritium and helium) results from the thermal neutron reaction: $^6\text{Li} + n \rightarrow ^4\text{He} + ^3\text{H}$. After irradiation specimens are to be annealed at 400°C to redistribute the gases and are then to be tensile or creep tested at temperatures in the range 20 to 500°C .

Tensile specimens of SAP with from 0 to 174 wt ppm Li (96% ^6Li) and 17 to 20 wt % Al_2O_3 have been irradiated in HFIR at ambient reactor coolant temperatures ($\sim 55^\circ\text{C}$) to neutron fluences that caused greater than 94% burnup of the ^6Li . Unirradiated tensile tests have been run at test temperatures of 20 to 500°C and previously reported.²⁸

²⁸P. J. Maziasz and K. Farrell, "HFIR Irradiations of SAP to Produce High Helium Contents," *Controlled Thermonuclear Materials Technology Annu. Prog. Rep. June 30, 1975*, ORNL-5082, pp. 38-39.

The total elongation was lower at all temperatures tested than results for SAP 895 (11.5 wt % Al_2O_3) reported by others,²⁹ and strength properties were greater. This is believed to result from the higher oxide content.

Immersion density measurements of the irradiated samples indicated little or no swelling either before or after postirradiation annealing at 400°C. Tensile tests of as-irradiated material at room temperature and 400°C indicate little change of properties due to the irradiation, but the ductility is low, about 0.3% elongation. Both irradiated and unirradiated specimens show a reduction in ductility after annealing for 1000 hr at 400°C. The reduction is slight for helium contents up to 260 at. ppm, but greater for about 1600 at. ppm He. A test matrix of annealing and testing at temperatures of 300, 400, and 500°C is under way to confirm and expand the above results. In addition, the temperature dependence of the properties after annealing at 400°C for 1000 hr will be determined.

1.6 IRRADIATION FACILITIES AND EXPERIMENTS

1.6.1 Use of Fission Reactors for Fusion Reactor First Wall Studies³⁰ — James A. Horak and Jerry H. Swanks³¹

The purpose of the work presented herein is to demonstrate one of the more important uses of existing fission reactors for the magnetic fusion reactor, MFR, first-wall alloy development program. The most important properties of the first wall are resistance to fracture and to swelling while operating in the MFR environment. One of the more important effects of the fusion reactor environment is the high ratio of He/dpa produced in the first wall. The first wall

²⁹G. L. Copeland, *An investigation of Alloying Effects in Aluminum Dispersion Strengthened with Al_2O_3* , ORNL-5088 (October 1975).

³⁰Summary of paper presented at ANS Special Session 6.9, "Simulation of Radiation Damage Effects: Theory and Practice," ANS 1977 Annual Meeting, June 12-17, 1977 New York.

³¹Operations Division

must operate in the temperature range from ≈ 0.3 to $\approx 0.5T_m$, where ductility and swelling are sensitive to He concentration, dpa, and the He/dpa.³²⁻³⁴

In mixed-spectrum fission reactors where both the fast (>0.1 MeV) and thermal neutron fluxes exceed 3×10^{18} n/m² s both He and dpa levels equal to or greater than those anticipated for an MFR at an operating level of at least 1 MW/m² can be produced in alloys that contain nickel, as shown in Fig. 1.12 for type 316 stainless steel. The fast neutrons produce the dpa and the helium is produced^{35,36} by the process

However, as shown in Fig. 1.12(a), irradiation in a mixed-spectrum reactor where the thermal-to-fast neutron ratio is approximately constant during the entire irradiation, the same He/dpa as that in an MFR first wall of type 316 stainless steel is achieved at only one time during irradiation. The helium concentration increases

³²F. W. Wiffen and E. E. Bloom, "Effect of High Helium Content on Stainless Steel Swelling," *Nucl. Technol.* 25: 113-23 (1975).

³³E. E. Bloom and F. W. Wiffen, "The Effects of Large Concentrations of Helium on the Mechanical Properties of Neutron-Irradiated Stainless Steel," *J. Nucl. Mater.* 50: 171-84 (1975).

³⁴P. J. Maziasz, F. W. Wiffen, and E. E. Bloom, "Swelling and Microstructural Changes in Type 316 Stainless Steel Irradiated under Simulated CTR Conditions," pp. I-259-88 in *Radiation Effects and Tritium Technology in Fusion Reactors*, CONF-750985 (March 1976).

³⁵J. A. Horak, "Use of Fission Reactors for CTR Bulk Radiation Effects Studies," pp. 830-41 in *Proc. Int. Conf. Radiation Test Facilities for the CTR Surface and Materials Program*, ANL/CTR-75-4 (1976).

³⁶J. A. Horak, J. O. Stiegler, and F. W. Wiffen, "Evaluation of Fission Reactors for CTR Materials Development," *Trans. Am. Nucl. Soc.* 22: 172-74 (1975).

Fig. 1.12. Helium Concentration as a Function of Displacements Per Atom in Type 316 Stainless Steel for an MFR First Wall and for Irradiation in: (a) Two HFIR and Two ORR Positions (b) Two Positions of ORR with Spectrum Tailoring. Percentage values are for the increases in the thermal neutron flux produced by varying the moderation between the reactor fuel and the test samples.

approximately as $(\phi_t t)^{1.5}$ and the dpa increases linearly with $\phi_t t$. Hence, the time when the He/dpa in a fission reactor exceeds that in an MFR is a function of the ratio of thermal to fast neutron flux. To utilize fission reactors for MFR first-wall development of materials that contain nickel the He/dpa during the entire irradiation must closely match that anticipated in an MFR.

For several valid reasons it is becoming increasingly obvious that a member of the type 316 stainless steel family will be utilized for the first wall of the early generations of MFRs.³⁷⁻⁴¹ Figure 1.12(b) shows the calculated He/dpa in type 316 stainless steel for an MFR at 1 MW/m² and for irradiation in two of the high-flux positions (E-5 and C-3) of the Oak Ridge Research Reactor (ORR). The He/dpa for type 316 stainless steel irradiated in the central irradiation positions of ORR can be maintained as close to that of an MFR as is thought necessary. This is accomplished by varying the thermal to fast neutron ratio as often as desired during the irradiation.

The ORR core loading arrangement is designed to be extremely flexible. The spectrum tailoring required to produce the correct helium and displacement production rates and the He/dpa can be controlled through the number of fuel elements in the core, the mass of ²³⁵U per fuel element, the thickness and species of moderator between the fuel and the test samples, and the insertion of thin pieces of aluminum containing about 10 at. % ²³⁵U between the fuel and test

³⁷E. E. Bloom, F. W. Wiffen, P. J. Maziasz, and J. O. Stiegler, "Temperature and Fluence Limit for a Type 316 Stainless Steel Controlled Thermonuclear Reactor First Wall," *Nucl. Technol.* 31: 115-22 (1976).

³⁸M. Roberts et al., *Oak Ridge Tokamak Experimental Power Reactor Study - Reference Design*, ORNL-TM-5042 (November 1975).

³⁹W. M. Stacey, Jr., et al., *Tokamak Experimental Power Reactor Studies*, ANL/CTR-75-2 (July 1975).

⁴⁰General Atomic Co., *Experimental Power Reactor Conceptual Design Study 12-Month Prog. Rep. June 1, 1974-June 30, 1975*, GA-A-13534 (1975).

⁴¹M. Murphy and J. Neff, eds., *Summary of the Proceedings of the Workshop on Conceptual Design Studies of Experimental Power Reactor-1*, (Oak Ridge, Sept. 9-10, 1975), ERDA-89 (November 1975).

samples. In the example shown in Fig. 1.12(b), extremely simple spectrum tailoring of the *present* ORR was used. No attempt was made to optimize the spectrum tailoring to produce a He/dpa closer to the MFR value. How close the He/dpa must be controlled to simulate the effects of the MFR environment is not yet known. It would be relatively easy to have the He/dpa value for ORR irradiation of type 316 stainless steel to be as close to that of an MFR as desired.

In summary, high-flux mixed-spectrum fission reactors, such as the ORR, can provide relevant simulation of the helium concentration, dpa, and He/dpa expected in an MFR. These reactors will be an important part of the DMFE alloy development program for first-wall materials that contain nickel for at least the next 5 to 10 years.

1.6.2 The Unique Role of HFIR in Fusion Reactor Materials Evaluation^{41a} — P. J. Maziasz and F. W. Wiffen

The lack of irradiation facilities with neutron flux and spectrum that match a α - t -fueled fusion reactor require the use of simulation techniques in examining irradiation effects in fusion reactor first-wall and structural materials. Our purpose is to examine the place of irradiations in the HFIR in evaluating the effects of the fusion reactor environment and to examine the results of experiments exploiting the HFIR advantages.

The High Flux Isotopes Reactor (HFIR) is a 100-MW(th) flux-trap reactor. The highest fast neutron flux is 1.4×10^{19} n/m² s (>0.1 MeV); the thermal flux at the same position is 2.3×10^{19} n/m² s. Irradiation temperatures from 280 to 950°C have been achieved by gradients across gas gaps between the nuclear-heated specimen and the specimen holder in thermal contact with the water coolant.

Neutron irradiation of a metal results in the displacement of atoms, mainly through elastic collisions, and transmutation of target atoms initiated by neutron captures. In a fusion reactor, the accumulated displacement damage and the helium produced in (n, α) reactions will be the most important components of the irradiation

^{41a}Taken from *Trans. Am. Nucl. Soc.* 26(TANSO 26): 189-90 (June 1977).

response. In HFIR irradiation fast neutrons result in a high level of displacement damage, and the high thermal neutron flux results in significant yields for any reaction having a high thermal neutron cross section. A unique reaction sequence of interest here is helium production from the major isotope of nickel. The two-stage *thermal* neutron reaction sequence is

The calculated response during HFIR irradiation of two alloys of interest is given in Table 1.6 compared with calculated damage production rates in a Tokamak fusion reactor first wall.

Table 1.6. The Simulation of Fusion Reactor Irradiation Response by HFIR Irradiation of Nickel-Containing Alloys

Material	Displacement Damage		Helium Generation	
	Calculated Response (dpa)	Fusion Equivalent (MW years/m ²)	Calculated Response (at. ppm)	Fusion Equivalent (MW years/m ²)
<u>One HFIR Cycle (23 days)</u>				
Type 316 SS	2.25	0.2	30	0.2
PE-16	2.25	0.2	101	0.7
<u>One Year in HFIR (at 92%)</u>				
Type 316 SS	33.7	2.9	1920	13
PE-16	33.7	2.9	6370	44

Irradiations in the HFIR offer the only method of achieving helium levels equivalent to long service in a fusion reactor. Although these helium levels are accompanied by a lower dpa level than would be achieved in the long fusion reactor service, the dpa level is as high as achievable in any other fission reactor. These unique advantages of HFIR can be exploited in evaluating the importance of high helium levels in determining the irradiation

response of some metals, and in cases where helium level can be shown to control the irradiation response, the results can be used to predict fusion reactor service behavior.

Samples of type 316 stainless steel, Inconel 600, and PE-16 have been irradiated at temperatures in the range 300 to 750°C. Postirradiation examination has resulted in the following conclusions:

1. At levels of helium anticipated in fusion reactor service the irradiation response is not adequately predicted by irradiations that produce only displacement damage.^{32-34,37}

2. Swelling is controlled by helium content, with cavities in equilibrium with the helium pressure.^{32,34} As a result, swelling is relatively temperature independent throughout the "normal" range of swelling temperatures, but larger swelling is observed at higher temperatures.

3. Ductility reductions are greater than observed after fast-reactor irradiation. At elevated temperatures, embrittlement is clearly dominated by helium content, with near-zero elongation resulting from intergranular fractures.³³

Future HFIR irradiations will be directed at more completely defining the helium effects on irradiation response and comparing the response of candidate alloys to the unique regime of irradiation conditions achievable in this reactor. The results will be translated to performance limits on planned fusion reactors and will be used in the development of alloys more resistant to degradation in fusion reactor services.

1.6.3 The Rate of Helium Generation in HFIR Irradiation of Nickel⁴² -
 F. W. Wiffen, E. J. Allen,⁴³ E. E. Bloom, H. Farrar⁴⁴
 T. A. Gabriel,⁴⁵ H. T. Kerr⁴³ and F. G. Perey⁴⁵

The extensive use of irradiation experiments in the HFIR-PTP (peripheral target positions), and the helium production from ^{58}Ni in alloys containing nickel make the calculation of the helium production rate important to the program. We have analyzed the results of helium measurements in a number of specimens irradiated in the HFIR-PTP to arrive at an equation that will predict the helium production.

The primary source of helium in alloys containing nickel under irradiation in a reactor with a thermal neutron spectrum is the reaction sequence

Examination of the available cross sections for these reactions and for other reactions that compete for ^{58}Ni and ^{59}Ni showed that the data are best fit if the cross section for the reaction

is taken to be $(11.4 \pm 1.7) \times 10^{-28} \text{ m}^2$ ($11.4 \pm 1.7\text{b}$). This value falls within the lower portion of the range of previous measurements of this cross section. The resulting equation for the production of helium is

⁴²Summary of an analysis in progress. A more detailed report will be issued soon.

⁴³Engineering Technology Division.

⁴⁴Atomics International Division, Rockwell International.

⁴⁵Neutron Physics Division.

$$\frac{N(\text{He})}{N(\text{Ni-58})} = 0.12488 - 0.13126 \exp(-1.77\phi t) + 0.00638 \exp(-36.09\phi t)$$

where

$N(\text{He})$ = atom concentration of ^4He ,

$N(\text{Ni-58})$ = initial atom concentration of ^{58}Ni , and

ϕt = total fluence in units of n/b.

The equation is plotted with the experimental data in Fig. 1.13, which shows the adequacy of the fit. The equation now is used in the design of all HFIR irradiation experiments.

Fig. 1.13. Amount of Helium Produced in ^{58}Ni During Irradiation in HFIR. The experimental data are derived from measurements in various alloys containing nickel. The solid curve is the empirical prediction of helium production, derived from the shown experimental data.

1.6.4 HFIR Elevated-Temperature Irradiations — F. W. Wiffen, J. W. Woods, and P. J. Maziasz

Irradiation of tensile specimens in HFIR at temperatures well above reactor coolant temperatures forms a continuing part of this program. These tests are designed to exploit the unique ability of HFIR irradiations to produce high helium concentrations in nickel-containing alloys concurrent with the production of high levels of displacement damage. The irradiation temperatures selected bracket the probable fusion reactor application range for the alloys under investigation. A comparison of results from these experiments with results from breeder reactor irradiation experiments, where helium production levels are usually quite low, will be definitive in assessing the effect of helium produced during irradiation on the irradiation response.

Irradiation temperatures above reactor coolant temperature are achieved by use of a gas gap between the specimen and the specimen holder. This gap serves as a barrier to transfer of the nuclear heat generated in the sample to the reactor coolant. The width of the gap thus sets the temperature gradient and the sample irradiation temperature. A schematic view of a section through the experiment is shown in Fig. 1.14, illustrating the experiment elements. Tensile specimens from these experiments are evaluated after irradiation to determine swelling, mechanical properties, fracture mode, and microstructure.

A summary description of HFIR-PTP elevated-temperature tensile experiments built during the past year is given in Table 1.7. Materials and irradiation conditions are given, as well as goal irradiation parameters.

1.6.5 HFIR Irradiation of Fatigue Samples — M. L. Grossbeck, M. J. Kania, and J. W. Woods

Irradiation temperatures for the first two irradiation experiments containing fatigue specimens will be 300 and 500°C. Heat transfer calculations have been made with a two-dimensional heat transfer code, the ORNL HEATING-III code. Gas gaps to impede the radial flow of

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Fig. 1.14. Schematic Cross Section Through One of 11 Tensile Specimen Positions in an Elevated-Temperature HFIR-PTP Irradiation Experiment.

Table 1.7. Summary Description of Elevated-Temperature Tensile Specimen Experiments Loaded into HFIR in the Reporting Period

HFIR-CTR Experiment	Number of 23- Day Exposure Cycles	Alloys in Experiment	Temperature Range (°C)	Goal Displacement Range (dpa)	Goal He Range (at. ppm)
4	2	PE-16	300-700	2-4.5	100-350
7	8	PE-16	300-700	9-18	1200-3000
8	8	PE-16	300-700	9-18	1200-3000
9	8	316	280-620	9-18	430-1100
10	8	316 + Ti	280-670	9-18	430-1100
11	8	316 + Ti	280-670	9-18	430-1100
12	5	316 + Ti	280-670	5-11	200-530
13	5	316	280-620	5-11	200-530
17	6	316 Welds	280-620	6-14	180-460
18	15	PE-16	300-700	17-27	3300-5600
		316	280-620	27-34	1600-2100

gamma-radiation-produced heat were made to obtain the elevated temperatures of the specimens. A typical temperature distribution is shown in Fig. 1.15.

Fig. 1.15. Typical Specimen Temperature Distribution in HFIR Irradiation of Fatigue Samples.

The irradiation capsules are the same in exterior design as used for previous HFIR materials irradiations. Each capsule contains ten specimens, and the interior has been modified to accommodate the large fatigue specimens (Fig. 1.16). Figure 1.17 shows one specimen placed in its holder assembly. The aluminum holder surrounding the specimen is separated longitudinally into two halves to permit assembly.

Both SiC and melt wire alloys are used to indicate temperature. Small stainless steel capsules, 1.57 mm in diameter, have been placed in holes drilled in two specimens. The capsules have been made small in order to minimize perturbation of the temperature distribution. Pure metals and eutectics have been selected for use in the capsules. The low-melting-point materials were prepared in the form of a granular powder. The results of out-of-reactor tests show that melting can be discerned by radiography (Fig. 1.18). The metals and alloys were

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Fig. 1.16. Radiograph of HFIR Irradiation Capsule Containing Fatigue Specimens.

Fig. 1.17. Specimen Holder Assembly.

Fig. 1.18. Radiograph of Temperature-Indicator Capsule Containing Selenium. 15 $\times$. (a) Before melting. (b) After melting.

selected for their melting points as well as for their neutron absorption properties. Materials used by previous investigators have had high neutron absorption cross sections, resulting in rapid transmutation, which may have been why they failed as temperature indicators. In this irradiation, Se, Pb, Pb-5 wt. % Pt, and Zn-5 wt. % Al will be used. The alloys are commercially available for use as furnace melt wire fuses.

The first HFIR fatigue sample irradiation experiment designed for 300°C has been built for irradiation of ten 20%-cold-worked type 316 stainless steel specimens. It was inserted in the HFIR in June 1977. The temperature indicators will be carefully evaluated after irradiation to determine their future utility. The first specimens are expected to be available for testing after October 1977.

1.6.6 HFIR Ambient Temperature Irradiation — F. W. Wiffen and J. W. Woods

The interest in the use of lower temperature first-wall and structural components in fusion reactors has revealed a deficiency in available irradiation effects data. This deficiency is the lack of data for irradiation temperatures below about 350°C, and is a direct result of the needs of the breeder reactor programs. With recent conceptual designs of fusion reactors with separately cooled near-plasma zones temperatures as low as 100°C are of interest to the fusion power program.

We have begun new experiments to expand the range of irradiation temperatures downward to below 100°C. The first experiment is under irradiation in an HFIR-PTP position. It contains 42 specimens in direct contact with the about 55°C coolant water. The sample temperatures during irradiation has been calculated to be in the range 65 to 80°C.

Samples used in this experiment are of the same design as used in elevated-temperature HFIR irradiation experiments. They are 39.4-mm-long (1.55-in.) rod tensile samples with a gage section 18.3 mm (0.72 in.) long and 2.03 mm (0.080 in.) diam. The experiment is composed of three housing tubes in a single assembly. Each housing tube contains a liner tube that holds the 14 tensile samples in place. Reactor coolant water

flows through the housing and container tubes, in direct contact with the samples. The irradiation experiment is shown schematically in Fig. 1.19.

Fig. 1.19. Schematic Representation of HFIR Ambient-Temperature Irradiation Experiment. One tube of the three-tube cluster has been sectioned to show one of the 14 specimens in it. Diameters shown are 7.92, 5.72, 5.59, 4.78, 4.06 mm.

Specimens loaded into this experiment included type 316 stainless steel in several conditions, weld-containing samples of type 316, PE-16, and Inconel 600. Two capsules of materials selected for the measurement of helium generation rate during HFIR irradiation were also included in the experiment in a joint effort with H. Farrar of Atomic International. Irradiation for five HFIR cycles will result in displacement damage levels of 5 to 11 dpa and helium levels of 200 to 2800 at. ppm, depending on the alloy and specimen position in the experiment. Postirradiation evaluation will concentrate mainly on tensile tests, supplemented as necessary by density measurement, metallography, fractography, and microscopy.

1.6.7 The Planned Use of ORR for DMFE Irradiations - F. W. Wiffen, J. W. Woods

The DMFE-convened Ad Hoc Committee on Use of Fission Reactors in the DMFE Alloy Development Program has recommended that the ORR be used as a primary irradiation facility for tests involving helium generation and atomic displacements in nickel-bearing alloys. This recommendation was made to take advantage of the ORR features of:

1. ability to control helium-to-displacement ratio,
2. displacement rates greater than produced by a 1 MW/m² wall loading,
3. large experimental volume,
4. instrumentation for the control and monitoring of experiments, and
5. dedication of the reactor to the DMFE program.

We plan to initiate irradiation of two experiments in the ORR core early in FY 1978. These experiments will be based on currently proven methods and will use existing instrumentation and no spectral tailoring.

The experimental goals are to improve the data base on the path A alloys (austenitic alloys) and to begin scoping the irradiation response of Path B (Fe-Ni-Cr superalloys) and Path C (reactive and refractory metals and alloys) alloys of the Alloy Development for Irradiation Performance (ADIP) plan. This is to include a wide range of irradiation and materials conditions on the DMFE Reference Heat of type 316 stainless steel and limited tests on other alloys.

Irradiation for one year will produce a midplane irradiation response in type 316 stainless steel of approximately 11 dpa and 80 at. ppm He.

1.6.7.1 Reactor and Equipment

Experimental positions will be C7 and E7. These have fuel on four sides, are "flux trap" positions. Access tubes for instrumentation are under design and reactor fuel has been ordered. Probable irradiation start date is about December 1977.

Temperature instrumentation is being overhauled. Pressurization systems are being checked. We are examining the possibility of operating some tubes under cyclic pressurization, but it is not certain that this can be available in time. In any case, the experiment will contain

dynamically pressurized tubes. The C7 experimental position can accommodate up to 20 independent pressure lines; E7 can take up to ten independent lines.

1.6.7.2 Dosimetry and Neutronics

The DMFE-funded Dosimetry Center at ANL will cooperate on dosimetry for these experiments. The dosimetry package will be planned by ANL dosimetry people, the experimenters, and ORNL Engineering Technology Division and Operations Division personnel (the Kerr-Allen model). Neutronics calculations to aid in experiment planning are to be provided by existing programming in Neutron Physics Division.

1.6.7.3 Planned Specimen Types

A mixed loading of various specimen geometries will be used to maximize available space and to satisfy required postirradiation testing to determine the several properties of interest. Probable specimen types include:

1. pressurized tubes - 25 to 51-mm-long (1-2 in.) tubes, about 5 mm diam (0.200 in.).
2. fatigue specimens - specimens developed for HFIR irradiation. Gage diameter about 3 mm (0.125 in.), threaded hourglass specimen machined from 6.4-mm diam (1/4 in.) rod.
3. tensile and creep specimens
 - a. SS-1 sheet specimens:
 - length: 44.5 mm (1.75 in.)
 - gage section: 20.3 by 1.52 by 0.76 mm (0.80 by 0.060 by 0.030 in.)
 - b. HFIR sub-mini rod
 - length: 31.8 mm (1.25 in.)
 - gage section: 18.3 mm by 2.03 mm diam (0.72 by 0.080 in.)
 - c. EBR-II rod
 - length: 47.5 mm (1.87 in.)
 - gage section: 28.58 mm by 3.18 mm diam (1.125 by 0.125 in.)
4. Other geometries may still be included but have not yet been established. These may include crack growth and fatigue experiments.

1.6.7.4 Irradiation Parameters and Experiment Contents

Irradiation for 365 days at full power (based on C-3 position neutronics) will produce the following response in type 316 stainless steel:

	dpa	Helium (at. ppm)
Top of experiment	4	
Midplane	10.4	
Peak flux position	11.0	80
Bottom of experiment	7.9	

This response derives from the best currently available neutron flux information.

Irradiation temperatures will span the range 250 to 650°C. Limits imposed by nuclear heating may restrict the number of samples that can be loaded at the lower temperatures. Pressures in stressed tube samples can be varied as required, controlled by choice of gas pressure and tube wall thickness.

The full experimental design and specimen loading will attempt to maximize use of available volume while meeting the broadest possible range of ADIP program objectives. The specimen materials to be loaded will be path A, path B, and path C alloys. These first experiments will emphasize scoping the effects of irradiation variables and thermal-mechanical treatment before irradiation on the irradiation performance of the DMFE reference heat of type 316 stainless steel. Path B specimens will include PE-16, and path C specimens will initiate scoping studies on titanium-base alloys and expand the irradiation response data base on vanadium-base alloys.

1.6.8 EBR-II Row 7 Irradiation Experiment -- M. L. Grossbeck, E. E. Bloom, J. W. Woods, and A. F. Zulliger⁴⁶

This irradiation is a joint experiment between programs at ORNL, HEDL, Battelle Pacific Northwest Laboratory, the University of Cincinnati, LASL, and Cornell University. The work is sponsored by ERDA Divisions of Reactor Development and Demonstration, Magnetic Fusion Energy, and Physical Research. The objectives of the DMFE parts of the experiment are to (1) investigate the effects of preinjected helium and irradiation temperature on bulk irradiation damage to candidate reactor materials, (2) investigate the effect of irradiation on Si_3N_4 and Y_2O_3 , and (3) correlate the effects of ion bombardment and neutron irradiation in bcc metals. A total of 2193 specimens are included, 984 of which are for the fusion reactor program, the largest contributor of specimens. The arrangement of specimens in the six large capsules is shown in Fig. 1.20. The scope and objectives of the individual portions of the experiment are outlined below. The subassembly and some of the experiments were described previously.⁴⁷

1.6.8.1 Irradiation Conditions

The subassembly (X-287) is being irradiated in row 7 of EBR-II in the 7A4 position. An exposure of 30,000 MWd has been authorized. It will result in a maximum fluence of $4 \times 10^{26} \text{ n/m}^2$ ($>0.1 \text{ MeV}$). Most specimens are contained in static sodium isolated from reactor coolant. The ceramic specimens are separately encapsulated. Neutron fluence dosimeters and temperature monitors have been included in the experiment.

1.6.8.2 Subassembly Description

Nine capsules are placed within a standard hexagonal EBR-II subassembly. Three sodium-filled capsules remain in thermal contact with the reactor coolant at temperatures between 370 and 400°C. Three

⁴⁶Engineering Division

⁴⁷M. L. Grossbeck and E. E. Bloom, "EBR-II Row 7 Irradiation Experiment," *Magnetic Fusion Energy Materials Technology Program Annu. Prog. Rep. June 30, 1975*, ORNL-5189, pp. 35-39.

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Fig. 1.20. Radiographs of Heat Pipe (top) and Gas Gap Capsules. Note the large number and variety of sample loadings in each capsule.

capsules are heat-pipe temperature controlled and are located directly above and welded to three gas-gap-controlled capsules. The heat pipes were designed by LASL personnel and are presently used by several EBR-II experimenters (LASL, NRL, and HEDL); thus feasibility of their use has been demonstrated. (The temperature control capability of a heat pipe is shown in Fig. 1.21.) The heat pipe capsules will operate at 575, 625, and 700°C. The gas-gap capsules have a specimen chamber surrounded by a gap containing low-pressure inert gas to conduct heat to the reactor coolant. The feasibility of the gas-gap concept has been demonstrated in several EBR-II experiments (e.g., X-034, -035, -100, and -100A). Two of the capsules are designed to operate at 525°C, the remaining one at 575°C.

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Fig. 1.21. Temperature Control Capability of a Heat Pipe.

Specimens are positioned in racks with provision for irradiation-induced swelling. Since refractory metals are included, each of the six capsules operating above reactor coolant temperature contains high-purity zirconium foil as a getter for oxygen.

1.6.8.3 Experimental Goals

Primary objectives of the fusion reactor portion of this experiment are to investigate the effects of large amounts of preinjected helium and define the temperature dependence of the radiation response of candidate materials. Helium has been injected into samples before irradiation in EBR-II. The validity of preinjection as a simulation technique for continuous helium production will be determined by comparison of helium-preinjected type 316 stainless steel samples irradiated in EBR-II with nonpreinjected samples irradiated in HFIR and possibly in the ORR. (In HFIR and ORR helium is produced continuously by the thermal neutron reactions described elsewhere.) Irradiations of stainless steel in both EBR-II and HFIR will bracket the He/dpa production rates expected in a fusion reactor, and by helium preinjection followed by irradiation in EBR-II the effectiveness of this as a simulation technique will be evaluated. For alloys that do not contain nickel, preinjection with helium or tritium doping followed by neutron irradiation are the only ways available at present to obtain helium and displacement damage levels expected in a fusion device.

Helium-doped materials include tensile samples with gage lengths of either 12 or 20 mm and pressurized tubes. The materials that have been doped with helium include type 316 stainless steel, PE-16, V-20% Ti, V-10% Cr-5% Ti, Nb, and Nb-1% Zr. Helium levels range from 2 to 200 at. ppm, and irradiation temperatures span the range 400 to 700°C.

Samples included for the evaluation of the effect of irradiation temperature on postirradiation tensile properties provide some overlap with the helium effects matrix. The major emphasis is on the bcc refractory metals, where mixed-spectrum fission reactor irradiations offer no unique advantages and fast reactor experiments provide higher displacement rates. The materials included are Nb, Nb-1% Zr, D-43, Mo,

and TZM. The range of irradiation temperatures is 400 to 700°C. Post-irradiation tensile tests will be conducted at temperatures from 20°C to the irradiation temperature.

To obtain information on the effects of fast-neutron irradiation on weldments in the potential first-wall alloys, a series of welded samples of type 316 stainless steel 20% cold worked, PE-16, and V-20% Ti has been included. Both gas tungsten-arc and electron-beam welding were employed, with the use of welding rods of several compositions. The type 316 stainless steel and PE-16 are being irradiated at 400, 525, and 575°C, and the V-20% Ti is being irradiated at all five temperatures.

Some of the samples included in this experiment were provided by DMFE-funded programs at other sites. The material content and goals of these parts of the experiment are not detailed here.

1.6.8.4 Status of the Experiment

The hazards analysis, fabrication, and assembly were completed during FY 1976. The experiment was accepted by EBR-II and assigned subassembly number X-287. Irradiation began with run 87 on January 8, 1977. It is scheduled to be removed in January 1979.

1.6.9 EBR-II Row 8 Irradiation Experiment — F. W. Wiffen, J. Bentley, J. A. Horak, and J. W. Woods

This joint experiment between ORNL and LASL was described in detail in an earlier report.⁴⁸ It included a number of ORNL specimens of Nb, V, and Mo alloys, as well as specimens of PE-16 and type 316 stainless steel. Design irradiation temperatures were 500, 600, 700, and 800°C.

The experiment was designated subassembly X-264 and was irradiated in position 8D4 of EBR-II. Irradiation between December 1975 and January 1977 accumulated an exposure of 15,915 MWd, 118% of the original goal exposure. LASL is currently disassembling the experiment and will return specimens to ORNL when sample recovery is completed.

⁴⁸F. W. Wiffen, J. Bentley, J. A. Horak, and J. W. Woods, "EBR-II Row 8 Experiment," *Controlled Thermonuclear Materials Technology Program Annu. Prog. Rep. June 30, 1975*, ORNL-5082, pp. 42-43.

1.7 CALCULATED IRRADIATION RESPONSE OF MATERIALS USING A FUSION-REACTOR FIRST-WALL NEUTRON SPECTRUM⁴⁹ — T. A. Gabriel,⁵⁰ B. L. Bishop,⁵¹ and F. W. Wiffen

The irradiation response of a number of elements to a fusion-reactor first-wall neutron spectrum has been calculated. The results were used to evaluate the composition sensitivity of helium generation in candidate alloys. For alloys within the Fe-Ni-Cr system the helium production rate can vary by a factor of four and is dominated by their nickel contents. Variation is also possible within a single alloy specification. In the case of type 316 stainless steel, compositional variation within the AISI alloy specification allows variation in helium production rate of $\pm 9\%$ about the mean. Variations in composition for this same alloy within the more tightly controlled RDT specification allows variation of only $\pm 2.4\%$ about the mean helium production rate. The calculated production rates for Cr, Fe, Ni, and type 316 stainless steel are given in Table 1.8.

⁴⁹Abstracted from a paper submitted to *Nuclear Technology* and to be issued as ORNL/TM-5956. See also Chap. 6 of this report.

⁵⁰Neutron Physics Division.

⁵¹Computer Sciences Division.

Table 1.8. Dependence of Helium Production Rate During Fusion Reactor Irradiation on Alloy Composition

Material	Helium Production Rate	
	(at. ppm/s)	(at. ppm per 1 MW-year/m ²)
Cr	32.6×10^{-7}	103
Fe	34.9	110
Ni	130	410
316, AISI Specification		
Min. He	43.1	136
Max. He	51.7	163
316, RDT Specification		
Min. He	48.2	152
Max. He	50.5	159

Coupled with the well-established effect of helium on the engineering properties of metals, this sensitivity in the helium production rate to alloy composition requires that each alloy be separately treated. The assumption of similar irradiation response to fusion reactor service for all alloys in a single class is not adequate.

The wide range of elements treated in this analysis allow most alloy compositions to be synthesized. The response of different alloys to fusion reactor service can be predicted from this information, and alloy comparisons are then on a consistent basis.

1.8 QUALIFICATION OF INNOVATIVE TECHNIQUES AS PROBES OF THE DAMAGE MICROSTRUCTURE — F. W. Wiffen, R. W. Hendricks, C. L. Snead, Jr.,⁵² and S. Spooner⁵³

Knowledge of the microstructural response to irradiation plays a prominent role in an estimate of the potential for application of any alloy in fusion power systems. If existing alloys are judged to be inadequate, microstructural response will be one of the key properties on which programs to develop improved alloys will be based. This emphasis results in considerable potential payoff for any improved diagnostic techniques that can be developed for microstructural evaluation.

Microstructural changes produced by irradiation are usually examined by transmission electron microscopy (TEM). While this technique is the most direct method and in many cases the most exact, it also suffers limitations. It is slow, requiring a great deal of time of highly skilled scientists, it samples a very small volume of material, and it has size limitations that exclude treatment of either very small or very large defect structures. The integrated response to irradiation can often be determined by measuring dimensional changes or the material density. This measurement tells only the sample swelling; while it is simple and inexpensive, it does not provide any information on controlling mechanisms.

⁵²Brookhaven National Laboratory.

⁵³Georgia Institute of Technology.

Two promising new techniques for probing the damage microstructure are small-angle scattering and positron annihilation. Both these techniques are being evaluated for usefulness on the DMFE Radiation Effects Program. In both cases the evaluation is against available TEM information. Information outside the limits of the TEM techniques is also being sought.

Small-angle scattering techniques using both x rays and neutrons have been shown to respond to irradiation-produced voids. Both methods offer the potential of rapid data gathering and on-line computer reduction of the data. The scattering measurements will generate the average size, volume fraction, and total surface area of a population of scattering centers in a metal polycrystal. If the scattering centers are ordered, the lattice parameter of the ordered structure can be determined. Information on the size distribution and the shape of the scattering centers is coupled; assumption or knowledge of one then yields the other.

The application of x-ray and neutron small-angle scattering techniques to the study of irradiated microstructures requires special instrumentation. The long-slit cameras are unsuitable for these studies because of multiple scattering problems and because of the oriented, faceted nature of cavities. The experiments must be performed in point geometry; the lost intensity is recovered by using two-dimensional position-sensitive detectors and on-line computer data acquisition systems. Appropriate x-ray and neutron small-angle scattering facilities have been recently commissioned at ORNL.

For most irradiated samples of interest to this program the major scattering at small angles is produced by cavities. However, in some cases high concentrations of small loops are believed to contribute to the scattering. The potential also exists for fine precipitate particles in high concentrations to contribute to the scattering. This could become important in some of the complex engineering alloys in which precipitation formation is expected.

Experiments are currently under way to test the ability of small-angle scattering to produce microstructural information in irradiated

samples of simple metal alloys. The technique is calibrated against the TEM characterization of the same samples.

Small-angle neutron scattering (SANS) with monochromatic 0.474-nm neutrons has been used to examine a set of irradiated niobium and Nb-1% Zr samples. The same samples and two sets of irradiated molybdenum and molybdenum-alloy samples are also being examined by small-angle x-ray scattering (SAXS) with a monochromatic beam of Mo K α radiation (71 pm).

Preliminary analysis of the scattering data indicates agreement between the calculated size of the scattering centers and the TEM measurements of average void size. Unprocessed data show signals from the ordered void arrays known to exist in some samples and in one sample an appreciable component of scattering believed to be due to a high concentration of very small dislocation loops. A full evaluation of the potential role of SANS and SAXS techniques in the program awaits completion of examination of these sample sets.

Positron annihilation has also been shown to be a sensitive probe of the defect microstructure of a solid. Since the rate of annihilation by combination with electrons is proportional to the electron density at the annihilation site, positron lifetimes are a measure of the local electron concentration of the sample. The energy spectrum of the gamma rays from the annihilation reaction also carry information on the momentum distribution of the annihilation electrons. Positrons in a metal tend to be trapped at defects with low electron densities. This results in increased positron lifetimes, and the increased lifetime of the total sample can be related to the defect concentration. The technique is sensitive to vacancies and vacancy-type defects since these have the low electron densities. In this program it is a potential probe for voids, gas bubbles, and dislocations. The sensitivity to low defect concentrations also suggests that the technique may be useful for defects in the submicroscopic size range.

Positron annihilation measurements following the isochronal annealing of helium-injected aluminum showed the positron lifetime to change without microstructurally observable changes occurring in the sample. This confirms the ability of the positron technique to complement the TEM measurements in tracing the early stages of defect cluster nucleation and growth. (See also Sect. 1.9.2.)

Both Doppler-broadening and positron annihilation lifetime measurements are under way in neutron-irradiated niobium and molybdenum alloys and in helium-doped samples of the same materials. Preliminary results show strong signals resulting from the defect microstructures, and trends with irradiation conditions are as predicted from other measurements. Planned work on these samples will establish the correlation between the positron annihilation signatures and the TEM observations. The changes produced by isochronal annealing at temperatures above the irradiation temperature will also be evaluated. There is a special interest in establishing the behavior of helium in these samples, as tracing helium migration by other techniques has proved to be very difficult.

1.9 SUMMARIES OF OTHER PUBLISHED PAPERS

1.9.1 Radiation Effects in Structural Materials for Fusion Reactors⁵⁴ -- F. W. Wiffen

Neutron irradiation of structural materials in the high-flux regions of a fusion reactor, especially the first wall of the reactor, will result in swelling due to cavity formation and loss of ductility through both lattice hardening and the effect of transmutation-produced helium on the fracture mode. These phenomena are generally expected, although the intensity and relative importance will be strong functions of the material and reactor operating conditions. A consideration of the effects of the 14-MeV *d-t* fusion neutrons on metals suggests that, although the 14-MeV neutrons will produce damage at a higher rate than lower energy neutrons, there is no basis for anticipating different forms of damage, except for the differences that are due to the higher rate of transmutation reactions.

Irradiation experiments designed to evaluate materials for fusion reactor service must be conducted in a number of facilities, since none of the ion bombardment, fission-reactor, or high-energy neutron sources

⁵⁴Summary of Chap. 5 of *Critical Materials Problems in Energy Production*, ed. by C. Stein, Academic Press, New York, 1976.

adequately simulate the fusion reactor environment. Used together in a well-coordinated evaluation program, the combined results from all of these facilities can be expected to lead to an understanding of the radiation effects that will occur in fusion reactors and to result in the development and qualification of materials for fusion reactor service.

1.9.2 Tracing the Evolution of Bubbles in Helium-Injected Aluminum by Means of Positron Annihilation⁵⁵ — C. L. Snead, Jr.,⁵²
A. N. Goland,⁵² and F. W. Wiffen

Pure aluminum was injected with 0.6 at. ppm He. Positron lifetime measurements were performed following isochronal annealing between 25 and 600°C. Transmission electron micrographs were taken after several of these anneals. The results are consistent with a model in which the injected helium quickly becomes substitutional. This substitutional helium is a trapping site for positrons. By monitoring the concentration and size of this trapping site through the measurement of the positron lifetimes we conclude that migration of the helium begins at about 100°C and that helium continually agglomerates up to about 250°C. Above this temperature the increasing size of the trapping site indicates bubble growth. Bubble growth proceeds up to 600°C, but whether by continued arrival of helium at nucleation sites or by bubble migration and coalescence (or both) is indeterminable. The major stage of bubble growth is between 300 and 600°C. Electron microscopy verified the presence of bubbles in both grain boundaries and matrix after annealing to 625°C, but only cavities in grain boundaries were observed on annealing to 500°C. Positron-lifetime measurements are a useful complement to electron microscopy in the study of bubbles in metals, especially in the early stages of the nucleation and growth of small cavities.

⁵⁵Abstract of *J. Nucl. Mater.* 64: 195-205 (1977).

1.9.3 The Microstructure and Swelling of Neutron Irradiated Tantalum⁵⁶ — F. W. Wiffen

Electron microscopy of tantalum that had been irradiated to 2.5×10^{26} n/m² (>0.1 MeV) at temperatures between 425 and 1050°C established that this temperature range effectively brackets the swelling temperatures of tantalum. No voids were detected after irradiation at 425°C. The temperature for maximum swelling was inferred to be near 600°C, and the swelling at about 1000°C was very small. At 585 and 790°C a high concentration of small voids resulted in technologically significant levels of swelling, 0.5 to 2.5%.

1.9.4 The Effects of CTR Irradiation on the Mechanical Properties of Structural Materials⁵⁷ — F. W. Wiffen

Mechanical properties of fusion reactor structural materials are important in determining the reliability and economics of fusion power. Furthermore, these properties are significantly affected by the high neutron flux experienced by components in the regions of the fusion reactor near the plasma. In general, irradiation hardens the material and reduces ductility. An exception to this is in some complex engineering alloys, where either hardening or softening can be observed depending on the alloy and the irradiation conditions. Regardless of this restriction, irradiation usually reduces ductility. Available tensile data examined in this paper show that significant ductility reduction can be found for irradiation conditions typical of fusion reactor operation. Consideration of these effects shows that extensive work will be needed to fully establish the in-service properties of fusion reactor structures. This information will be used by designers to develop conditions and design philosophies adapted to avoid the most deleterious conditions and minimize stresses on structures. The

⁵⁶Abstract of a paper to be published in *Journal of Nuclear Materials*.

⁵⁷Abstract of ORNL/TM-5624 (November 1976). The paper was developed from a talk at the ANL-AUA Faculty Institute, "Curriculum Development in Fusion," Aug. 9-13, 1976. The proceedings of the Institute will be published by ANL.

information will also be used as input to alloy development programs with goals of producing materials more resistant to property degradation during irradiation.

Clearly, a great deal of additional work will be required both to understand the effect of fusion reactor irradiation on properties and to develop optimal alloys for this application.

1.9.5 The Effects of Irradiation on the Fracture of Austenitic Stainless Steels⁵⁸ - M. L. Grossbeck, J. O. Stiegler, and J. J. Holmes⁵⁹

Irradiated materials fracture by mechanisms that occur in unirradiated materials in addition to mechanisms related to irradiation. This review will be limited to austenitic stainless steels for use as core structural materials for fast breeder reactors.

The microstructural changes that occur in different temperature regions have a strong influence on fracture behavior and can also be classified by temperature. Below about $0.4T_m$, where small dislocation loops strengthen the lattice, short-term tensile tests result in ductile failure.

Above about $0.4T_m$, where voids and a dislocation structure are formed, a new fracture mechanism appears in which plastic deformation is confined to small groups of adjacent slip planes. This "channel fracture" is associated with channel deformation, which was first observed in irradiated copper and later confirmed in molybdenum, nickel, and stainless steel. Channels swept clear of defects are formed by extensive dislocation motion within the localized region. In stress-rupture tests at the same temperatures, grain boundary fracture dominates. Here, crack growth proceeds in a stable manner until the crack is sufficiently large for unstable crack propagation, resulting in failure. A significant amount of work on crack growth rates in irradiated stainless steels indicates that the crack growth rate is little affected by irradiation.

⁵⁸Summary of paper presented at the International Conference: Radiation Effects in Breeder Reactor Structural Materials, June 19-23, 1977, Scottsdale, Arizona.

⁵⁹Hanford Engineering Development Laboratory, Richland, Wash.

The major effect of irradiation in this temperature region appears to be the reduction in fracture toughness, resulting in failure at an earlier stage of growth. Application of J-integral analysis to type 316 stainless steel confirms the reduction in toughness of material irradiated to low fluences. However, further work is necessary before this method can be used to predict failure in core materials.

Above approximately $0.6T_m$, fracture is complicated by irradiation-enhanced precipitation of second phases and precipitation of transmutation-produced helium. Here, grain boundary fracture also dominates, but cracks are blunted as a result of reduced yield strength, grain rotation, and internal helium pressure. Fracture surfaces reveal cavities, suggesting that failure was due to bubble coalescence.

Even below $0.6T_m$, cavity coalescence appears in slow stress-rupture tests, and helium has been shown to play a major role in fracture. Bloom and Clausing have fractured specimens of type 304 stainless steel in an ultrahigh vacuum system equipped with a residual gas analyzer and an Auger spectrometer. Measuring the amount of helium released upon fracture showed that the concentration of helium at the boundaries was approximately proportional to the neutron fluence. This work demonstrated the presence of helium at grain boundaries rather conclusively. Auger spectroscopy revealed depletion in chromium and enrichment in iron and nickel on the grain boundary fracture surfaces. This result is consistent with that of Okamoto and Wiedersich, who attribute the behavior to an irradiation-induced defect drag mechanism.

In examination of type 316 stainless steel cladding of fuel pins irradiated in EBR-II, Hunter and co-workers found that fracture is enhanced in regions where fuel was in contact with the cladding. Measurements of helium profiles revealed that implantation of helium from ternary fission is a possible cause. Certainly much future research on fracture of irradiated materials will focus on helium.

1.9.6 Mechanisms of Ductility Loss in CTR First-Wall Structural Materials⁶⁰ - F. W. Wiffen

The major irradiation-produced problem in metal structural components in the high-flux regions of fusion reactors will be loss of ductility. Available conceptual reactor designs and existing radiation-effects data are combined to illustrate the mechanisms of ductility loss in materials and operating conditions of potential fusion reactor interest. In the very large and complex fusion reactor components, stresses will be imposed directly by coolant temperature gradients, imposed cyclically by the duty cycle of the reactor, and developed over long time periods by irradiation-produced swelling. Accommodation of these stresses will require some plastic deformation, although the required levels of ductility cannot yet be specified.

Several ductility reduction processes are possible, depending on the choice of structural material and the temperature-flux parameters of reactor operation. In some body-centered cubic (bcc) metals, the ductile-to-brittle transition temperature (DBTT) can be raised by irradiation to well above room temperature, and perhaps to the reactor operating temperature. Failures in this case are predominantly by a cleavage mode, with some component of grain boundary separation. In metals and loading conditions where failures are ductile and transgranular, elongation to fracture can be severely reduced, although the local ductility as measured by reduction of area at the fracture may be very high. The failure mode in this case occurs by the onset of plastic instability at yielding. All deformation is restricted to the necked region. Ductility is also reduced by the presence of insoluble gas in the grain boundaries. This allows grains to separate at stresses well below those required to deform the matrix. A general loss of ductility usually accompanies the lattice hardening resulting from irradiation-produced defects. This is of importance mainly as it relates to the other ductility loss mechanisms mentioned here.

⁶⁰Taken from *Trans. Am. Nucl. Soc.* 23: 134 (1976).

Three types of behavior have been established in bcc metals with potential for fusion reactor application.⁶¹ In the case of molybdenum alloys, the DBTT can be raised to at least 550°C by neutron irradiation. While the ranges of irradiation and test conditions examined are too restricted to rule out molybdenum alloys for fusion reactor use, the data do demonstrate the need for further testing to evaluate the ability of molybdenum structures to withstand the stresses generated during shutdown cycles of a reactor. Ductility loss by the onset of plastic instability is important under some conditions in niobium and tantalum alloys, where as low as 0.1% uniform elongation in irradiated samples has been measured. In service this could result in the situation in which any component overloaded to its yield stress will deform to fracture under constant or decreasing load. Under other irradiation and test conditions, niobium and tantalum alloys exhibit a general ductility loss while still retaining appreciable uniform elongation. Vanadium alloys behave similarly. Ductility reductions are observed, but the uniform elongation remains adequate for structural application.

In austenitic stainless steels, the leading candidate material for first-generation fusion reactors, high helium generation rates promote grain boundary decohesion at low stresses. This intergranular failure mode will limit the application of type 316 stainless steel^{62,63} to temperatures below 600°C. The importance of helium-enhanced intergranular failure in limiting the fusion reactor application of other classes of alloys has not generally been established, although limited evidence

⁶¹F. W. Wiffen, "The Tensile Properties of Fast Reactor Neutron Irradiated BCC Metals and Alloys," pp. 176-97 in *Defects and Defect Clusters in B.C.C. Metals and Their Alloys*, Nucl. Metall. 18 (Proc. Int. Conf. Aug. 14-16, 1973), National Bureau of Standards, Gaithersburg, Md.

⁶²E. E. Bloom and F. W. Wiffen, "The Effects of Large Concentrations of Helium on the Mechanical Properties of Neutron-Irradiated Stainless Steel," *J. Nucl. Mater.* 58: 171-84 (1975).

⁶³E. E. Bloom, F. W. Wiffen, P. J. Maziasz, and J. O. Stiegler, "Temperature and Fluence Limits for a Type 316 Stainless-Steel Controlled Thermonuclear Reactor First Wall," *Nucl. Technol.* 31: 115-22 (1976).

suggests that it is important in some other alloys.⁶⁴ Stainless steel irradiated under simulated fusion reactor conditions at lower temperatures also shows reduced tensile ductilities, but the failure mode has not been established.

Proposed fusion reactor operating conditions will lead to severely reduced ductility of the reactor structural materials. Conditions have been identified where ductility limitations are set by the intergranular fracture mode, by the onset of plastic instability at yielding, and by the shift of the DBTT into the operating temperature range of the reactor. The available data establish that very careful reactor design will be required to keep stresses developed during operation compatible with the material properties, and that future detailed experimentation will be required to define the temperature-fluence-strain-rate dependence of ductility reduction in proposed fields of reactor operating parameters.

⁶⁴D. Kramer et al. "A Survey of Helium Embrittlement of Various Alloy Types," p. 109 in *Irradiation Embrittlement and Creep in Fuel Cladding and Core Components*, British Nuclear Energy Society, London, 1973.

2. SURFACE STUDIES

R. E. Clausing

2.1 SXAPS STUDIES IN ORMAK — L. C. Emerson and R. E. Clausing

During the last year of operation of the ORMAK experiment a series of surface studies was carried out using an *in situ* soft x-ray appearance potential spectrometer (SXAPS). The experiments were designed to assist in the identification of the impurities residing on the liner or "first wall" of ORMAK and to characterize their behavior. Impurities greatly affect the quality of the plasma, and plasma-wall interactions are responsible for the transfer of wall impurities to the plasma. Such interactions occur in all tokamak devices and may be responsible for essentially all plasma impurities.

Soft x-ray appearance potential spectroscopy was chosen primarily for two reasons: (1) it is as least as sensitive, if not more so, as any other surface analytical technique and (2) the technique, especially the hardware, is simple, thus facilitating *in situ* measurements. The most important disadvantage of SXAPS is that, at its present state of development, it is only a qualitative technique. However, this does not represent a real disadvantage in this application, as the experiments were intended to simply identify the surface contaminants and to provide a monitor for the conditions of the liner wall.

The spectrometer was mounted in a metal tube and attached to the ORMAK liner at a position on the torus approximately diagonal to the limiter. A schematic diagram of the instrument is shown in Fig. 2.1. The wall sample assembly consisted of an octagonal wheel that could be rotated via a mechanical linkage from a position outside the vacuum vessel. One face of the wheel was always flush with the liner wall and when rotated 180° faced the spectrometer. The surface of the sample wheel was prepared in the same way as the ORMAK liner (i.e., type 304L stainless steel base material overcoated with a 0.2- μ m-thick gold layer to provide an inert surface). One face of the sample wheel consisted of a thin stainless steel cover plate to provide a calibration standard for the spectrometer. A second face was covered by a thin gold foil

Fig. 2.1. SXAPS Probe for ORMAK. The voltage on the sample wheel is varied so that the minimum electron energy required to generate characteristic soft x rays from the surface can be determined. This is done by detecting the increase in photoelectron current from the photocathode as the electron energy is increased through the threshold. Sophisticated electronics is required, but the detector is simple and does not require carefully focused electron beams or energy analyses.

to test the effectiveness of the antidiffusion barrier. The spectrometer was provided with strip heaters capable of maintaining a probe temperature of 180 to 200°C. Laboratory studies had earlier shown that these temperatures were necessary to prevent probe contamination when operating in an environment contaminated by hydrocarbons.¹

The first and perhaps the most important result from the spectroscopic measurements indicates the extent of the hydrocarbon contamination. This result confirmed what had been found earlier with another version of the appearance potential spectrometer and also seen in laboratory Auger electron spectroscopic studies using swatch samples from the

¹R. E. Clausen, L. C. Emerson, L. Heatherly, and R. J. Colchin, "Surface Impurities and 'Clean-Up' Techniques in ORMAK," *J. Nucl. Mater.* 63: 495-500 (1976).

liner.² Both hydrogen and oxygen glow discharge cleaning within the liner were effective in removing the carbon contamination from the sample wheel. Spectra obtained following oxygen discharges always showed evidence of large amounts of oxygen on the surface. The height of the oxygen peaks generally diminished with time both from the action of normal tokamak plasmas and because of recontamination with hydrocarbons. This recontamination was quite rapid. Evidence of carbon could be found within 15 min after its removal by glow discharge cleaning. Strong chromium and iron lines were always found on the stainless steel face after discharge cleaning. The intensity of these lines also diminished with time, apparently as a result of overcoating with hydrocarbon contaminants.

Gradually, over a period of some weeks, evidence of other contaminants appeared on the gold-coated sample surfaces. Iron was the most prevalent element, with some chromium being found as well as traces of calcium. Although occasional signs of iron and chromium appeared on the surface of the gold foil, the primary contaminant on the foil was carbon, with oxygen lines always appearing following discharge cleaning using oxygen. The chromium and iron signals seen on the gold surfaces could have resulted from the inadvertent exposure of an edge of an adjacent stainless steel face to the electron source. The lack of consistent clear-cut evidence of iron and chromium on the surface of the gold foil suggests that the stainless steel components, especially iron, are not associated with the plasma but are diffusing through the platinum and gold coatings to appear on the surface of the gold-coated parts of the sample wheel.

The results obtained with the *in situ* SXAPS measurements have been valuable in that they have shown not only the extent of the hydrocarbon contamination but the rapidity with which recontamination occurs following cleanup using glow discharges. These studies have also pointed out the need for an analytical facility with which samples

²R. E. Clausing, L. C. Emerson, L. Heatherly, R. J. Colchin, and J. C. Twichell, "Measurement and Modification of First-Wall Surface Composition in the Oak Ridge Tokamak (ORMAK)," *J. Vac. Sci. Technol.* 13: 437 (1976).

can be alternately exposed to tokamak plasmas and a surface analysis capable of quantitative elemental determination. Such a facility is presently under development.

2.2 DEVELOPMENT OF SURFACE ANALYSIS STATION FOR ISX — L. C. Emerson, R. E. Clausing, and L. Heatherly

The Impurity Study Experiment (ISX) will consist of a tokamak slightly larger than ORMAK and dedicated to the study of various aspects of the surface and plasma impurity problems. Construction of the machine is well-advanced, and initial operation is planned for the summer of 1977.

One of the first experiments to be installed on ISX will be the surface physics experiment. Basically, this experiment will be divided into three separate phases, with each succeeding phase building upon its forerunner and each providing an additional degree of sophistication in analytical and experimental capability.

2.2.1 Phase I

The initial surface physics experiment will be installed entirely within the protective enclosure surrounding the machine and as close to the torus as is physically possible. Thus, it will not be possible to obtain data during normal machine operation as the enclosure cannot be occupied then. The analytical experiment to be used consists of an existing high-resolution Auger electron spectrometer to provide quantitative elemental surface analysis and a thermal desorption apparatus to quantitatively monitor gas evolution. The thermal desorption apparatus consists of an electron bombardment gun and a quadrupole mass spectrometer. The analytical chamber is also equipped with precision sample handling manipulators to permit samples to be transferred to and from the chamber as well as to be accurately positioned within the chamber. A separate ion pump is flanged directly to the analysis chamber to provide the maximum pumping speed.

The analysis chamber is connected to ISX by means of a 38-mm-diam (1 1/2-in.) tube, which contains the sample transport mechanism. The

tube is divided into three sections separated from each other by all-metal sealed gate valves and from the analysis chamber by a metal sealed tee valve. The transfer system is equipped with a separate ultrahigh vacuum pumping system and can be used to lock samples in and out. The valving arrangement ensures that the analysis chamber is always isolated from the ISX environment and that the transfer tube is exposed to the ISX environment only during the moments when samples are being introduced to or removed from the exposure positions. The exposure module is equipped with apparatus to provide a precise indication of sample position as well as sample temperature.

2.2.2 Phase II

The essential difference between Phase I and Phase II lies in the location of all the analytical equipment outside the machine enclosure, so data can be obtained during routine tokamak operation. This change necessitates that all the transfer operations within the enclosure be remotely operable. While this adds greatly to the complexity of the facility, the advantages of being able to obtain data from individual shots are very significant and will add greatly to the value of the facility.

The analytical facility will be unchanged from that of Phase I with the possible exception of a modification to the quadrupole mass spectrometer to provide secondary ion mass spectroscopy (SIMS) as an additional surface analytical tool. The transfer tube diameter will be increased to 0.15 m (6 in.), and additional pumping will be provided to enhance the ultrahigh vacuum capability of the system.

We anticipate that Phase II operation will follow Phase I by approximately three months. If the present schedule holds firm, we may expect the implementation of Phase II by October or November 1977.

2.2.3 Phase III

The plans for Phase III provide for the addition of a sample preparation facility and a more versatile analysis chamber to allow a more complete sample characterization. The sample preparation equipment will include coating apparatus and deposition monitoring equipment.

Recent changes in ISX plans may influence the implementation of Phase III. Present plans for ISX call for operation until February 1978, at which time the experiment will be modified to permit the installation of a second liner, which is compatible with neutral beam injection heating. ISX-B is scheduled to begin operation in July 1978. The nature of the surface physics program at that time will be influenced by the results obtained during the Phase I and Phase II experiments and by the needs of the program at that time.

2.3 WALL MATERIAL TEST FACILITY - R. E. Clausing, L. C. Emerson, and L. Heatherly

We previously reported³ results indicating that the equivalent of 20×10^{19} to 50×10^{19} hydrogen molecules/m² could be loaded into oxidized stainless steel surfaces. These data were obtained with a cold cathode dc glow discharge cell described earlier.² More recently we have extended these studies using a multielement hot-cathode gas discharge apparatus, which allows a much greater degree of flexibility in operating parameters.⁴ A schematic diagram of the apparatus is shown in Fig. 2.2. The principal features of the apparatus are the hot cathode, which allows for a wide range of operating pressures, and an arrangement of the discharge electrodes and biasing power supplies so as to provide control over both the ion energy and ion flux.

The discharge produces plasma-wall interaction parameters that are quite similar to those in a typical tokamak. The ion flux to the wall is in the range up to 10 A/m^2 , and the ion energies can be varied up to values in excess of 1000 eV. The wall of the discharge cell is a refluxing wall, as are the liners of present day tokamaks.

³R. E. Clausing, L. C. Emerson, L. Heatherly, and R. J. Colchin, "Wall Conditioning Studies for ORMAK and ISX," paper presented at the International Symposium on Plasma Wall Interaction, Oct. 18-22, 1976, Jülich, West Germany.

⁴L. C. Emerson and R. E. Clausing, "Hydrogen Loading and Clean-Up of Tokamak Wall Materials during Glow Discharge Cleaning," paper presented at 2nd Joint Conference, Canadian Institute of Chemistry/American Chemical Society, Montreal, Canada, May 30-June 2, 1977.

THE HOT CATHODE GLOW DISCHARGE IS MORE COMPLEX BUT MORE VERSATILE

- a. ION FLUXES FROM 0 TO $>1 \text{ mA/cm}^2$
- b. ION ENERGY $\sim 20 \text{ eV} - 1000 \text{ eV}$ (VELOCITY NORMAL TO SURFACE)
- c. PRESSURE $\sim 10^{-2} \text{ Torr}$ TYPICALLY
- d. WALL IS A REFLUXING WALL
- e. SAMPLE SURFACE CAN INCLUDE ENTIRE TUBE WALL

Fig. 2.2. Multielement Hot-Cathode Glow Discharge Apparatus. Electrons supplied by the hot cathode permit stable glow discharge to be obtained over a wide range of operating parameters.

Samples can be transferred under ultrahigh vacuum conditions directly from the discharge cell to a separate analysis chamber. The discharge cell and the transfer system have their own separate ion pumps, which can be valved off during operation at pressures above their operating range. The analysis chamber is isolated from the transfer system by another valve so that a low base pressure can be maintained there continuously. The transfer system allows samples to be rapidly moved from one part of the system to another and permits the isolation of these parts by means of gate valves.

The test facility is equipped with its own diagnostics, which include a quadrupole mass spectrometer to analyze the gas and impurity concentrations and a visible-near ultraviolet optical spectrometer to identify plasma impurities in the discharge. Two capacitance manometers are used in normal operation: one in a feedback arrangement to control a motor-driver leak valve to maintain a preselected pressure in the

cell and the other connected in the gas supply line and used as a differential manometer to measure flow rates.

The analysis chamber contains an Auger electron spectrometer and a second quadrupole mass spectrometer, which, when used with an electron gun to provide heating, yields thermal desorption information. The chamber also is equipped with an ion sputter gun, which provides depth profiling capability. Additional ports are available so that the analytical capabilities could be extended if the need arises.

The initial studies carried out with the new facility have extended the hydrogen loading studies made with the cold-cathode discharge cell.² One series of studies used oxidized stainless steel samples exposed to a standard hydrogen glow discharge for various periods of time. When we stop the discharge, the desorption of hydrogen from the sample is followed as a function of time. A typical set of data, presented in Fig. 2.3, shows the hydrogen desorbed following a 30-min glow discharge. The slope of the desorption curve immediately after the discharge ends indicates that a monolayer of hydrogen is desorbed within the first 0.2 s. The rate of desorption decreases with time, but over the course of the 30 min required to obtain the data in Fig. 2.3 the pressure increase of 20 mPa (1.5×10^{-4} torr) corresponds to the desorption of 5×10^{20} atoms/m² of hydrogen. Other desorption experiments have been followed for much longer times, and hydrogen continues to desorb for many hours. The hydrogen is sorbed in the surface oxide layer, and a heavily oxidized surface holds a significantly larger amount of hydrogen than does a surface only lightly oxidized. The thickness of the oxide can be varied reproducibly by exposure to the reducing atmosphere of the glow discharge.

Measurements of the amount of hydrogen thermally desorbed from these samples indicate that the atoms are only loosely bound and that a substantial fraction of those held in or near the surface are desorbed in a matter of hours, even at room temperature. From the characteristics of the desorption curve the activation energy for the desorption of hydrogen is found to be of the order of 0.1 MJ/mol (1 eV/atom). Another series of studies, carried out to determine the effect of ion energy

Fig. 2.3. Desorption of Hydrogen From Stainless Steel. The pressure rise following a 30-min glow discharge is caused by the rapid desorption of large amounts of hydrogen from an oxidized stainless steel surface. The first break in the curve indicates a change in the x axis time base and the second break represents a decrease in the y axis sensitivity. The pressure scale is in units of 10^{-6} torr. The 100 mark corresponds to 13 mPa.

on the level of the water impurity, has shown that low-energy ions tend to increase the water vapor levels in the plasma. These studies were made spectroscopically during the glow discharges through a sapphire window mounted along the axis of the discharge tube. In addition to water, C, O, Cr, Fe, and Ni have been tentatively identified as plasma impurities.

These studies are being continued systematically to determine the effect of various conditions on the plasma-wall interaction. These conditions include ion flux, ion energy and temperature as well as various wall materials, both homogeneous and coated walls.

3. COMPATIBILITY STUDIES

J. H. DeVan

3.1 LITHIUM THERMAL CONVECTION LOOP TESTS¹ — J. E. Selle, J. R. DiStefano, and J. H. DeVan

A thermal convection loop test program was initiated to study the corrosion of austenitic stainless steels in lithium under dynamic flow conditions in a temperature gradient and to determine the effect of various additives to lithium on its corrosion behavior. Tests have been conducted as a function of temperature between 500 and 650°C with a 200°C temperature difference, as a function of time (up to 10,000 hr), and as a function of lithium purity.

Table 3.1 summarizes the thermal convection loop conditions and gives the status of the tests. The maximum corrosion rates for type 316 stainless steel after 3000 hr are plotted as a function of temperature in Fig. 3.1. The data appear to follow an Arrhenius-type relation:

$$C = C_0 e^{-Q/RT}, \quad (1)$$

where

- C = is the corrosion rate,
- Q = is the activation energy,
- R = is the gas constant, and
- T = is the absolute temperature.

The data were fitted to the equation

$$1000/T = 1.12 - 0.0969 \ln C \quad (2)$$

by linear regression analysis and from the slope of the curve, Q was found to be about 39 kJ/mol (14 kcal/mol).

¹Based on J. E. Selle, "Corrosion of Iron-Based Alloys by Lithium," pp. 453-62 in *Proc. Int. Conf. Liquid Metal Technology in Energy Production* (held May 3-6, 1976, Seven Springs Mountain Resort, Champion, Pa.), M. H. Cooper, ed., CONF-760503-P2 (1976).

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Table 3.1. Summary of Lithium Thermal Convection Loop Tests

Loop	Alloy Type	T_{max}^a (°C)	Time (hr)	Remarks
1	304L	600	3,000	Completed
2	321	600	3,000	Completed
3	316	600	3,000	Low nitrogen (completed)
4	316	600	3,000	High nitrogen (completed)
5	316	600	10,000	Complete 6/24/77
6	316	600	3,000	Completed
7	316	650	3,000	Completed
8	316	500	3,000	Completed
9	316	600	3,000	Al addition complete 7/13/77
10	316	600	3,000	Mn addition complete (~9/1/77)
11	316	600	3,000	Ca addition not started

^aTemperature difference 200°C.

Fig. 3.1. Comparison of Corrosion Rates of Type 316 Stainless Steel in Thermal and Forced Convection Systems Operated with Molten Lithium. Temperatures and corrosion rates were measured at the heater exits.

Types 304L and 321 stainless steel were also tested for 3000 hr at 600°C, and weight change data were similar to those for type 316 stainless steel. The effect of time on corrosion rate has not yet been determined.

Additives to lithium are also under test as corrosion inhibitors. Such additives have included nitrogen, calcium, aluminum, and manganese, which were selected after results of capsule tests had been reviewed to determine the elements that had the most significant effects on corrosion. Aluminum and manganese are potential corrosion inhibitors. Nitrogen is a common impurity in lithium and generally increases corrosion rate. Calcium is a strong nitrogen getter and is relatively soluble in lithium.

3.2 COMPATIBILITY TESTS IN STATIC LITHIUM - J. E. Selle

Capsule tests have been conducted in lithium (1) to identify the effects of impurities on corrosion of stainless steels in lithium, (2) to study the effect of impurity-gettering elements added to the lithium in reducing corrosion, and (3) to determine if *in-situ* coating of stainless steels with a diffusion barrier will effectively inhibit corrosion. Tests were conducted in type 316 stainless steel capsules at 500, 600, and 700°C for times up to 10,000 hr.

Stainless steel specimens gained weight during exposure to lithium containing additions of silicon, manganese, cobalt, aluminum, and carbon. With the possible exception of carbon, all the above additions show potential for providing a diffusion barrier to inhibit dissolution of alloying constituents of the stainless steels by lithium under these conditions. Calcium and yttrium effectively gettered nitrogen from lithium in these tests.

3.3 DETERMINATION OF OXYGEN IN LITHIUM² — J. E. Strain³ and J. H. DeVan

In 1969 the Metals and Ceramics Division of ORNL established a 14-MeV neutron activation analysis facility to determine oxygen in reactive metals, especially lithium and potassium. Using this facility, we conducted a cooperative study with R. M. Yonco and V. A. Maroni of Argonne National Laboratory (ANL) to measure the solubility of lithium oxide in liquid lithium. Aliquots of saturated lithium-oxygen solutions were drawn into thin-walled nickel capsules by the ANL workers, and the individual capsules were analyzed for total oxygen content at ORNL. A total of 28 solubility determinations were made, covering the temperature range 195 to 734°C. The dependence of solubility S (mol % Li_2O) on temperature was of the form

$$\ln S = 6.6105 - 7140.2/T, \quad (1)$$

where T is the Kelvin temperature.

3.4 THE REDUCTION OF Al_2O_3 IN NIOBIUM-LITHIUM SYSTEMS AT 1000°C (Ref. 4) — J. E. Selle and J. H. DeVar

Various grades of Al_2O_3 were sealed inside capsules of niobium and Nb-1% Zr and exposed to liquid lithium for 3000 hr at 1000°C. Similar but unsealed capsules were tested under vacuum. The Al_2O_3 was reduced in the lithium-containing capsules but not in the vacuum capsules. Metallography and electron microprobe analysis showed reaction products in the form of compounds of niobium, aluminum, and zirconium. Lithium acted as a sink for oxygen.

²Abstracted from J. E. Strain and J. H. DeVan, "Determination of Oxygen in Reactive Materials by 14-MeV Neutron Activation Analysis," *Trans. Am. Nucl. Soc.* 26(TANSAO 26): 167 (June 1977).

³Analytical Chemistry Division.

⁴Abstracted from ORNL/TM-5828 (in press).

3.5 FUSED SALT AND HYDROXIDE COMPATIBILITY STUDIES — J. R. Keiser

Using three thermal convection loops, we have been studying the compatibility of three nonmetallic coolants with type 316 stainless steel and Hastelloy N. The operating parameters and loop and coolant compositions are given in Table 3.2. Tabular corrosion specimens contained in each loop were periodically weighed to calculate the corrosion rate for each system.

Loop 25, which contained NaOH and KOH in the eutectic ratio, developed a major leak after only eight weeks of operation. The failure occurred in a tube-to-plate weld, but extensive internal cracking was observed in all the hotter sections of Hastelloy N tubing. During the first 500 hr exposure, corrosion rates for type 316 stainless steel in the hydroxides were quite high, reaching $-19 \mu\text{g}/\text{m}^2 \text{ s}$ ($-59 \text{ mg}/\text{cm}^2\text{-year}$) at the 430°C position. Weight increases of up to $3.2 \mu\text{g}/\text{m}^2 \text{ s}$ ($10 \text{ mg}/\text{cm}^2\text{-year}$) were measured at the 365°C position. From the cracking of the Hastelloy N and the measured corrosion rates for type 316 stainless steel, it appears that molten hydroxides could not be considered for fusion reactor applications without some means of corrosion inhibition.

In contrast, corrosion in the nitrate-nitrite mixture of loop 28 has been relatively low. The loop was operated with a maximum temperature of 430°C for the first 3000 hr of specimen exposure, and a maximum corrosion rate of $-5.20 \mu\text{g}/\text{m}^2 \text{ s}$ ($-16.4 \text{ mg}/\text{cm}^2\text{-year}$) was measured during the first 1000 hr. Corrosion during the following 2000 hr was negligible. After the maximum temperature was raised to 505°C , the maximum corrosion rate observed in the hottest section of the loop was $-2.53 \mu\text{g}/\text{m}^2 \text{ s}$ ($-8.0 \text{ mg}/\text{cm}^2\text{-year}$). The chromium concentration of the salt has been gradually increasing, indicating removal of chromium from the stainless steel in the hot section of the loop. The weight change results indicate that both type 316 stainless steel and Hastelloy N can satisfactorily contain nitrite-nitrate mixtures over an extended period.

Corrosion rates of type 316 stainless steel in the LiF-BeF_2 salt have been relatively low over nearly 10,000 hr of specimen exposure

Table 3.2. Status of Fused Salt and Hydroxide Thermal Convection Loop Tests

Loop	Loop Material	Specimen Material	Coolant Composition	Cover Gas	Operating Temperatures, °C		Operating Time (hr) as of 6/1/77
					max	min	
25	Hastelloy N ^a	Type 316 stainless, 12 specimens; Hastelloy N, 4 specimens	NaOH-KOH (56-44 mol %)	H ₂	440	365	1,331
28	Hastelloy N ^a	Type 316 stainless, 12 specimens; Hastelloy N, 4 specimens	KNO ₃ -NaNO ₂ -NaNO ₃ (53-40-7 wt %)	N ₂	430 505	365 ^b 425 ^c	7,018
31	Type 316 stainless steel ^d	Type 316 stainless, 16 specimens	LiF-BeF ₂ (66-34 mol %)	Ar	650	505	12,455

^aNi-16 wt % Mo-7 wt % Cr.

^bFirst 4000 hr.

^cSubsequent period.

^dFe-18 wt % Cr-10 wt % Ni-2 wt % Mo.

time. At 650°C, initial corrosion rates were as high as $-2.53 \mu\text{g}/\text{m}^2 \text{ s}$ ($-8.0 \text{ mg}/\text{cm}^2\text{-year}$), but subsequent rates have generally been $-0.95 \mu\text{g}/\text{m}^2 \text{ s}$ ($-3.0 \text{ mg}/\text{cm}^2\text{-year}$) or less. Studies have shown that addition of the reductant beryllium to the salt nearly eliminates corrosion as long as the beryllium remains in the salt. The reductant may be required to counteract the long-term effects of transmutation products associated with fusion reactor applications.

4. MATERIALS ENGINEERING

K. C. Liu

The objective of this program is to identify materials engineering problem areas and needs as well as to assess the practical implementation of existing design rules and criteria to the design analysis for a magnetic fusion power reactor. Specific and basic materials data needs are then to be defined for the development of a comprehensive materials test plan to provide essential information in support of MFE research and development effort.

4.1 ANALYSIS OF DESIGN CODES AND CRITERIA AND IDENTIFICATION OF MATERIALS ENGINEERING AND DEVELOPMENT NEEDS OF MAJOR MFE DEVICES

Conceptual design studies of EPR and the demonstration fusion power reactor by ORNL, ANL, GA, and others have implied that these reactor systems will have their own unique challenges as well as many common difficult problems, because of the extreme in operating temperatures, stress loading, and hostile environments. Severe thermal stresses will certainly occur in components of these reactor systems, as earlier analysis studies^{1,2} on some models of first-wall and blanket systems have pointed out. Changes in material characteristics and increasing stresses resulting from radiation swelling may play an even more significant role in fusion power feasibility and economy.³ Design rules and criteria appropriate for fusion reactor design and analysis use are not as yet developed. Therefore, many designs follow available technologies and rely upon the best engineering judgment in interpreting available materials data obtained from systems involving fission.

¹A. P. Fraas and A. S. Thompson, *ORNL Demonstration Reactor Study: Fluid Flow, Heat Transfer, and Stress Analysis Consideration in the Design of Blankets for Full-Scale Fusion Reactors*, ORNL/TM Report (draft), Oak Ridge National Laboratory, Oak Ridge, Tennessee (Sept. 30, 1976).

²G. M. Fuller et al., "Fusion Reactor First Wall/Blanket System Analysis," McDonnell Douglas Astronautics Company, presented at ERDA Headquarters, June 28, 1976.

³S. Harkness, "Definition of DMFE Alloy Development Property Goals," ANL, presented to ERDA/DMFE, May 9, 1977.

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In reviewing reports aimed at conceptual design, we have found that many results are based on the analysis of a particular first-wall model.^{3,4} Differences in modeling and changes in loading and/or temperature could lead to significant differences in stress distribution and magnitude. This could in turn predict markedly different deformation and failure behavior of the first wall. Although many aspects of the analyses are germane to different conceptual first-wall designs, specific conclusions as to the service life of all first walls cannot be drawn in general from the result of current analyses. Care should be exercised in evaluating these conclusions.

We have initiated an analysis project to study thermal fatigue problems in fusion devices. Every concept of a fusion device points to thermal and cyclic fatigue crack propagation as major threats to the structural integrity of fusion power reactors. Our primary concern is the adequacy of applying to fusion existing design rules and criteria that are being used in design of nuclear components for fission. Scope of the planned project has been tentatively identified as follows.

1. Review of typical thermal stress problems in first-wall and blanket systems. The origins and characteristics of thermal stresses in typical first-wall and blanket systems are to be examined. To facilitate the discussions a few typical models such as tubular panel, rectangular block module, spherical cap module, which are being considered in current design practice, will be used. A detailed analysis of the ORNL demonstration first-wall and blanket module will be given. Factors that contribute to stresses in the structures are to be analyzed separately and the results summarized.

2. Fatigue behavior of structural materials. An assessment of time-independent (elastic and plastic) and time-dependent (creep) fatigue of structural materials that are pertinent to fusion reactors will be summarized. The effort will be extended into the areas of crack growth characteristics. Factors, such as environment and irradiation, that influence the behavior will be included.

⁴B. A. Cramer, "Material Property Requirements for Long-Life First Wall Structures," McDonnell Douglas Astronautics Company, presented to ERDA/DMFE, May 9, 1977.

3. Rules and criteria for analyzing fatigue requirements — existing methods and development needs. Adequacy and validity of existing rules and methods for designing systems subjected to thermal fatigue will be examined. Results of the analysis obtained in item 1 will be used as a basis in analyzing fatigue requirements by existing code methods and by the methods currently being under development.

4. Effects of analysis techniques and assumptions on stress calculation and its impact on fatigue life prediction. Results of structural analysis are often influenced by the method of modeling and assumptions employed in the analysis. The thermal stresses in first-wall and blanket structures are highly dependent on the boundary constraints and the flexibility of mutually interacting components. Problems of thermal buckling and inelastic behavior will be examined and considered in fatigue life prediction.

5. Availability and needs of design data for fatigue analysis. Fatigue tests and creep-fatigue tests in the laboratory are generally conducted under simple loading conditions, and the durations are very short compared with expected operating times for reactor system components. Thus, large extrapolations are required to make the data useful in design analysis. To facilitate a good fatigue analysis valid for the conditions of a fusion reactor, thermal fatigue tests simulating actual operating duty conditions and cycles would be most meaningful. Thus cross correlation with conventional fatigue data could be obtained. Availability and needs of design data for fracture mechanics and crack growth behavior will be examined.

6. Method of reducing thermal stresses in first-wall and blanket systems. Methods of reducing thermal stresses in first-wall and blanket systems at operating temperatures will be explored in this item. In principle the technique is to introduce residual stresses in the structure when it is cold. The residual stresses are relieved by thermal stresses and the state of stresses at the operating condition can be minimized.

7. Summary. A summary and recommendations will be made and published in a topical report.

4.2 COORDINATION OF PREPARATION OF MATERIAL DEVELOPMENT AND ENGINEERING TEST PLAN IN SUPPORT OF MAJOR MFE DEVICES

While at the Brookhaven Workshop for Superconducting Magnet Structural Design Analysis, contacts were established to set up several working groups to serve the needs of the magnet design community. Since then we have proposed to the organizer (BNL) agenda for three working groups on Mechanical Properties, Design Methods, and Nondestructive Testing.

5. MAGNET DEVELOPMENT

5.1 STRESS EFFECTS ON SUPERCONDUCTORS — D. S. Easton

Studies to evaluate the effects of stress on the superconducting and mechanical properties of both multifilamentary composite and tape conductors have continued. To determine what parameters are important in these effects, a cooperative effort has been initiated with AERE-Harwell, Intermagnetic General Corporation (IGC), and Sumitomo Electric Industries, Ltd., Japan, whereby they will supply samples to ORNL for study. Harwell and IGC are providing Nb_3Sn conductors and Sumitomo has supplied three different V_3Ga conductors.

The Harwell materials were received in the unreacted state (niobium filaments in a bronze matrix) and contain 1369 filaments. They vary as to the niobium content and have both twisted and untwisted filaments. Samples have been heat-treated for: (a) 600°C for 162 hr, (b) 700°C for 24 hr, and (c) 700°C for 136 hr. The resulting microstructures are shown in Fig. 5.1. The average diameter of the filament was 6 μm and the thicknesses of the Nb_3Sn layers formed after reaction were: (a) ~0.8 μm , (b) 1.0 μm , and (c) 2.0 μm .

A scanning electron micrograph of the fracture (4.2 K) surface of a single filament obtained by chemically removing the matrix is shown in Fig. 5.2. The unreacted niobium core has failed in a ductile manner, while the outer layer of Nb_3Sn has fractured brittly.

Tensile tests at 4.2 K show that samples with about 2.0- μm layers failed in a brittle manner at 1 to 1.3% strain, while samples with the thinner layers showed serrated yield behavior and failure strains up to 10%. All samples had ultimate tensile strengths at 4.2 K of approximately 800 MPa.

Samples strained to 0.8% showed intermittent cracking of the Nb_3Sn layer (Fig. 5.3). These cracks would undoubtedly result in irreversible degradation of the critical current density, J_c . The J_c of two monolithic conductors (Airco), identical with the exception that one had twisted and the other nontwisted filaments, were affected differently by stress (Fig. 5.4). The nontwisted filament conductor showed degradation at

Fig. 5.1. Cross Sections of Harwell Multifilamentary Conductors After Various Heat Treatments. 1000 $\times$. (a) 600°C at 162 hr. (b) 700°C at 24 hr. (c) 700°C at 156 hr.

Fig. 5.2. Ductile Fracture in Niobium Core Surrounded by Nb₃Sn with More Brittle Fracture in Single Filament Fractured at 4.2 K.

Fig. 5.3. Superconducting Material Strained 0.8% at 4.2 K After 156-hr Heat Treatment at 700°C. Intermittent cracks in the superconducting layer will result in irreversible degradation in current density.

Fig. 5.4. Comparison of Degradation of Critical Current Density in Otherwise Identical Twisted and Nontwisted Superconducting Filaments.

much lower stresses than did the twisted version. In both samples the degradation was reversible up to approximately 200 MPa.

Voltage-current ($V-I$) curves for a variety of materials are shown in Fig. 5.5. The curves were taken at zero stress and in a magnetic field of 7 T. Some material shows zero voltage (all current carried in the superconductor) until it suddenly transforms to the normal state. Other material shows a voltage (current carried by both matrix and superconductor) even at very low currents, indicating a poor transfer of current from the matrix to superconductor. This could result in some I^2R heating in large applications and at joints.

Stress-induced heating of a Nb-Ti-Cu composite (Fig. 5.6) occurs upon both initial stress loading and cyclic loading. The upper curve in Fig. 5.6 illustrates the case of initial loading. The open circles

Fig. 5.5. Characteristic Voltage-Current Curves for Various Conductors. The need to standardize on a criterion for determining the critical current is clearly illustrated.

Fig. 5.6. Energy Generated by Both Initial and Cyclic Loading of Multifilamentary Composites of Nb-Ti Alloy and Copper. The initial loading shows appreciable stored energy by difference between the mechanical input and the thermal response. The corresponding stored energy on cyclic loading is negligible.

are calculated from areas under a stress-strain curve, while the closed circles are obtained from experimentally observed temperature rises when the sample was loaded to some stress level for the first time. The values from stress-strain curves lie above those obtained by calorimetric measurements. This difference indicates a large amount of stored energy, either as energy of cold work in the copper matrix, or as elastic strain energy in the Nb-Ti filaments. Cyclic loaded values (lower curve) show agreement between calorimetric and stress-strain determinations. Thus all the energy loss from cyclic loading is converted into heat. This work or energy loss could be a significant factor in the operation of large magnetic devices.

5.2 RADIATION EFFECTS ON INSULATOR MATERIALS — C. J. Long and R. H. Kernohan¹

During FY 1977, a small program was begun to assess the effect of radiation on insulators for superconducting magnets. The uniqueness of the program lies in irradiation at liquid helium temperatures (4 K) and in the attempt to perform as many measurements as possible *in situ*. It draws upon personnel of Metals and Ceramics and Solid State Divisions, design input from the Superconducting Magnet Development Program of the Fusion Energy Division, and facilities provided by Solid State.

Over the past several years, a number of reports have noted that the component in a superconducting magnet with the least radiation resistance is the organic or organic-inorganic composite electrical insulation and that, furthermore, the radiation limits of the best known organics at higher temperatures (77–280 K) are comparable to the radiation fluences expected at the plasma side of the toroidal field coils in EPR or commercial tokamak reactors. Because the temperature is significantly below any for which previous measurements on similar materials had been made, extrapolations were considered unreliable.

A program was planned to use the existing 4-K Low-Temperature Irradiation Facility (LTIF) at the ORNL Bulk Shielding Reactor.

¹Solid State Division.

Materials chosen were drawn from those currently or potentially used in superconducting magnets that gave promise of adequate radiation resistance. Glass-fiber reinforced organics, especially epoxies, seem best, but a variety of special-purpose materials such as films and coatings may also be tested.

Testing methods are limited by the fact that the cold space is a cylinder 200 mm high and 25 mm in diameter at the bottom of a 5.5-m tube also 25 mm in diameter. The philosophy adopted has been to use miniature specimens for a variety of electrical (breakdown and resistivity) and mechanical (shear and bending) tests to screen candidate materials. Apparatus is available for measuring mechanical shear, although some modifications had to be made for handling mini-specimens. Similarly, flexure testing equipment had to be adapted for use with small specimens. Apparatus was acquired and a test stand designed and built for measuring volume resistivity of insulators (up to $10^{12} \Omega \text{ m}$). Some preliminary testing of samples has been made with the above equipment. Methods for consistent electrical breakdown tests are still under development.

The radiation capsule, feedthrough, and associated equipment have been designed and are being fabricated.

We contemplate that electrical resistivity will be tested at 4 K at a reactor power level that will approximately duplicate radiation conditions in the toroidal field coil of an operating fusion machine. However reactor power will then be increased by a factor of 10^4 so that several days of operation at this level will simulate 20 to 30 years of operation. Periodic power shutdowns and warmups during the several days of irradiation will simulate more nearly the probable radiation and temperature history of the toroidal field coils in an actual operating fusion machine.

Knowledge gained in this preliminary experiment may point the way to more meaningful and sophisticated tests in the future. Also, the relative values of the various tests will become more evident.

6. NEUTRONICS CALCULATIONS FOR RADIATION DAMAGE STUDIES

T. A. Gabriel,¹ R. T. Santoro,¹ B. L. Bishop,¹ and F. W. Wiffen

During the past year the primary knock-on atom (PKA) data base RECOIL,² derived from ENDF/B-IV data and composed of most elements of interest to the magnetic fusion energy (MFE) community, has been used extensively to generate radiation damage information in metals (i.e., gas production and atom displacements) for many different neutron environments, including those currently available (ORR, HFIR, ORIC *d*-Be source, EBR-II, CP-5, etc.) and those envisioned for the future (fusion reactors, *d*-Li, etc.). The primary purposes of these neutronics calculations are to aid in the planning of experiments and in the interpretation of experimental data and to assist experimentalists in deciding which currently available neutron environments can best reproduce the damage anticipated in future fusion reactors.

Up to now, existing neutronics calculations available for fusion reactor spectra have mainly been performed for the constituent materials in conceptual point-form designs. Results from different designs are not directly comparable because of different approaches used in performing the calculations. One set of calculations is available,³ comparing the response of several alloys of potential interest in a single fusion reactor spectrum. However, the results are not presented in a form to allow evaluation of the effect of alloy modification, nor are they based on the latest available neutron cross-section data. Calculations have been carried out⁴ to present a self-consistent set of calculations of

¹Neutron Physics Division.

²T. A. Gabriel, J. D. Amburgey, and N. M. Greene, "Radiation-Damage Calculations: Primary Knock-on Atom Spectra, Displacement Rates, and Gas Production," *Nucl. Sci. Eng.* 61: 21-32 (1976).

³G. L. Kulcinski, D. G. Doran, and M. A. Abdou, "Comparison of Displacement and Gas Production Rates in Current Fission and Future Fusion Reactors," p. 329 in *Properties of Reactor Structural Alloys After Neutron or Particle Irradiation*, ASTM STP 570, ASTM, 1975.

⁴T. A. Gabriel, B. L. Bishop, and F. W. Wiffen, *Calculated Irradiation Response of Materials Using a Fusion Reactor First-Wall Neutron Spectrum*, ORNL/TM-5956 (June 1977).

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the neutronic response of a number of elements and alloys to a projected fusion reactor first-wall neutron spectrum.⁵ Some of the results are given in Tables 6.1 and 6.2. Shown in Table 6.1 is the displacement and gas production rates in typical candidate alloys at the first wall of a fusion reactor. The total displacement and gas production in typical candidate first-wall alloys for a fusion reactor operation of 40 MW years/m² is given in Table 6.2. The sensitivity to the nuclear data used and to the exact neutron spectrum is found to be the greatest in the helium production rates.

Results calculated by RECOIL were used to obtain the expected increase in radiation damage in the superconducting magnets due to penetrations (injector tubes, diagnostic ports, etc.) in the shield of a tokamak fusion reactor.⁶ The geometry of the reactor as well as the penetrations and locations of the positions where the damage is calculated is shown in Fig. 6.1. Adjoint Monte Carlo calculational techniques were employed. The calculated damage and the increase in damage due to the penetration are given in Table 6.3. All results are for a wall loading of 1 MW/m². As can be seen, substantial increase in the damage due to streaming can be expected, and additional shielding will be necessary.

Since the calculated results depend on the data contained in RECOIL, a constant assessment of these data is necessary. Sensitivity calculations on some of the data contained in the RECOIL data base are in progress to ascertain what data should be improved. This analysis includes the sensitivity of the group average cross sections (damage and gas) to different in-group weight schemes and the sensitivity of some of the damage energy partial cross sections to different angular distributions. Calculations have been carried out for aluminum using four different weighting schemes:

⁵R. T. Santoro, V. C. Baker, and J. M. Barnes *Neutronics and Photonics Calculations for the Tokamak Experimental Power Reactor*, ORNL/TM-5466 (March 1977).

⁶R. T. Santoro, J. S. Tang, R. G. Alsmiller, Jr., and J. M. Barnes, *Monte Carlo Analysis of the Effects of a Blanket-Shield Penetration on the Performance of a Tokamak Fusion Reactor*, ORNL/TM-5710 (in press).

Table 6.1. Displacement and Gas-Production Rates in Typical Candidate Alloys at the First Wall of a Fusion Reactor^a

Alloy	Effective Displacement Threshold Energy (eV)	Displacement Damage (dpa/s)	Gas-Production Rate, at. ppm/s	
			Helium	Hydrogen
316 SS ^b	40	3.64×10^{-7}	45.6×10^{-7}	$169. \times 10^{-7}$
FE-16 ^c	40	3.72	76.1	248.
Al	25	4.63	101.	94.5
V-20% Ti	40	3.65	18.7	72.5
Nb	60	2.31	9.18	33.3
Mo	60	2.38	d	d

^aNeutron wall loading = 1 MW/m².^bFe-19.4 at. % Cr-12.4 at. % Ni-1.4 at. % Mo.^cNi-35.3 at. % Fe-18.1 at. % Cr-2.0 at. % Mo.^dNo ENDF/B-IV data on gas production.Table 6.2. Total Displacement and Gas Production in Typical Candidate First-Wall Alloys for a Fusion Reactor
Operation of 40 MW year/m²

Alloy	Effective Displacement Threshold Energy (eV)	Displacement Damage (dpa)	Production, at. ppm	
			Helium	Hydrogen
316 SS ^a	40	460	5,750	21,300
FE-16 ^b	40	470	9,590	31,200
Al	25	580	12,700	11,900
V-20% Ti	40	460	2,360	9,140
Nb	60	290	1,160	4,200
Mo	60	300	(1,890) ^c	(3,780) ^c

^aFe-19.4 at. % Cr-12.4 at. % Ni-1.4 at. % Mo.^bNi-35.3 at. % Fe-18.1 at. % Cr-2.0 at. % Mo.^cThere is no ENDF/B-IV data on gas production in molybdenum. These numbers are taken from Kulcinski, Doran, and Abdou and are based on calculated cross sections and on a slightly different first-wall neutron spectrum.

NOTE: DIMENSIONS IN m

Fig. 6.1. Diagram of Tokamak Reactor Showing Locations for Damage Calculations.

Table 6.3. Radiation Damage Rates at Various Locations in Fusion Reactor
Magnet Coils (Neutron Wall Loading = 1 MW/m²)

Material	Displacement Threshold Energy (eV)	Damage Rate, dpa/year or at. ppm/year for Each Detector Position					Factor Increase Due to Penetrations for Each Detector Position				
		1	2	3	4	5	1	2	3	4	5
<u>Atomic Displacement Rate</u>											
316 SS	40	3.4×10^{-4}	1.9×10^{-4}	7.4×10^{-3}	8.2×10^{-3}	1.0×10^{-2}	95	5	26	20	35
Nb	60	3.0×10^{-4}	1.7×10^{-4}	9.2×10^{-3}	8.6×10^{-3}	1.3×10^{-2}	66	3	23	15	32
Cu	30	4.2×10^{-4}	2.4×10^{-4}	1.1×10^{-2}	1.2×10^{-2}	1.3×10^{-2}	60	4	26	18	32
Al	25	3.7×10^{-4}	1.9×10^{-4}	1.9×10^{-2}	1.1×10^{-2}	1.9×10^{-2}	36	2	30	26	31
<u>Hydrogen Gas Production Rate</u>											
316 SS	40	1.5×10^{-2}	9.4×10^{-3}	7.2×10^{-2}	4.1×10^{-1}	6.6×10^{-2}	4862	126	36	94	15
Nb	60	1.4×10^{-2}	2.2×10^{-3}	1.4×10^{-2}	9.2×10^{-2}	8.5×10^{-3}	5666	110	40	141	13
Cu	30	1.7×10^{-2}	1.0×10^{-2}	8.6×10^{-2}	4.3×10^{-1}	1.1×10^{-1}	4250	100	41	100	27
Al	25	6.7×10^{-3}	4.3×10^{-3}	2.9×10^{-2}	2.0×10^{-1}	2.0×10^{-2}	4466	102	32	111	11
<u>Helium Gas Production Rate</u>											
316 SS	40	4.8×10^{-3}	3.0×10^{-3}	2.0×10^{-2}	1.3×10^{-1}	1.2×10^{-2}	4336	150	64	173	16
Nb	60	9.6×10^{-4}	6.0×10^{-4}	4.1×10^{-3}	2.4×10^{-2}	2.3×10^{-3}	7384	200	54	160	15
Cu	30	2.8×10^{-3}	1.8×10^{-3}	1.2×10^{-2}	7.9×10^{-2}	7.7×10^{-3}	6222	120	40	158	15
Al	25	1.0×10^{-2}	6.4×10^{-3}	4.4×10^{-2}	2.8×10^{-1}	2.8×10^{-2}	6250	160	55	155	16

1. $1/E$ -Maxwellian,
2. Constant-Maxwellian,
3. Fission-Maxwellian,
4. $1/E\sigma_T$ -Maxwellian.

Weighting scheme 1 is the standard weighting used to create RECOIL. Only for $1/E\sigma_T$ -Maxwellian weighting are there any significant differences in the results obtained. Some of the results are summarized in Table 6.4, which gives the change relative to the standard.

Table 6.4. Changes in Deposited Damage Energy in Aluminum Due to the Different Group Weighting Schemes

Type of Neutron Environment	Change Relative to Deposited Damage Energy Obtained Using Standard Cross-Section Set, Σ , for Each Type of Weighting		
	2	3	4
First wall of fusion reactor	0.0	0.0	-2.6
HFIR PTP ^a ($z = 0$)	0.0	0.0	-3.5
HFIR PTP ^a ($z = 0.20$ m)	0.0	0.0	-3.8
Fast reactor	0.0	0.0	-6.5

^aPeripheral target position.

In ENDFB/4 there have been improvements in the secondary neutron spectra for inelastic unresolved reactions for several elements (Fe, Cr, Co, etc.). These neutrons distributions are presently much harder in energy than those contained in ENDFB/3. For damage cross section calculations up to now^{2, 7-9} it has been assumed that for inelastic

²J. D. Jenkins, *RICE: A Program to Calculate Primary Recoil Atom Spectra from ENDF/B Data*, ORNL/TM-2706 (February 1970).

⁹D. G. Doran, "Neutron Displacement Cross Sections for Stainless Steel and Tantalum Based on a Lindhard Model," *Nucl. Sci. Eng.* 49: 130 (1972).

⁹D. M. Parkin and A. N. Goland, *A Computational Method for the Evaluation of Radiation Effects Produced by CTR-Related Neutron Spectra*, BNL-50434 (1974).

unresolved reactions isotropic neutron emission occurs. Given the harder neutron distributions and isotropic emission, the inelastic damage energy cross sections obtained with ENDFB/4 data are somewhat larger (~10-20%) than one would obtain from ENDFB/3 data. Due to the uncertainties associated with these cross sections, calculations are in progress to determine the degree of sensitivity of inelastic damage energy cross sections to different angular distributions. If the sensitivity is large enough, additional differential experimental data will be needed to refine the calculational techniques.