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ORNL-5189

**Magnetic Fusion Energy Materials
Technology Program Annual Progress
Report for Period Ending June 30, 1976**

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OAK RIDGE NATIONAL LABORATORY

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Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
Price: Printed Copy \$5.00; Microfiche \$2.25

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ORNL-5189
Distribution
Category UC-20

Contract No. 4-7405-eng-26

METALS AND CERAMICS DIVISION

MAGNETIC FUSION ENERGY MATERIALS TECHNOLOGY PROGRAM ANNUAL
PROGRESS REPORT FOR PERIOD ENDING JUNE 30, 1976

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SEPTEMBER 1976

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Oak Ridge, Tennessee 37830
operated by
UNION CARBIDE CORPORATION
for the
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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FOREWORD

The objectives of the Magnetic Fusion Energy (MFE) Program in the Metals and Ceramics Division, which is described in this report, are to continue to solve the materials problems of the Thermonuclear Division of ORNL and to meet the needs of the national MFE program, directed by the ERDA Division of Magnetic Fusion Energy. This work is a continuation of the program described in the previous annual progress report.¹ The principal areas of work include radiation effects, surface studies, compatibility studies, and support for the superconducting magnet development program.

During the past year, the program emphasis has changed from a long-term base technology to some specific problems identified by the three national Experimental Power Reactor (EPR) studies.²⁻⁴ One direct result is more emphasis on austenitic stainless steels and nickel-base alloys as the structural material for the blanket. In addition, these studies identified a multitude of critical materials problems that are not yet being addressed and instilled a needed sense of urgency in the program. At the same time, long-term material studies are needed, such as the effects of radiation on aluminum alloys and refractory metals proposed for various demonstration reactor designs,⁵⁻⁹ so this work has continued.

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

²M. Roberts et al., *Oak Ridge Tokamak Experimental Reactor Study - Reference Design*, ORNL-TM-5042 (November 1975).

³W. M. Stacey et al., *Tokamak Experimental Power Reactor Studies*, ANL/CTR-75-2 (June 1975).

⁴C. C. Baker et al., *Experimental Power Reactor Design Study*, GA-A-33534 (July 1975).

⁵A. P. Fraas, *Conceptual Design of the Blanket and Shield Region and Related Systems for a Full Scale Toroidal Fusion Reactor*, ORNL-TM-3086 (May 1973).

⁶R. G. Mills et al., *A Fusion Power Plant*, MATT-1050 (August 1974).

⁷B. Badger et al., *UWMAK I, A Wisconsin Toroidal Fusion Reactor Design*, UWFD-68, Vols. I and II (March 1974 and May 1975).

⁸B. Badger et al., *UWMAK II, A Conceptual Tokamak Power Reactor Design*, UWFD-112 (October 1975).

⁹B. Badger et al., *UWMAK III, High Performance, Noncircular Tokamak Power Reactor Design*, UWFD-150 (December 1975).

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Two premises underlie the ORNL materials program:

1. To change out the first wall of a tokamak will be a costly and difficult task in view of the levels of induced radioactivity and the size and complexity of the assembly.
2. The lifetime of the structural materials can be increased significantly by judicious control of the composition, microstructure, and operating temperatures and stress levels.

To optimize the lifetime requires extensive testing in the fusion reactor environment or one that simulates it as closely as possible, with emphasis on displacement damage, gas production, temperature, and stress levels. Equally important is a basic understanding of the phenomena governing the response of the material to the environment. Consequently, in spite of the limited budget, we have maintained the dual approach of extensive experimental testing and evaluation combined with basic studies aimed at understanding.

This report contains a summary description of the materials work done in FY 1976 for the MFE program.

SUMMARY

1. RADIATION EFFECTS

The Radiation Effects program is concerned with the irradiation performance of fusion reactor structural materials for application in the first wall and high-flux regions of the blanket. An assessment of type 316 stainless steel irradiated in the High Flux Isotope Reactor to combined displacement and helium levels equivalent to several years of fusion reactor operations was completed. On the basis of swelling and uniform strain in postirradiation tensile tests it was concluded that lifetimes of at least 5 MW-years/m² could be obtained for design temperatures not exceeding 550°C. Creep-rupture and fatigue properties or irradiation creep rates could restrict this limit, but no experimental measurements are available yet. Preliminary results showed that the addition of about 0.25% Ti to type 316 stainless steel led to enhanced ductility in postirradiation tensile tests and to increased rupture lives in postirradiation creep tests.

Initial observations of Incorel 600 irradiated in HFIR showed swelling significantly greater than expected from fast-reactor irradiations and from ion bombardment studies. The alloy PE-16 also irradiated in HFIR showed appreciable swelling but only for irradiation temperatures above 600°C. The apparent resistance of TZM to void formation found after low-fluence irradiations was eliminated at higher neutron exposures. Void formation was suppressed, however, for V-20% Ti irradiated in EBR-II to fluences of about 4×10^{26} n/m² at temperatures ranging between 440 and 780°C. An irradiation to simulate the properties of Sintered Aluminum Product (SAP) in a fusion reactor environment was completed. The material was doped with ⁶Li and irradiated in HFIR; displacement damage was produced by fast neutrons and ⁴He and ³H by the reaction of thermal neutrons with ⁶Li.

A program for studying fatigue properties of irradiated fusion reactor first-wall materials was initiated. The use of fission reactors having high thermal and fast neutron fluxes to simulate fusion reactor damage in materials containing nickel was evaluated. A limitation of this approach is that the helium is produced at an accelerating rate so that the ratio of helium to displaced atoms is appropriate to the fusion reactor case at only one time. By properly altering the core configuration of the Oak Ridge Research Reactor, it was shown that a very nearly constant ratio can be obtained that closely approximates the fusion reactor situation. A major experiment to evaluate the usefulness of fast reactors for studying radiation effects problems in fusion reactors was designed and constructed.

The primary knock-on atom (PKA) data base RECOIL, derived from ENDF/B-IV data and composed of most elements of interest to the fusion reactor community, was completed and documented. The data base was used extensively to calculate radiation damage in metals (i.e., gas production and atom displacements) for different neutron environments.

currently available (ORR, HFIR, ORIC, EBR-II, etc.) and for expected neutron environments envisioned in future fusion reactors.

Highlights of an international conference held in Gatlinburg last fall to discuss the status of radiation effects work related to fusion reactors are reported.

2. SURFACE STUDIES

Progress is reported on the use of glow discharge and ion bombardment methods for cleaning the ORMAK liner.

3. COMPATIBILITY STUDIES

Static capsule tests have been initiated and will be used for preliminary studies on the effects of additives to lithium on the corrosion rate of type 316 stainless steel. Thermal convection loops constructed of austenitic stainless steel produced a maximum corrosion rate of 14 $\mu\text{m}/\text{year}$ (0.55 mpy) ($3.7 \text{ mg}/\text{cm}^2$ in 3000 hr) at 600°C. The direct addition of beryllium metal to Li_2BeF_4 lowered the oxidation potential of the salt, resulting in a significant reduction of the corrosion rate of type 316 stainless steel. Mass transfer rates in boiling potassium systems are notably lower than in all-liquid systems, and in the absence of contamination effects, recirculating boilers have shown negligible corrosion.

4. MAGNET DEVELOPMENT

Tensile data at 4.2 K have been tabulated for 20 different superconducting multifilamentary (MF) composite conductors. Current density (J_c) was measured as a function of stress or strain for a number of Nb-Ti and Nb₃Sn MF conductors. The Nb-Ti samples show degradation in J_c less than 15% at large strains up to 2.0%. However, in composites containing Nb₃Sn filaments, values for J_c were degraded as much as 50% at strains of 0.5%. Differences in the behavior of J_c were found between monolithic and cable conductors.

We assisted in the preparation of specifications for superconducting wire. Scaling laws were derived and a relaxation program was developed for calculating eddy-current effects in the operation of thermonuclear experiments.

1. RADIATION EFFECTS

J. O. Stiegler and E. E. Bloom

The performance of early fusion reactors will undoubtedly be limited to some extent by the properties of materials. The Radiation Effects Program is directed at defining the irradiation performance limits of materials for 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 having a neutron spectrum appropriate to the fusion reactor. Until such sources become available, the program must rely on simulation techniques, fission reactor irradiations in our case. We are using such irradiations to establish acceptable operating ranges and performance limits for several classes of materials suggested for fusion reactor applications. Most of our attention is directed at the stainless steels, but we are also examining nickel- and refractory-metal-base alloys and SAP as longer range or backup materials. Since we are only simulating fusion reactor damage in our experiments, we are also studying the validity and limitations of fission reactor irradiations. In this chapter we report work directly supported by the Division of Magnetic Fusion Energy. More basic studies supported by the Division of Physical Research are reported in the Annual Progress Report for that program.

1.1 TEMPERATURE AND FLUENCE LIMITATIONS FOR A TYPE 316 STAINLESS STEEL FUSION REACTOR FIRST WALL¹

1.1.1 Purpose

Conventional iron-base alloys, such as type 316 stainless steel, are regarded as the most likely alloys for use as first walls in at least the first generation of fusion power reactors.^{2,3} This choice is based

¹Summary published in *Trans. Am. Nucl. Soc.* 22: 178 (November 1975). Full text of paper accepted for publication in *Nuclear Technology*.

²B. Badger et al., *UWMAK-I, A Wisconsin Toroidal Fusion Reactor Design*, UWFD-68, University of Wisconsin (November 1973).

³R. G. Mills, ed., *A Fusion Power Plant*, MATT-1050, Princeton University (August 1974).

on the extensive knowledge and experience with these alloys in gas, molten salt, and liquid metal coolants, well established fabricability and weldability, availability, and an extensive body of information on their response to neutron irradiation gained from the LMFBR programs.

At a neutronic wall loading of 1 MW/m^2 , the neutron flux at the first wall of a fusion reactor is $3.7 \times 10^{18} \text{ n m}^{-2} \text{ sec}^{-1}$, with about 20% of the neutrons with energies near 14 MeV. For a type 316 stainless steel first wall, about 10 displacements per atom (dpa) and 200 at. ppm He will be produced per year of operation. This is compared with about 60 dpa/year and 10 at. ppm/year neutronically generated helium in a type 316 stainless steel structural material in a mixed-oxide-core LMFBR. It has been known for some time that a few atomic ppm helium can markedly reduce the high-temperature tensile and creep-rupture ductility. This reduction of ductility, coupled with fast-neutron-induced swelling and low-temperature hardening, has led to predictions of first-wall lifetimes as short as two years.^{2,4}

The purpose of this paper is to consider recent results from irradiation experiments conducted in HFIR to assess more realistically temperature and fluence limitations on the lifetime of a type 316 stainless steel first wall than can be made on the basis of fast-reactor irradiations. Initial criteria will be swelling and uniform strain as used by Kulcinski et al.⁴ In HFIR, helium is produced by a two-step nuclear reaction with thermal neutrons: $^{58}\text{Ni}(n, \gamma)^{59}\text{Ni}$ followed by $^{59}\text{Ni}(n, \alpha)^{56}\text{Fe}$; thus, the high mixed thermal and fast flux in HFIR results in about 35 dpa and 1900 at. ppm He per year in stainless steel. This better simulates the neutron response of a fusion reactor first wall than does fast-reactor irradiation. Samples of annealed and cold-worked type 316 stainless steel have been irradiated at 380 to 785°C to damage levels⁵ up to about 60 dpa and 4200 ppm He. Swelling, tensile and

⁴G. L. Kulcinski et al., "Radiation Damage Limitations in the Design of the Wisconsin Tokamak Fusion Reactor," *Nucl. Technol.* 22: 20 (1974).

⁵Displacements per atom values are one-half of values previously reported. All dpa numbers in this paper are consistent with the method of calculation recommended by the IAEA Specialists Meeting on Radiation Damage Units for Ferritic and Stainless Steels. Helium contents are revised from earlier published work, reflecting more complete measurements.

creep-rupture properties, and microstructural changes have been reported previously.⁶⁻⁸

1.1.2 Summary of Experimental Observations

In the range 380 to 600°C, a cold-worked structure has superior swelling resistance over a solution-annealed structure. At about 60 dpa and 4200 at. ppm He, a maximum density decrease of 3.3% occurred in the 20%-cold-worked structure, compared with 8.7% in the annealed structure. At the temperature for maximum swelling in fast-reactor irradiation (550-600°C) the density decreases for both the cold-worked and the annealed alloys were significantly greater than for samples of the same heat irradiated in EBR-II under similar irradiation conditions.⁹ This observation suggests that helium content, not dpa level, controls the swelling. Above 600°C annealed and cold-worked structures swell about the same because of recovery of the cold work, and the swelling increases with temperature instead of decreasing as in a fast reactor irradiation. Swelling considerations thus limit wall temperature to about 600°C. Within this temperature limitation and the criterion of a maximum first-wall swelling of 10% at end of life,⁴ the pessimistic limitation set by our swelling results is a wall lifetime of greater than 6 years. Since this limit is based on a solution-annealed condition and correlation on the basis of dpa, less restrictive lifetimes may be predicted for cold-worked material and for correlation on the basis of helium content.

Tensile properties were altered in the same direction as for a fast reactor irradiation but to a greater extent. Samples irradiated in the

⁶F. W. Wiffen and E. E. Bloom, "Effect of High Helium Content on Stainless Steel Swelling," *Nucl. Technol.* 25: 113-23 (1975).

⁷E. 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).

⁸P. J. Maziasz, F. W. Wiffen, and E. E. Bloom, "Swelling and Microstructural Changes in Type 316 Stainless Steel Irradiated Under Simulated CTR Conditions," pp. 259-88 in *Radiation Effects and Tritium Technology for Fusion Reactors* (Proc. Intern. Conf. Gatlinburg, Tenn., Oct. 1-3, 1975), Vol. I, ed. by J. S. Watson and F. W. Wiffen, CONF-750989.

⁹P. S. Sklad and E. E. Bloom, ORNL, unpublished results.

annealed condition to about 50 dpa and 3500 ppm He showed an increased yield strength between 350 and 600°C and, except at 350°C, a reduced ultimate tensile strength compared with the unirradiated condition. In the 20%-cold-worked condition yield and ultimate tensile strengths decreased at all temperatures. Both the annealed and cold-worked material exhibited little strain hardening following irradiation. Below 450°C the fracture strain is usually controlled by plastic instability, which is in turn related to the work hardening behavior. For annealed materials, irradiation decreases the work hardening coefficient and thus the uniform strain. In type 304 stainless steel irradiated in the EBR-II this effect saturates at about 70 dpa at 0.5% uniform strain and 2.5% total elongation.¹⁰ These values are consistent with type 316 stainless steel irradiated in HFIR to very high helium contents. Thus, we conclude that for temperatures below 450°C the mechanical behavior of stainless steel at high dpa and high helium contents is related primarily to hardening phenomena. The uniform strain will not drop below about 0.5% and should not be a limiting property.

With increasing temperature the effects of helium become more pronounced. At 500°C, 0.7% uniform strain was retained after 60 dpa and 4000 at. ppm He. At 575°C the uniform and total elongations dropped below 0.5% in the interval between 28 dpa, 1700 at. ppm He and 60 dpa, 4000 at. ppm He. At 650°C both the uniform and total elongations were zero for irradiations to about 60 dpa and 4200 ppm He. Using the criterion of 0.5% residual uniform elongation^b a first-wall temperature limit of 500 to 550°C would be imposed. Fluence limits are more difficult to judge because of synergistic effects between defect hardening and helium. However, if the criterion of 0.5% uniform strain is used, 40 dpa at 550°C or 60 dpa at 500°C should be tolerable, allowing a minimum of 4 to 6 years fusion reactor operation at a wall loading of 1 MW/m² (using again the pessimistic dpa correlation).

¹⁰R. L. Fish et al., "Swelling and Tensile Property Evaluations of High-Fluence EBR-II Thimbles," p. 149 in *Effects of Radiation on Substructure and Mechanical Properties of Metals and Alloys*, Spec. Tech. Publ. 529, ed. by John Moteff, American Society for Testing and Materials, Philadelphia, 1973.

1.1.3 Conclusions

If swelling and uniform strain are used as performance criteria, 20%-cold-worked type 316 stainless steel can be used as first-wall construction materials with lifetimes of at least 5 MW-years/m² (50 dpa and 1000 at. ppm He) for design temperatures not exceeding 550°C. However, additional materials behavior certainly should be considered. At 550°C thermal creep-rupture properties are drastically reduced (rupture life reduced by 5×10^3 as a result of irradiation to 40 dpa and 3000 ppm helium).⁷ On the basis of tensile data a marked reduction in fatigue life is also expected and may limit the lifetimes of fusion devices operating in a pulsed mode. Irradiation creep rates could also be limiting but have not yet been experimentally determined.

1.2 COMPARISON OF TYPE 316 AND TITANIUM-MODIFIED TYPE 316 STAINLESS STEELS IRRADIATED IN HFIR - P. J. Maziasz and E. E. Bloom

1.2.1 Purpose

Previous studies¹¹⁻¹³ of types 304 and 316 stainless steel have shown that small (~0.25%) titanium additions substantially increase postirradiation tensile ductility and creep-rupture life after irradiation in EBR-II. A critical question in developing materials for fusion reactor applications is whether or not titanium additions continue to be beneficial under high helium production rates. Fusion reactor conditions can be simulated for materials containing nickel by irradiation in fission reactors such as HFIR having

¹¹W. R. Martin and J. R. Weir, "Solutions to the Problems of High-Temperature Irradiation Embrittlement," pp. 440-57 in *Effects of Radiation on Structural Materials*, Spec. Tech. Publ. 426, American Society for Testing and Materials, Philadelphia, 1967.

¹²E. E. Bloom and J. R. Weir, "Development of Austenitic Stainless Steels with Improved Resistance to Elevated-Temperature Irradiation Embrittlement," pp. 261-89 in *Irradiation Effects in Structural Alloys for Thermal and Fast Reactors*, Spec. Tech. Publ. 457, American Society for Testing and Materials, Philadelphia, 1969.

¹³E. E. Bloom, J. M. Leitnaker, and J. O. Stiegler, *Effect of Neutron Irradiation on the Microstructure and Properties of Titanium-Stabilized Type 316 Stainless Steel*, ORNL-TM-4731 (January 1975).

high fast and thermal neutron fluxes; displacements are produced by the fast neutrons and helium by thermal neutrons via the reaction sequence $^{58}\text{Ni}(n,\gamma)^{59}\text{Ni}$ followed by $^{59}\text{Ni}(n,\alpha)^{56}\text{Fe}$. The purpose of this work is investigation of the behavior of titanium-modified type 316 stainless steel after irradiation in HFIR under displacement and helium production rates characteristic of fusion reactors.

1.2.2 Experimental Details

Tensile specimens of standard and titanium-modified type 316 stainless steel having the composition given in Table 1.1 were irradiated in HFIR at temperatures between 535 and 785°C. Displacement levels from 3.5 to 25 dpa and helium contents from 60 to 4200 at ppm were achieved in the irradiations. Both solution annealed and 20%-cold-worked materials were irradiated. Postirradiation examination included immersion density measurements as well as tensile and creep-rupture tests at temperatures near the irradiation temperatures.

1.2.3 Results

The titanium addition had no significant effect on the swelling of either the solution annealed or 20%-cold-worked materials. Although tensile data for which meaningful comparisons can be made are very limited at present (see Table 1.2) the trends are consistent with those found in fast-reactor irradiations - namely, that ductilities are significantly enhanced in the temperature range where failure occurs by the growth of grain boundary cracks (above about 550°C). Creep-rupture data are similarly limited (Table 1.3), but they show that for tests at 550°C the titanium-modified material is significantly stronger (lower minimum creep rate, increased rupture life) and that it probably exhibits greater elongations than the standard steel.

1.2.4 Conclusions

Preliminary results indicate that small titanium additions to type 316 stainless steel are beneficial by decreasing effects of the large helium concentrations expected in fusion reactors. As in fast-reactor

Table 1.1. Composition of Standard and Titanium-Modified
Type 316 Stainless Steel

Alloy	Content, wt %										
	Cr	Ni	Mo	Mn	Si	Ti	C	N	S	P	B
Standard	18.0	13.0	2.58	1.9	0.8	0.05	0.05	0.05	0.016	0.013	0.0005
Modified	17.0	12.0	2.5	0.5	0.4	0.23	0.06	0.006	0.012	0.010	0.0007

Table 1.2. Tensile Properties of Standard and Titanium-Modified
Type 316 Stainless Steel Irradiated in HFIR

Alloy	Temperature, °C		Exposure Level		Stress, MPa (ksi)		Strain, %	
	Irradiation	Test	(dpa)	(at. ppm He)	Yield	Ultimate	Uniform	Total
<u>20%-Cold-Worked Material</u>								
Standard	600	575	3	40	509(73.9)	540(78.3)	0.5	0.5
Modified	590	575	3	40	399(57.9)	436(63.2)	3.1	5.1
Standard	575	575	3	40	415(60.3)	435(63.1)	0.5	0.5
<u>Annealed Material</u>								
Standard	580	550 ^a	45	3160	207(30.0)	241(35.0)	0.7	0.7
Modified	580	550	45	3160	336(48.7)	336(48.7)	0.3	0.3
Standard	680	650	60	4240	57 (8.3)	57 (8.3)	b	b
Modified	680	650	47	3300	170(24.7)	208(30.2)	1.0	1.0
Standard	780	750	48	3420	69(10.1)	69(10.1)	0.14	0.14
Modified	680	700	47	3300	189(27.4)	209(30.3)	1.1	1.1

^aInterpolated from tests at surrounding temperatures.

^bFailed in elastic region.

Table 1.3. Creep-Rupture Properties of Standard and Titanium-Modified Type 316 Stainless Steel After Irradiation in HFIR

Alloy	Condition	Temperature, °C		Exposure Level		Stress		Rupture Life (hr)	Creep Rate (%/hr)	Total Elongation (%)
		Irradiation	Test	(dpa)	(at. ppm He)	(MPa)	(ksi)			
Standard	Annealed	535	550	26	1930	310	45	0.5	0.1	0.1
Modified	Annealed	550	550	35	2650	310	45	1886	0.0007	1.5
Standard	202 Cu	535	550	26	1930	241	35	12.4	0.06	0.9
Modified	202 Cu	540	550	26	1936	241	35	>7000 ^a	<0.0001	>0.37

^aTest in progress

irradiations, ductility is enhanced in the temperature range where intergranular cracking leads to failure.

1.3 EVALUATION OF NICKEL-BASE ALLOYS FOR FUSION REACTOR FIRST-WALL APPLICATIONS - F. W. Wiffen

1.3.1 Introduction

Among the large number of alloys that have been considered as candidate structural materials for the first wall and other components in the high-flux regions of a fusion reactor are a number of nickel-base alloys. Interest in nickel alloys was focussed by the PPPL conceptual reactor study,¹⁴ which specified PE-16 as the structural material. In this design, the PE-16 will operate in the temperature range 250 to 660°C, both as low-stress components and as coolant tubes carrying 50 atm He. The nominal radiation response for PE-16 in the first wall of this reactor is 27 dpa and 392 at. ppm He per year. Alloy PE-16 was specified as reference material in this reactor study on the basis of its resistance to swelling relative to austenitic stainless steels, under conditions simulating LMFBR irradiation.

Previous ORNL work on type 316 stainless steel showed that swelling under irradiation that closely simulates fusion reactor conditions can be quite different from swelling under fast-reactor irradiation. The

¹⁴R. G. Mills, ed., *A Fusion Power Plant*, MATT-1050, Princeton University (August 1974).

differences are due to the greater helium production rates under fusion reactor conditions and require that results from the LMFBR programs be used with caution in the fusion program.

1.3.2 Experiment Description

Simulated fusion reactor conditions are achieved in HFIR irradiations. The high nuclear heating rate in this reactor makes it possible to attain elevated temperatures in sealed irradiation capsules. The mixed neutron energy spectrum of HFIR results in displacement rates several times higher than proposed for fusion reactors (due to HFIR's fast neutrons) and very high helium production rates due to the unique reaction sequence with thermal neutrons (see Sect. 1.1.1).

Three HFIR capsules were built to investigate radiation effects in two classes of nickel-base alloys under conditions of high helium production rates accompanying the production of displacement damage. The alloys included are PE-16 (typical of precipitation-strengthened nickel alloys) and Inconel 600 (typical of solid-solution-strengthened nickel alloys). Both alloys are reported to be swelling resistant under limited ion bombardment conditions. The goals of these experiments are (1) to investigate swelling under conditions of concurrent displacement damage and transmutation helium production and (2) to examine tensile properties of these materials as a function of irradiation and test temperatures. Samples of Inconel 600 were included in both the solution-annealed and 20%-cold-worked conditions. Alloy PE-16 was irradiated both as solution annealed and aged. Irradiation temperatures were from 300 to 700°C. HFIR irradiation for 9000 Mwd was planned to give helium contents from 300 to 2000 at. ppm and displacement levels of 4 to 9 dpa.

To measure swelling we determined length increases and changes in sample density. Evaluation of irradiation effects on the mechanical properties has begun with tensile testing at the irradiation temperature.

1.3.3 Results

Preliminary results of swelling produced by HFIR irradiation of PE-16 and Inconel 600 at temperatures from 300 to 700°C show that helium production at the rates projected for fusion reactor service leads to swelling

quite different than observed in these alloys under LMFBR conditions. These experiments have shown, for irradiation producing 4 to 9 dpa and helium contents of 350 to 1050 at. ppm in PE-16 and 620 to 1800 at. ppm in Inconel 600:

1. Inconel 600 swells in the entire temperature range studied; PE-16 shows appreciable swelling only above 600°C.
2. Swelling values much larger than expected from fast reactor and ion bombardment studies occur for several irradiation temperatures.
3. Densification is observed in PE-16 at low irradiation temperatures.
4. Solution-annealed and aged PE-16 differ little in swelling behavior.
5. Swelling of Inconel 600 is only weakly temperature dependent between 300 and 600°C, but increases with higher temperatures.
6. Swelling increases with increasing helium content in the Inconel 600.
7. Cold work does not suppress swelling of Inconel 600.

The few tensile tests completed show a general reduction in ductility produced by the irradiation, with the greatest effect observed for the highest irradiation and test temperature. At 700°C, Inconel 600 with 1260 at. ppm He was brittle, failing during the elastic portion of the tensile test at a stress only 0.2 times the yield stress of the unirradiated material.

1.3.4 Plans

Future evaluation of these experiments will include completion of tensile tests at all irradiation temperatures. Examination by electron microscopy, fractography, and metallography will be used to identify mechanisms of fracture. 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.

1.4 EVALUATION OF REFRACTORY METALS AND ALLOYS FOR FUSION REACTOR FIRST-WALL APPLICATIONS

1.4.1 Neutron-Irradiation Effects in Molybdenum and Molybdenum Alloys¹⁵ - J. Bentley and F. W. Wiffen

1.4.1.1 Purpose

The high strength of molybdenum alloys at elevated temperatures makes these alloys attractive candidates for structures in advanced fusion power systems. The strength properties would allow potential service temperatures as high as 1000°C and have led to the specification of molybdenum alloys in several conceptual reactor designs. Among the main uncertainties in the possible use of these alloys is their irradiation response in the fusion reactor environment. This paper reports microstructural effects, emphasizing swelling, for neutron irradiation at temperatures in the range of interest for fusion reactor application.

1.4.1.2 Experiment Description

Sheet specimens of Mo, Mo-0.5% Ti, and TZM alloy (Mo-0.5% Ti-0.08% Zr) were irradiated in EBR-II at 425 to 1000°C to fluences of 2.5×10^{26} n/m² (>0.1 MeV) or higher.

1.4.1.3 Results and Discussion

Limited data on the void component of the damage structure as determined by transmission electron microscopy (TEM) for the molybdenum and Mo-0.5% Ti specimens irradiated at 585 and 790°C have been reported previously.¹⁶ Although void concentrations in Mo-0.5% Ti were higher than those in molybdenum following irradiation at 585°C generally there was little other difference between the microstructures of the two materials. This behavior contrasts with the marked difference in

¹⁵Paper submitted for presentation at the Second ANS Topical Meeting on the Technology of Controlled Nuclear Fusion, Richland, Wash., Sept. 21-23, 1976.

¹⁶F. W. Wiffen, "The Effect of Alloying and Purity on the Formation and Ordering of Voids in BCC Metals," pp. 386-96 in *Radiation-Induced Voids in Metals*, J. W. Corbett and L. C. Ianniello, eds., AEC Symp. Ser. 26, CONF-710601 (April 1972).

microstructure between molybdenum and TZM alloy, which has been observed in material irradiated to much lower fluences. Bentley et al.¹⁷ compared molybdenum and TZM irradiated at 750 and 850°C to fluences of 1 and 3×10^{24} fission n/m² and observed an almost complete absence of voids in the alloy, while voids did form in the molybdenum. However, the damage structure in the TZM consisted mainly of a high concentration of small vacancy-type dislocation loops. The segregation of oversize alloying elements was proposed to be the mechanism for stabilizing the loops.

Examination by TEM of specimens of TZM irradiated at 790°C to 2.5×10^{26} n/m² and at ~1000°C to 4.4×10^{26} n/m² has revealed that the suppression of void formation that occurs at low fluences does not persist to these higher fluences typical of projected fusion reactor operation. The damage structures consist of a dislocation structure of networks and large loops (analyzed to be interstitial) with a high concentration of voids forming an imperfect void lattice. Quantitative data are summarized in Table 1.4.

Table 1.4. Measured Void Parameters in Two Specimens of TZM

Irradiation Temperature, °C	790	~1000
Fluence, n/m ² (>0.1 MeV)	2.5×10^{26}	4.4×10^{26}
Void Concentration, ^a m ⁻³	8.0×10^{22}	1.6×10^{22}
Mean Void Diameter, ^b nm	5.7	10.9
Void Volume Fraction, %	0.78	1.1
Void Lattice Parameter, ^c nm	32.5	50.5

^aConcentrations obtained by use of foil thicknesses measured by stereomicroscopy and thickness fringe counts.

^bDiameter of the void of mean volume.

^cAs measured on micrographs and also from diffraction patterns.

¹⁷J. Bentley, B. L. Eyre, and M. H. Loretto, "Suppression of Void Formation in Neutron Irradiated TZM," pp. 297-311 in *Radiation Effects and Tritium Technology for Fusion Reactors* (Proc. Intern. Conf. Gatlinburg, Tenn., Oct. 1-3, 1975), Vol. I, ed. by J. S. Watson and F. W. Wiffen, CONF-750989.

The reason for the reversion of the TZM to "normal behavior," in the sense of the formation of voids and not vacancy loops, in the present study is not clear. If the previous explanation¹⁷ was correct (i.e., segregation of alloying elements stabilized the vacancy loops), then the loops likely did not form during the present irradiations. This could be due to the higher (by an order of magnitude) neutron flux or to differences in the starting material. We think that materials variables are the more likely explanation. The vacancy loops may not be stable at high fluences, perhaps because of loop growth and coalescence to form dislocation networks.

Some qualitative differences may exist between the TZM and molybdenum damage structures. In particular, in TZM the void concentration is higher, the void size is smaller, and the dislocation density is higher than in the molybdenum. At the lower irradiation temperature in addition to dislocation segments, rafts, and ordered voids there are small isolated dislocation loops, which show some evidence of ordering onto a superlattice.

In summary, this work has shown that the suppression of void formation occurring in TZM irradiated at $\sim 800^\circ\text{C}$ to $\leq 3 \times 10^{26}$ fission nm^{-2} does not persist to higher fluences ($> 10^{26}$ n/m^2) and that there is little qualitative difference between the damage structures in TZM and molybdenum.

1.4.2 Swelling and Microstructural Changes in Irradiated Vanadium Alloys¹⁸ - J. Bentley and F. W. Wiffen

1.4.2.1 Purpose

Vanadium-base alloys are attractive for use as the first-wall structural material for fusion reactors because of (1) moderate strength, making the alloys useful to about 800°C , (2) low activation rates compared with niobium or stainless steel, thus creating much reduced problems

¹⁸ Abstract of paper submitted to *Nuclear Technology* and also presented at the American Nuclear Society Winter Meeting in November 1975 [*Trans. Am. Nucl. Soc.* 22: 176 (1975)].

with afterheat and radioactive waste management,¹⁹⁻²¹ and (3) a demonstrated resistance of vanadium-titanium alloys to irradiation-produced swelling and embrittlement.^{16,22,23} Swelling and microstructural characterization has been completed on three vanadium alloys to evaluate further the effects of neutron irradiation and their potential for use as structural materials in fusion reactors.

1.4.2.2 Experiment Description

Arc-melted 500-g ingots of V-10Z Cr and V-20Z Ti and an 18-kg ingot of VANSTAR-7 were made and reduced to rod stock by hot breakdown of stainless-steel-clad billets followed by cold swaging to 6.3 mm in diameter. Tensile specimens 47.5 mm long were machined to have button-head grips with a diameter of 6.0 mm and a gage diameter of 3.2 mm. They were given final vacuum anneals for 1 hr at 900°C (V-20Z Ti), 1100°C (V-10Z Cr), or 1200°C (VANSTAR-7) to produce fully recrystallized microstructures. Compositions are given in Table 1.5. The samples were irradiated in EBR-II. Fluences and values of displacements per atom calculated with the Linhard model (threshold displacement energy 25 eV) are given in Table 1.6.

¹⁹D. Steiner and A. P. Fraas, "Preliminary Observations on the Radiological Implications of Fusion Power," *Nucl. Safety* 13: 353-62 (September-October 1972).

²⁰D. Steiner, "Long-Lived Activities and Radioactive-Waste Management Associated with D-T Fusion Reactors," *Nucl. Fusion* 11: 307-08 (1971).

²¹F. W. Wiffen, "The Effect of Alloying and Purity on the Formation and Ordering of Voids in BCC Metals," pp. 386-96 in *Radiation-Induced Voids in Metals*, J. W. Corbett and L. C. Ianniello, eds., AEC Symp. Ser. 26, CONF-710601 (April 1972).

²²R. Carlander, S. D. Harkness, and A. T. Santhanam, "Effects of Fast-Neutron Irradiation on Tensile Properties and Swelling Behavior of Vanadium Alloys," pp. 399-414 in *Effects of Radiation on Substructure and Mechanical Properties of Metals and Alloys*, Spec. Tech. Publ. 529, ed. by John Moteff, American Society for Testing and Materials, Philadelphia, 1973.

²³J. D. Elen, "Defect Structure of Fast Neutron Irradiated Vanadium and Vanadium Titanium Alloys," pp. 351-42 in *Septième Congrès International de Microscopie Electronique* (Grenoble, 1970), Vol. II, Société Française de Microscopie Electronique, Paris, 1970.

Table 1.5. Ingot Analysis of Vanadium Alloy Specimens

Material	Alloy Additions, wt %		Impurities, wt ppm		
	Cr	Others	C	N	O
V-20% Ti		19.5 Ti	145	45	575
V-10% Cr	9.9		140	550	77
VANSTAR-7	9.1	3.3 Fe 1.2 Zr	500	90	760

Table 1.6. Conditions for EBR-II Irradiation of Vanadium Alloys

Material	Fluence, >0.1 MeV (n/cm ²)	Exposure (dpa)	Irradiation Temperature, °C		
			Nominal	Uncertainty ^a	Range During Irradiation
V-10% Cr	1.5 × 10 ²⁶	17	496	±10	
V-10% Cr	1.5	17	580	±20	
V-10% Cr	1.5	17	690	±30	
V-10% Cr	1.5	17	805	±40	
VANSTAR-7	1.5	17	496	±10	
VANSTAR-7	1.5	17	580	±20	
VANSTAR-7	1.5	17	690	±30	
VANSTAR-7	1.5	17	805	±40	
V-20% Ti	1.3	15	470	±10	
V-20% Ti	3.5	40	700		680-760
V-20% Ti	4.0	45	440		420-460
V-20% Ti	4.0	45	580		550-650
V-20% Ti	6.1	69	780		680-880

^aDue to uncertainty in nuclear heating rate at the reactor location.

1.4.2.3 Results

Swelling values were determined from the changes in density of irradiated tensile samples, measured by the immersion technique using water with a wetting agent. Transmission electron microscopy (TEM) examination of specimens sectioned from the heads of tested tensile samples was also performed.

Results and discussion of the immersion density measurements and quantitative data from TEM examination of the microstructures of the V-10% Cr and VANSTAR-7 alloys have been reported.²⁴ In summary, voids were observed in all specimens, but significant swelling (~1%) occurred in specimens of V-10% Cr irradiated at 690 and 805°C.

Results of the immersion density measurements and quantitative data from TEM examination of the V-20% Ti alloy are given in Table 1.7. Examination by TEM of specimens of V-20% Ti irradiated at all conditions shown in Table 1.6 failed to detect the presence of voids (an observation that agrees with the small²⁵ immersion density changes shown in Table 1.7). The only damage structure observed was a dislocation substructure. At all irradiation temperatures except the highest (~780°C) the dislocation structure has the form of a network, but some large resolvable loops were also present [Fig. 1.1(a) and (b)]. Values of the measured dislocation densities are given in Table 1.7. There is some evidence that the dislocation density increased with increased fluence at approximately constant irradiation temperature (440-470°C), and the results show clearly that, at nearly constant fluence ($3.5-4.0 \times 10^{26}$ n/m²), the dislocation density decreased with increased irradiation temperature. The dislocation structure in the specimen irradiated at about 780°C to

²⁴J. Bentley and F. W. Wiffen, "Swelling and Microstructural Changes in Irradiated Vanadium Alloys," *Controlled Thermonuclear Materials Technology Program Annu. Prog. Rep. June 30, 1975*, ORNL-5082, pp. 21-26.

²⁵The quoted density change of 0.3% for the specimen irradiated at 780°C is the mean of two results, one of which indicates a negative swelling. The specimen on which the TEM was performed had a density change of 0.67%, but the TEM still gave no indication of voids. For these specimens the temperatures achieved at the end of the irradiation were near 900°C, and the errors in measuring density change are probably greatly increased compared with other specimens because of impurity contamination.

Table 1.7. Measured Swelling and Dislocation Densities for V-20% Ti Irradiated in EBR-II

Irradiation Temperature (°C)	Fluence, >0.1 MeV (n/m ²)	Immersion Density Decrease (%)	Dislocation Density (m ⁻²)
470	1.3×10^{26}	a	6.2×10^{13}
440	4	0.1	40
580	4	0.04	17
700	3.5	0.01	4.1
780	6.1	0.3	3.5

^aNot determined.

^bConcentrations obtained by use of foil thicknesses measured by stereomicroscopy.

Fig. 1.1 Electron Micrographs Showing Dislocation Structures in Irradiated V-20% Ti. Diffracting conditions as shown; foil thicknesses ≈ 180 nm. Irradiation temperatures and fluences: (a) 440°C, 4×10^{26} n/m². (b) 580°C, 4×10^{26} n/m². (c) 780°C, 6.1×10^{26} n/m².

$6.1 \times 10^{26} \text{ n/m}^2$ consisted of a partially recovered structure of sub-grain boundaries with a low density of dislocation segments within the subgrains, as shown in Fig. 1.1(c). No apparent change in the distribution or morphology of the precipitates in the V-20Z Ti resulted from the irradiation except for some precipitation on the polygonized networks in the sample irradiated at about 780°C.

1.4.2.4 Discussion

A significant result of this work is the complete absence of voids in the V-20Z Ti alloy, which agrees with previous results for V-20Z Ti and other vanadium-titanium binary alloys.^{16,22,23} The mechanism of swelling suppression is not obvious, although a number have been proposed for vanadium alloys, including:

1. high dislocation densities in alloys,²⁶
2. gettering of impurity interstitial atoms by alloying elements leading to either a reduction in the number of void nucleation sites^{16,22} or higher effective surface energies,²⁶
3. the formation of high concentrations of coherent precipitates,²²
4. enhanced recombination due to the presence of substitutional solute atoms.^{22,26}

Although no direct evidence has been obtained, short-range ordering might occur and contribute. The experiments of Carlander et al.²² showed that at oxygen contents high enough that oxide precipitates were observed, voids would also form in vanadium-titanium alloys. Unfortunately this does not provide information that can be used to help eliminate any of the above mechanisms.

²⁶A. F. Barlett et al., "High Temperature Irradiation Damage Structures in Fast Reactor Irradiated Niobium and Vanadium Alloys," pp. 122-49 in *Radiation Effects and Tritium Technology for Fusion Reactors* (Proc. Intern. Conf. Gatlinburg, Tenn., Oct. 1-3, 1975), Vol. I, ed. by J. S. Watson and F. W. Wiffen, CONF-750989.

The absence of voids and the small loss in ductility²²⁻²⁷ following irradiation makes V-20% Ti appear attractive for use as a fusion reactor first-wall structural material. The evidence that voids have formed in the other two alloys makes them somewhat less attractive, although the levels of swelling achieved at the relatively low fluences are probably not technologically significant except in the V-10% Cr at the two higher temperatures.²⁴ The V-10% Cr and VANSTAR-7 also show a greater loss in ductility produced by irradiation²⁸ than does V-20% Ti. On the basis of resistance to both swelling and embrittlement, at the fluences for which data have been obtained, the V-20% Ti is superior to the other two alloys for fusion reactor application.

1.4.2.5 Conclusions

This work has confirmed the resistance to swelling of neutron-irradiated V-20% Ti and has shown that V-10% Cr and VANSTAR-7 do not have an intrinsic swelling resistance. The results show that vanadium alloys as a class cannot be regarded as swelling resistant. For the fusion reactor application, a comprehensive study of swelling would be a required part of the radiation effects evaluation within any vanadium alloy development program, paying particular attention to the helium production rates expected. The role of impurities, especially interstitial impurities, in the development of the microstructure during irradiation must be evaluated quantitatively for vanadium alloys to be developed to exploit their resistance to irradiation-produced degradation of mechanical and dimensional properties.

Of the three alloys examined, V-20% Ti possesses the superior resistance to irradiation-produced swelling. This, coupled with its superior resistance to irradiation-produced loss of ductility, gives it the greatest potential of the three alloys for application in fusion systems.

²⁷J. A. Horak and F. W. Wiffen, "Mechanical Properties of Irradiated V-20 Wt % Ti," *Controlled Thermonuclear Materials Technology Program Annu. Prog. Rep. June 30, 1975*, ORNL-5082, pp. 26-27.

²⁸F. 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. Met. 18, ed. R. J. Arsenault (Proc. 1973 Intern. Conf. Aug. 14-16, 1973), National Bureau of Standards, Gaithersburg, Md.

1.5 HFIR IRRADIATIONS OF SAP TO PRODUCE HIGH HELIUM CONTENTS — P. J. Maziasz and K. Farrell

1.5.1 Purpose

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, tests are now under way to determine the effects of gas contents typically expected during operation on the mechanical properties of SAP.

HFIR irradiation of samples doped with ^6Li has been chosen to simulate fusion reactor irradiation of SAP. During 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:

The reaction cross section is about 950 b ($9.5 \times 10^{-26} \text{ m}^2$).

1.5.2 Experimental Description and Status

Samples of SAP with from 0 to 174 wt ppm Li (96% ^6Li) have been irradiated in HFIR to neutron fluences ($>0.1 \text{ MeV}$) of 1.4 to $4.6 \times 10^{25} \text{ n/m}^2$ at ambient reactor coolant temperature ($\sim 55^\circ\text{C}$). Unirradiated tensile tests have been run at test temperatures of 20 to 500°C . Immersion density measurements have been made on the irradiated samples, and these results indicate no detectable swelling. Planned work includes annealing the samples at 400°C to coalesce the finely dispersed gas to simulate the distribution of gas produced by fusion reactor operation at an equivalent gas content. The mechanical properties of the irradiated and annealed samples will be examined, and, in particular, the creep-rupture life will be determined. Investigations of various annealing times and their effect on the gas distribution to see if a steady state has been reached are under way.

1.6 A PROGRAM FOR STUDYING FATIGUE PROPERTIES OF FUSION REACTOR FIRST-WALL MATERIALS — M. L. Grossbeck and K. Liu

1.6.1 Introduction

The pulsed operation of Tokamak machines causes cyclic stresses in structural components. In addition to severe cyclic stresses, the first wall will be subjected to helium formation as well as displacement damage. It is therefore, essential to conduct an experimental program to evaluate the effect of cyclic stresses on irradiated first-wall 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 crudely known, initial studies will employ postirradiation tests of materials irradiated in fission reactors.

1.6.2 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,²⁹ and since ductility is the most important index of resistance to low-cycle fatigue,³⁰ irradiation should reduce low-cycle fatigue life. However, defect clusters created by irradiation may impede dislocation motion, resulting in slower progression of fatigue damage. The competition

²⁹E. 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.

³⁰R. E. Peterson, "Fatigue of Metals: Engineering and Design Aspects," *Mater. Res. Stand.* 3: 122 (1963).

between these two effects, which should be temperature dependent, determines the effect of irradiation on fatigue life.

Helium can also have opposing effects on fatigue behavior. Helium bubbles or helium defect clusters may pin dislocations, resulting in lattice strengthening.^{31,32} An opposing effect may result from sweeping helium bubbles to grain boundaries³³ by dislocation motion due to cyclic stress. The presence of helium bubbles at grain boundaries reduces ductility severely in stainless steel above about 550°C.²⁹ The extent to which each of the opposing mechanisms operates in a given temperature region remains to be determined.

1.6.3 Objectives of Present Research

Because of the uncertainties in the behavior of fusion reactor first-wall materials under a periodic burn cycle, a research program on this effect must have the following objectives:

1. Determine the direction of the effect at various temperatures.
A sufficiently high fluence and helium content must be employed to emphasize the effect and make an unambiguous determination of its direction.
2. Determine the mechanism of interaction of helium with irradiated materials under cyclic stress. Before intelligent suggestions for improvement of fusion reactor first-wall materials can be made, the mechanism of failure must be understood.
3. Make proposals for improvement of alloys for fusion reactor first-wall use.

³¹C. F. Serpan, Jr., et al., "Microstructural Effects of Implanted Helium on Fatigue of 316 Stainless Steel," *Trans. Am. Nucl. Soc.* 16: 71-73 (1973).

³²D. 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).

³³D. S. Whitnell and R. S. Nelson, "The Thermal Release of Helium Injected into Nickel, Stainless Steels and Nimonic PE-16, *Radiat. Eff.* 14: 249-55 (1972).

1.6.4 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 sec.

During operation, heat is deposited in the first wall primarily by neutrons, alpha particles, and electromagnetic radiation. This 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 Wisconsin Tokamak wall stresses are considered.³⁴ 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

A more recent calculation³⁵ using finite element analysis and considering irradiation-produced swelling indicates that compressive stresses as high as 350 MPa may exist during transients.

Since the plasma side of the wall experiences full stress reversal, a stress cycle with which to test first-wall materials should be one of 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, 500, and 600°C, the same temperatures at which the other mechanical tests on irradiated fusion reactor materials will be conducted. A second series of tests will be conducted to evaluate the effect of hold time.

³⁴The University of Wisconsin Fusion Feasibility Study Group, *A Wisconsin Toroidal Fusion Reactor Design*, 1974.

³⁵J. W. Davis, McDonnell Douglas Astro. Corp., private communication, 1975.

Because the effect of hold time in fatigue tests appears to saturate,³⁶ a test cycle considerably shorter than the actual duty cycle can be justified. It is necessary to shorten the cycle in order to complete a test 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.

When fusion reactor designs become more firm and calculations of wall stresses become available, additional tests may be conducted using partially reversed strain in an attempt to simulate the actual stress cycle.

1.6.5 Materials for Examination

The materials studied will be those considered candidates for first-wall application.³⁷ Type 316 stainless steel will receive the highest priority in the test program. Annealed and 20%-cold-worked titanium-modified stainless steel will be included in order to develop information on an alloy that has inherently higher ductility. The precipitation-hardened alloy PE-16 will be investigated in a later stage of the study because of its higher strength and potentially lower swelling.

1.6.6 Irradiation

A stainless steel first wall will accumulate about 200 ppm He in a year of operation at a wall loading of 1 MW/m^2 . Since a primary objective of this study is to determine the effect of cyclic stress on helium behavior in first-wall materials, the irradiation facility should produce at least as much helium as is expected in fusion reactor application. The High Flux Isotope Reactor (HFIR) meets this requirement

³⁶C. R. Brinkman, G. E. Korth, and R. R. Hobbs, "Estimates of Creep-Fatigue Interaction in Irradiated and Unirradiated Austenitic Stainless Steels," *Nucl. Technol.* 16: 297-307 (1972).

³⁷F. W. Wiffen, "Plan for CTR Bulk Radiation Effects Program," unpublished document, ORNL, December 1974.

for short exposures. Conditions will be adjusted to attain damage and helium levels of 10 dpa and 200 ppm. Samples will be irradiated in HFIR by the methods developed previously. The specimens will be arranged in capsules containing ten each. Gamma heat conducted through helium gas surrounding the specimens will provide the specified elevated temperatures.

In subsequent experiments to examine effects of microstructure the ORR will be used to allow large numbers of samples to be irradiated in a controlled environment. Similar helium and displacement levels will be obtained.

1.6.7 Experimental Apparatus

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

Since the atmosphere has a significant influence on fatigue properties at high temperatures,³⁰ 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 is being designed for use with the testing system.

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

1.6.8 Postfracture Examination

Scanning electron microscope fractography studies will be made on the specimens to determine the nature of the fracture mechanism. When correlated with mechanical tests, fractography provides very valuable information on the fracture process. It is important to establish whether internal helium results in intergranular fracture in fatigue loading as it does in tensile loading.

³⁰H. 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. Met. Soc. AIME* 245: 947-53 (1969).

Samples for transmission electron microscopy (TEM) will be cut from regions adjacent to the fracture surface. The TEM is necessary to observe helium bubbles and to view the dislocation structure resulting from cyclic deformation.

The above techniques will be supplemented by vacuum fusion analysis for interstitials and other techniques that become necessary as the investigation progresses.

1.6.9 In-Reactor Investigation

Although much useful information will be gained by the program previously discussed, in order to simulate reactor application accurately, fatigue damage should be accumulated simultaneously with radiation damage. The next series of experiments will employ an instrumented reactor facility with the loading device in the reactor itself. A hydraulic or thermal expansion system might be used to provide the cyclic stress.

1.6.10 Status of the Project

Specifications for the servo-hydraulic fatigue testing system have been sent out for competitive bidding. Delivery is expected within 90 days. Preliminary design of the vacuum system has been completed, and estimates have been requested from several vendors.

A hot cell with very low background radiation has been obtained. Since the specimens to be tested will not give rise to particulate contamination, use of a clean hot cell will enable easy access to service the equipment in the cell. Modifications to the cell are now being planned to accommodate the large load frame. Adjacent cells will be used for disassembly of the irradiation capsules to minimize transport of the radioactive specimens and to better utilize hot-cell personnel.

The irradiation capsules will be modeled after existing HFIR capsules. Only the interior design need be changed. Heat transfer calculations and capsule design have begun.

1.7 USE OF FISSION REACTORS FOR FUSION REACTOR FIRST-WALL MATERIALS DEVELOPMENT - J. A. Horak

1.7.1 Purpose

This work is a portion of the effort to optimize the utilization of existing and proposed neutron irradiation facilities in the development of materials for fusion reactors. The information presented here is restricted to the structural materials for the first wall. For an irradiation facility to be useful in the development of first-wall materials it must satisfy the following criteria.

1. Capability for producing dpa and helium levels at actual and accelerated rates. For a fusion reactor operating at 1 MW/m^2 these values are 13 and 175 per year, respectively, for type 316 stainless steel, which is a prime candidate for the first wall of fusion reactors.
2. Capability of actively producing during irradiation the temperature and stress conditions that will exist in the first wall.
3. Capability for *in situ* determination of the most important (limiting) materials properties.
4. Capability for actively reproducing during irradiation the chemical environment to which the first wall will be exposed.
5. A large experimental volume with the above capabilities.

The following properties must be measured as a function of the principal materials and irradiation parameters:

Ductility - Uniform and total elongation

Stresses - Yield and fracture

Creep - Rate, time to rupture, strain at rupture

Fatigue - Rate of cycling, stress per cycle, strain at failure, cycles to failure

Swelling.

The parameters for all these properties are: (1) temperature, (2) fluence, (3) flux, (4) composition, (5) microstructure, (6) stress, (?) chemical environment, and (8) welding. Because of the large size of this matrix an experimental volume of at least 1 liter is required.

1.7.2 Evaluation of Irradiation Sources

The proposed and existing neutron irradiation facilities are listed below together with comments pertinent to their use for first-wall materials studies.

Proposed Irradiation Facilities

Operating $d-t$ reactor

Positive feature:

1. Perfect match of fusion reactor neutron spectrum and chemical species.

Negative features:

1. If wall loading is low ($<1 \text{ MW/m}^2$) cannot provide real-time or accelerated testing; flux, temperature, and stresses will also be too low.
2. Probably low duty factor in early experimental fusion reactors.
3. Not available until late 1980s.

d^+ , Li source

Positive features:

1. High flux for accelerated testing.
2. Large volume for accelerated and real-time testing for $\geq 1 \text{ MW/m}^2$.
3. Easy to instrument and to control temperature, stress, and chemical environment.
4. High duty factor (>0.9).

Negative features:

1. Does not match fusion reactor neutron energy spectrum.
2. Not available until about 1983.

$d-t$ 14-MeV neutron source

Negative features:

1. Flux too low for even real time testing for any wall loadings.
2. Experimental volume too small for required test matrix and instrumentation.
3. Does not match fusion reactor neutron energy spectrum.

Existing Irradiation Facilities

Fission reactors

High-flux mixed spectrum reactors (flux at least about 1×10^{18} n m⁻² sec⁻¹ of fast and thermal neutrons.

Positive features:

1. Can produce dpa and helium at real time up to ~ 2 MW/m² and at accelerated rates below this in alloys containing nickel.
2. Instrumentation available to actively control during irradiation the temperatures, stresses, and chemical environments relevant to fusion reactors.
3. Can conduct *in situ* measurements.
4. Have the large experimental volume necessary for the required test matrix.
5. Have high duty factor.

Negative features:

1. Do not match fusion reactor neutron energy spectrum.
2. Cannot produce relevant helium concentrations in materials that do not contain nickel.

Fast neutron reactors

Positive features:

1. Can produce dpa greatly accelerated over that in a fusion reactor at 1 MW/m².
2. Large experimental volume.
3. Passive temperature and stress control; active control may be possible later.
4. Good simulation of the fusion reactor neutron energy spectrum for regions beyond the first wall.

Negative features:

1. Cannot produce relevant helium concentrations in any structural material.
2. No capability for *in situ* measurements.
3. Currently no active control of temperature, stress, or chemical environment.

Ion accelerators

Positive features:

1. Highly accelerated dpa rates, good for swelling studies.
2. Can actively control temperature, stress, and chemical environment.
3. Can conduct *in situ* measurements.

Negative features:

1. Extremely difficult to conduct mechanical properties studies.
2. Time dependence of kinetic processes must be established.

From these lists we see that fission reactors that have high fast and thermal neutron fluxes combined with the instrumentation for *in situ* control and measurement provide the best neutron irradiation source for simulating the effects of the fusion reactor environment. In addition, they are the only facilities that can provide accelerated testing for a nominal 1 MW/m^2 wall loading, and they are the only facilities that are available for first-wall development for at least the next seven to eight years.

Table 1.8 lists the reactors in the U.S.A. that satisfy the flux criteria; also presented is information important for the use of these reactors for first-wall development. In the range of interest the concentration of helium is proportional to the thermal neutron fluence to approximately the 1.5 power and the displacement level is proportional to the fast neutron fluence to the first power. For this reason it becomes important to be able to alter the neutron spectrum during an irradiation to keep the He/dpa close to the proportion in the first wall of a fusion reactor. The capability to alter the neutron spectrum provides a method to avoid situations of the type shown in Fig. 1.2. For irradiations of type 316 stainless steel in HFIR, the helium generation rate is so rapid that after only a short time the He/dpa is too great to be relevant to first-wall conditions. For such irradiations the effects of helium are greatly exaggerated relative to the displacement

Table 1.8. Physical Parameters for Fission Reactors Having a High Flux of Fast and Thermal Neutrons

Facility	Flux, $\text{a}^{-2} \text{sec}^{-1}$		Max Diam (cm)	Max Length (m)	Volume, liters		Duty Factor	Change
	Thermal	$>0.1 \text{ MeV}$			Total	At Flux Given		
ATR								
West Flux Trap	3.0×10^{10}	4×10^{10}	101	1.22	9.8	2.5	0.71	Probably not
CEFR								
Core Loop E-7	3.0	2.6	74	0.91	5.3	1.3	0.80	Probably not
MFIR								
V15 or V16	1.3	4	191	0.102	~2.3	0.6	0.70	No
H								
Process Tube	1.0	1.0	32	6.10	4.83	3.62	0.60	Probably not
Control Rod Hole	1.0	0.2	89	6.10	13.77	10.3	0.60	No
ORR								
C-3	2.4	3.8	67	0.61	2.9	0.7	0.88	Yes
C-5	3.5	3.7	67	0.61	2.9	0.7	0.88	Yes
E-5	3.6	3.8	67	0.61	2.9	0.7	0.88	Yes

Fig. 1.2. Helium Content as a Function of Displacement Level for Type 316 Stainless Steel.

effects and property measurements would be misleading for fusion reactor applications. Irradiation of type 316 stainless steel in position E-5 of ORR provides a very good simulation for a fusion reactor for irradiation times up to 1.3 year. However, continued irradiation in E-5 at the same fast and thermal neutron fluxes leads to too high He/dpa. Figure 1.2 also shows for irradiation times less than two years in position C-3 of ORR that the He/dpa for type 316 stainless steel does not approximate the fusion reactor situation as well as in position E-5. For irradiation times longer than three years in C-3 the He/dpa becomes too high for valid fusion reactor simulation.

Of the reactors listed in Table 1.8, ORR is the most amenable to neutron spectrum tailoring and in general is the most versatile reactor in existence for first-wall studies. The magnitude of the total neutron flux in the high-flux experimental positions — positions C-3, -5, and -7 and E-3, -5, and -7 of Fig. 1.3 — can be controlled by the number of fuel elements used for the core and/or the mass of ^{235}U per fuel element. The thermal neutron flux can be increased by placing additional moderator between the fuel and the test specimens. This is usually accomplished by placing a beryllium sleeve around the experimental assembly and/or increasing the thickness of water between the fuel elements and the test specimens. The fast neutron flux can be increased while decreasing the thermal neutron flux by placing a sleeve of aluminum containing from 5 to 15 at. % ^{235}U around the experimental assembly. In principal it is possible, without difficulty, to have any thermal and fast neutron fluxes desired over the range from about 1 to $6 \times 10^{18} \text{ n m}^{-2} \text{ sec}^{-1}$. The permutations are too great to be presented here.

Figure 1.4 provides an example of what can be accomplished in ORR by merely rearranging the core and/or by placing moderating material between the fuel and the experimental assembly. Figure 1.4 illustrates that for type 316 stainless steel the He/dpa achieved in ORR very closely approximates that produced in a fusion reactor operating at 1 MW/m^2 . This close fit could also be accomplished by placing sleeves containing ^{235}U around the experiment. For long irradiations (exceeding about two years) the use of the ^{235}U sleeves would be preferred for the last half of the

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Be - Beryllium element
 Fuel - Fuel element
 SR - Shims rod
 Exp - Experiment or
 experimental facility
 PBDL

A-1 Be	A-2 Be	A-3 Fuel	A-4 Fuel	A-5 Fuel	A-6 Fuel	A-7 Be	A-8 Be	A-9 Be
B-1 Be	B-2 Be	B-3 Fuel	B-4 SR	B-5 Fuel	B-6 SR	B-7 Fuel	B-8 Be	B-9 Be
C-1 Be	C-2 Fuel	C-3 Fuel	C-4 Fuel	C-5 Fuel	C-6 Fuel	C-7 Fuel	C-8 Exp	C-9 Be
D-1 Be	D-2 Fuel	D-3 Fuel	D-4 SR	D-5 Fuel	D-6 SR	D-7 Fuel	D-8 Exp	D-9 Be
E-1 Be	E-2 Fuel	E-3 Exp	E-4 Fuel	E-5 Fuel	E-6 Fuel	E-7 Fuel	E-8 Exp	E-9 Be
F-1 Be	F-2 Fuel	F-3 Fuel	F-4 SR	F-5 Fuel	F-6 SR	F-7 Exp	F-8 Be	F-9 Be
G-1 Be	G-2 Be	G-3 Be	G-4 Be	G-5 Be	G-6 Be	G-7 Be	G-8 Be	G-9 Be

Fig. 1.3. Typical ORR Core Configuration. Positions C-3, -5, and -7 and E-3, -5, and -7 have both high thermal and fast neutron fluxes and can be instrumented for active control of temperature, stress, and chemical environment. The poolside facility can accommodate a mockup of a fusion reactor blanket module. The total flux in the poolside facility is $5.5 \times 10^{18} \text{ n m}^{-2} \text{ sec}^{-1}$ at 43 mm (1.7 in.) from the core.

Fig. 1.4. Helium Content as a Function of Displacement Level for Type 316 Stainless Steel Irradiated in Various Positions of the ORR. Percentage figures refer to enhancement of thermal flux by rearrangement of the reactor core or by placing beryllium sleeves around the experiment.

irradiation to maintain a high dpa rate. This would provide accelerated testing since for type 316 stainless steel the helium production rate is already higher than in a fusion reactor at 1 MW/m^2 .

Although type 316 stainless steel has been used as an example, the other nickel-bearing alloys, PE-16 (43% Ni) Inconel 600 (77% Ni), that are candidate first-wall materials could be irradiated in ORR to produce He/dpa very similar to those that will exist in a first wall that is constructed from these materials.

1.7.3 Conclusions

There are fission reactor irradiation facilities that can be utilized for the development of first-wall alloys. For alloys that contain nickel, He/dpa ratios can be produced that are very close and even identical to those that will exist in a first wall.

The reactor that is best suited for the fusion reactor first-wall materials development program is the ORR for the following reasons:

1. Helium and dpa at rates accelerated over 1 MW/m^2 operation can be produced.
2. Instrumentation exists for active control of temperature, stress, and chemical environment during irradiation.
3. The capability for *in situ* determination of properties is present.
4. The experimental volume at high flux is large enough to accommodate the required test matrix.
5. The total neutron flux and the ratio of fast to thermal neutron fluxes in an experimental position can be easily modified. This is mandatory to provide the correct He/dpa for first-wall development.
6. The ORR is available for the long-term irradiations (up to about five years) that will be required to achieve end-of-life conditions for a power-producing reactor. It also has the high duty factor (88%) required to attain this fluence.

1.8 EBR-II ROW 7 IRRADIATION EXPERIMENT — M. L. Grossbeck and E. E. Bloom

1.8.1 Introduction

This irradiation is a joint experiment between programs at ORNL, HEDL, Battelle Pacific Northwest Laboratories, 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 experiments 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. The scope and objectives of the individual portions of the experiment are outlined below.

1.8.2 Subassembly Description

Nine capsules will be placed within a standard hexagonal EBR-II subassembly. Three capsules will be filled with sodium and sealed but will be in thermal contact with the reactor coolant and will, therefore, operate at temperatures between 370 and 400°C. Three capsules will be heat-pipe temperature controlled and will be placed directly above and welded to three gas-gap-controlled capsules. The heat pipes have been 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. 1.5. 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.

Specimens will be positioned in racks with provision for irradiation-induced swelling. Since refractory metals will be included, each of the

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Fig. 1.5. Temperature Control Capability of a Heat Pipe.

six capsules operating above reactor coolant temperature will contain high-purity zirconium foil as a getter for oxygen.

Plans are for inclusion of the capsules in a modified M-1A sub-assembly for irradiation in the 7N4 position of EBR-II. Irradiation for approximately 18 months is planned to give a neutron fluence on the reactor midplane of about 4×10^{26} n/m² (>0.1 MeV). Adequate neutron fluence dosimeters and temperature monitors have been included in the experiment.

1.8.3 Description of Individual Experiments

A fusion reactor first wall will be subjected to a neutron flux of about 5×10^{18} n m⁻² sec⁻¹ (>0.1 MeV). A significant fraction of these neutrons will have energies of 14.1 MeV. The different neutron energy spectrum of a fusion reactor compared with a fission reactor will result in significantly higher helium generation rates. Primary objectives of the fusion reactor portion of this experiment are to investigate the effects of large amounts of helium and define the temperature dependence

of the radiation response of candidate first-wall construction materials. Helium will be 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 the ORR. In thermal reactor. (HFIR and ORR) helium is produced continuously by the thermal neutron reactions described in Sect. 1.1.1. 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 followed by neutron irradiation is the only way available at present to obtain damage levels expected in a fusion device.

1.8.3.1 Helium Injection Experiment

This experiment will provide a control for the helium injection experiment as well as provide information on the effects of temperature and stress under irradiation on the mechanical and swelling properties of fusion reactor materials. Miniature tensile specimens (0.25 by 2.5 by 31.8 mm) designed to permit postirradiation testing outside a hot cell, larger tensile specimens (0.76 by 4.95 by 44.5 mm), and pressurized tubes will be employed. A total of 18 helium-doped and undoped large samples will be irradiated at each of the following temperatures: 525, 575, 625, and 700°C; 24 at 400°C. In addition, 13 miniature tensile samples at 400°C will be irradiated outside the core to achieve only half the fluence accumulated by the remaining samples.

1.8.3.2 Study of Irradiated Weldments

To obtain information on the effects of fast-neutron irradiation on weldments in the potential first-wall alloys, a series of welded samples will be included. Materials to be examined will be type 316 stainless steel 20% cold worked, PE-16, and V-20% Ti. Both gas tungsten-arc and electron-beam welding will be employed with the use of welding

rods of several compositions. Samples will be cut in both the longitudinal and transverse directions with respect to the weld, and separate samples will be made from the weld region and the heat-affected zone. The type 316 stainless steel and PE-16 will be irradiated at 400, 525, and 575°C, and the V-20% Ti will be irradiated at all five temperatures.

1.8.3.3 Neutron-Ion Irradiation Correlation

This experiment is being conducted by Battelle Pacific Northwest Laboratories. The purpose of the experiment is to compare the microstructure of neutron-irradiated material and helium-preinjected neutron-irradiated material with identical material that has been ion bombarded. The molybdenum is the same reference material being used in the bcc Ion Correlation Experiment. Some nickel specimens will be preinjected with helium so as to compare with ion-bombarded specimens that have been bombarded with both nickel and helium ions.

1.8.3.4 Evaluation of $\text{Si}_3\text{N}_4\text{-Y}_2\text{O}_3$ Insulator Materials

This experiment is being conducted by Los Alamos Scientific Laboratory as part of its program on fusion reactor insulator materials. Hot-pressed samples of Si_3N_4 containing additions of Y_2O_3 in increments of 5 wt % from zero to 20 wt % are included.

Silicon nitride is a refractory ceramic whose low coefficient of thermal expansion and high thermal conductivity provide high thermal shock resistance. Additions of Y_2O_3 have been found to improve the elevated-temperature strength compared with samples with MgO additions. Samples of the latter irradiated to a dose of $3 \times 10^{25} \text{ n/m}^2$ have also exhibited low swelling. This experiment is the first in which material with Y_2O_3 will be irradiated and will provide samples for swelling, mechanical properties, and electron microscopical investigations.

1.8.4 Status of Experiment

The design of the experiment has been completed. All capsule parts and specimen racks have been fabricated except an outer housing tube, which is now being machined. The hazards analysis is now in progress

and is expected to be completed by the end of FY 1976. The experiment is expected to be shipped to EBR-II in September.

1.9 NEUTRONICS CALCULATIONS FOR RADIATION DAMAGE STUDIES¹⁹ - T. A. Gabriel, J. D. Amburgey, and R. T. Santoro

The primary knock-on atom (PKA) data base RECOIL, derived from ENDF-B-IV data and composed of most elements of interest to the fusion reactor community, has been completed and documented.⁴⁰ The data base has been used extensively to calculate radiation damage in metals (i.e., gas production and atom displacements) for different neutron environments currently available (ORR, HFIR, ORIC, EBR-II, etc.) and for expected neutron environments envisioned in future fusion reactors.^{41,42} 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 produce the damage anticipated in fusion reactors.

At present extensive interest has been generated in utilizing the ORR as a prime experimental facility where fusion reactor irradiation damage conditions in first-generation reactor materials such as type 316 stainless steel, PE-16, and Inconel 600 can be approximated. The ORR is a very favorable facility since the irradiated material can be instrumented and since localized modifications in the neutron environment can be implemented through changes in the material composition of the sample container. Preliminary calculations indicate that by using a slightly modified neutron environment in the ORR (i.e., a change in the thermal-to-fast ratio),

¹⁹Contribution from the Neutron Physics Division.

⁴⁰T. A. Gabriel, J. D. Amburgey, and N. M. Greene, *Radiation-Damage Calculations: Primary Recoil Spectra, Displacement Rates, and Gas-Production Rates*, ORNL/TM-5160 (March 1976); to be published in *Nuclear Science and Engineering*.

⁴¹R. T. Santoro, private communication. The neutron flux for these calculations is for the experimental power reactor design No. 2.

⁴²R. T. Santoro and V. C. Baker, *Neutronics and Photonics Calculations for the Tokamak Experimental Power Reactor*, ORNL/TM-5466 (1976).

the damage obtained in nickel-bearing alloys can closely approximate the damage expected on the first wall of a fusion reactor (see Fig. 1.4).

Some calculated damage data for type 316 stainless steel, using neutron spectra from the ORR (C-3), the HFIR (PTP), and an experimental power reactor⁴¹ (first wall), are shown in Figs. 1.6 through 1.8 as functions of axial position and irradiation time. For nickel-bearing alloys and for a large thermal component of the neutron flux, such as found in ORR or in HFIR, the two-step reaction

dominates the helium production.⁴²

The radiation damage expected⁴² throughout a fusion reactor is shown in Fig. 1.9. As can be seen, the damage at the rear of the fusion reactor shield is reduced by many orders of magnitude from that of the first wall. At present, radiation damage in the magnets is not thought to be a major problem.

An additional option has been included in the analysis program associated with RECOIL. Shown in Fig. 1.10 are the output obtained and a comparison of the PKA recoil spectra obtained for HFIR (peripheral target positions) and for a fusion reactor,⁴¹ each multiplied by the ΔE of the recoil group and by the effective recoil energy (i.e., that portion of the recoil energy that will produce further damage). The peaks at about 600 eV in the HFIR results are due to neutron capture in iron.

⁴³For these calculations, the He production in type 316 stainless steel was assumed to be given by

$$\text{He (at. ppm)} = 1.24 \times 10^{-35} (\phi_{\text{THERMAL}} t)^{1.68}.$$

The neutron fluxes for these calculations are for the experimental power reactor reference design (see ref. 42).

Fig. 1.6. Calculated Displacement Rates and Displacements in Type 316 Stainless Steel for Three Different Neutron Environments.

Fig. 1.7. Calculated Hydrogen Production Rates (at. ppm/sec) and Production (at. ppm) in Type 316 Stainless Steel for Three Different Neutron Environments.

Fig. 1.8. Calculated Helium Production Rates (at. ppm/sec) and Production (at. ppm) in Type 316 Stainless Steel for Three Different Neutron Environments.

Fig. 1.9. Calculated Damage in a Fusion Reactor as a Function of Radial Distance from the Reactor First Wall.

Fig. 1.10. Primary Knock-on Atom Spectra in HFIR (PTP) and in a Fusion Reactor (First Wall) Multiplied by the ΔE of the Recoil Group and by the Effective Recoil Energy.

1.10. HIGHLIGHTS OF RADIATION EFFECTS PRESENTATIONS AT THE
"INTERNATIONAL CONFERENCE ON RADIATION EFFECTS AND TRITIUM
TECHNOLOGY FOR FUSION REACTORS" - F. W. Wiffen

A well-attended international conference on Radiation Effects and Tritium Technology for Fusion Reactors was held in Gatlinburg, Tennessee, on October 1-3, 1975. This meeting, sponsored by ORNL, ERDA, ANS, and AIME, attracted more than 300 attendees, including a good representation from outside the U.S.A. The six radiation effects sessions contained 56 papers, with the greatest emphasis on the examination of irradiation effects on microstructure and on the analytic treatments of radiation effects (theory, computer simulation, and modeling).

The greatest value of the radiation effects presentations was as a review of the state of the art of our efforts in evaluating fusion reactor radiation effects. Not a great deal of important new data were presented.

It was encouraging to note from the many presentations that most of the established groups involved in radiation effects studies are now focusing at least a part of their efforts on the needs of the fusion reactor programs. At the same time, however, it was disappointing to see the lack of direction in a large part of the work purported to be fusion reactor technology. Too little of the work was of direct application to the needs of fusion reactor systems design. In particular, while reactor design is focusing on near-term reactors and moving towards the specification of conventional alloys for structures, too much of the radiation effects work continues to be on refractory metals. There was also a noticeable lack of coordination between the various programs represented. Much of the work presented was repackaged and reinterpreted results from previous work on both fundamental and LMFBR programs and thus the connection with fusion reactor needs was often tenuous. It became apparent that in many cases the work presented reflected "easy" experiments, and that near-term visibility is being achieved at the expense of long-term progress towards the true program goals. The amount of efforts directed towards fusion reactor needs suggests that this problem will soon be overcome, and that better coordination and direction will point all these efforts toward the mid-term and long-term needs of reactor design.

Some of the highlights of the radiation effects program follow: the theory efforts under way at several sites are now incorporating fusion reactor conditions into the formulation. These now include the effects of primary knock-on atom energy, stress effects, effects of insoluble gases (neutronically produced helium), and effects of duty cycles pertinent to the various reactor design concepts. While none of the theories or analytical treatments are yet fully predictive, all are able to explain most available observations. It is to be hoped they will soon be complete enough for modest extrapolation beyond the range of observation and for the definition of critical experiments.

Three papers described experiments that use ion bombardment of stressed samples to evaluate irradiation creep under simulated reactor conditions. These papers contained enough data to demonstrate that the technique is viable and that thermal and irradiation creep rates can be simulated, separated, and measured.

Several papers compared the rate of damage production under irradiation with neutrons of average energy near 14 MeV with rates under irradiation with ions or lower energy neutrons. The interpretation of observations ranged from results being in perfect agreement with the predictions of correlations based on damage energy to disagreement of a factor of 2 with predictions. In general, it can be said that 14-MeV neutron irradiation has not yet revealed any unexpected results, and that observations are at least within a factor of 2 of expectations based on correlation with other irradiation effects studies.

Results of refractory metals experiments continue to identify materials and conditions with unexpected swelling resistance and a resistance in niobium and Nb-1% Zr to helium embrittlement at modest helium levels. Ion simulation experiments on these materials continue to be plagued by the susceptibility of refractory metals to oxygen pickup. However, an awareness of this by the involved experimenters suggest that solutions to the problem will be found.

ORNL exploitation of the advantages of irradiating nickel-containing alloys in the HFIR to simulate fusion reactor service continued with the evaluation of microstructural changes in type 316 stainless steel.

These irradiations resulted in displacement damage levels in the range 40 to 60 dpa and helium levels of 3000 to 4300 at. ppm. Swelling was correlated with helium level, showing that helium bubbles, not voids, accounted for the swelling. Below about 600°C cold-worked material swelled less than solution annealed material, but in both conditions the high helium levels resulted in greater swelling than for irradiations at the same temperature and to the same dpa levels in a fast reactor.

The examination of radiation effects in magnet components showed that the greatest uncertainty is in the response of insulators and spacers to the anticipated neutron flux at the magnet. Radiation effects in the superconductor are unlikely to limit operation, but resistivity increases in the stabilizing elements (copper or aluminum) may require periodic warming of the magnet to room temperature to restore high conductivity at low temperature.

In summary, this meeting served a valuable purpose in bringing together the international community involved in radiation effects studies. The papers provided a good review of the status of various programs and highlighted some interesting new results. The many programs now in place are taking full advantage of the facilities and experience provided by the past 30 years of radiation effects work. The vigorous effort toward evaluating potential fusion reactor radiation effects can be expected to yield rapid progress in the next few years.

1.11 ABSTRACTS OF PUBLISHED PAPERS

1.11.1 The Investigation of Neutron Radiation Effects in Structural Materials for CTR Use⁴⁴ - F. W. Wiffen

Neutron irradiation of structural materials in the high-flux region of a Controlled Thermonuclear Reactor (CTR), especially in the first wall of the reactor, will result in swelling due to cavity formation and

⁴⁴Summary of pp. 64-88 in *Proc. Int. Conf. Radiation Test Facilities for the CTR Surface and Materials Program, July 15-18, 1975, at Argonne Laboratory*, ANL/CTR-75-4.

loss of ductility through both lattice hardening and the effect of transmutation-produced helium on the fracture mode. The intensity and relative importance of the effects will be a strong function of the material and reactor operating conditions. A consideration of the effects of the 14-MeV D-T fusion neutrons, based on calculated damage response, on the sparse available data from high-energy neutron irradiation, and on an intuitive understanding of past radiation effects experimentation, 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.

Irradiation experiments designed to evaluate materials for CTR service must be conducted in a number of facilities. Used in a well-coordinated evaluation program, the combined results from all available irradiation facilities will lead to an understanding of the radiation effects that will occur in CTRs. The experimental program will involve three distinct phases: (a) correlation experiments, (b) screening studies, and (c) qualification and design data generation. Each of these phases will evaluate a number of physical and mechanical properties. The end result of this program will be the qualification of a few materials for CTR use.

1.11.2 Use of Fission Reactors for CTR Bulk Radiation Effects Studies⁴⁵ - J. A. Horak

The neutron irradiation facilities in some existing fission reactors for use in the CTR bulk radiation effects program have been evaluated. There are two major requirements for a neutron source for CTR materials development. One requirement is a high flux, $>10^{14}$ n cm⁻² sec⁻¹, of neutrons that are capable of producing, at an accelerated rate, the atomic displacements and principal nuclear transmutations that will be produced in structural alloys by fusion neutron radiation. This flux

⁴⁵Summary of pp. 830-41 in *Proc. Int. Conf. Radiation Test Facilities for the CTR Surface and Materials Program, July 15-18, 1975, at Argonne National Laboratory, ANL/CTR-75-4.*

must be combined with a sufficiently large experimental volume, several hundred cubic centimeters, for irradiation of the hundreds of samples that are required to develop the structural material and design data for fusion reactors. In addition, since radiation can affect the mode of deformation and fracture, it is desirable to have the capability of active temperature and stress control during irradiation. There are irradiation facilities in fission reactors that satisfy these criteria for alloys that contain nickel. Because of the helium produced by thermal neutron absorption in nickel the helium-to-displacement ratios that will exist in proposed fusion reactors can be produced in alloys such as stainless steels in reactors that have a high thermal neutron flux combined with a high fast neutron flux. This is especially important since there is a high probability that an austenitic stainless steel will be the structural material for the reactors to be built early in the 21st century. Facilities are available in fission reactors for the relevant cryogenic irradiation and testing of superconducting magnet materials and magnets. The evaluation indicates that fission reactors can be utilized to provide valuable and required information for the development of materials for fusion reactors.

**1.11.3 Creep and Tensile Properties of Helium Injected Nb-1% Zr⁴⁶ -
F. W. Wiffen**

Specimens of commercial purity Nb-1% Zr were helium doped by cyclotron alpha bombardment near room temperature to gas contents near 2 and 20 at. ppm He, corresponding to helium production in a CTR first wall during approximately one month and one year of operation at 1 MW/m² neutronic wall loading. These specimens were tensile and creep tested to evaluate the effect of helium, in the absence of other components of CTR radiation effects, on the mechanical properties. Tensile tests at 1000 and 1200°C show no significant effect of either helium level on the strength or elongation values. Creep-rupture tests at the same temperatures on Nb-1% Zr showed rupture elongations somewhat reduced

⁴⁶Abstract of pp. 344-61 in *Radiation Effects and Tritium Technology for Fusion Reactors* (Proc. Intern. Conf. Gatlinburg, Tenn., Oct. 1-3, 1975), CONF-750989.

in the helium injected samples, but the dependence on helium content and on test temperature does not fit the usual effect of helium on elevated-temperature ductility. We thus conclude that in the absence of displacement damage this alloy is resistant to elevated-temperature embrittlement for helium contents up to 20 at. ppm. The resistance to embrittlement results from the precipitation of helium on a submicroscopic level and from the alloy's inherent resistance to grain boundary separation.

1.11.4 Swelling and Microstructural Changes in Type 316 Stainless Steel Irradiated Under Simulated CTR Conditions^{4,7} - P. J. Maziasz, F. W. Wiffen, and E. E. Bloom

Tensile specimens of type 316 stainless steel were irradiated in the High Flux Isotope Reactor (HFIR) to displacement damage levels of 42-60 dpa (displacements/atom) and helium contents of 3000-4300 at. ppm at temperatures from 380 to 680°C. The samples were irradiated in both the annealed condition (1 hr at 1050°C) and the 20% cold worked condition. The microstructures of these samples were examined by transmission electron microscopy (TEM). The results were compared with specimens of the same heat of steel irradiated in EBR-II to approximately the same conditions with the exception of helium content, which was 15 at. ppm. Swelling in annealed and cold worked samples with high helium content was approximately constant with temperature from 380 to 550°C and increased rapidly from 550 to 680°C. Swelling in cold-worked samples was less than in the annealed samples for all temperatures except 680°C. Swelling was larger at all temperatures for samples containing large helium concentrations than for the same material with small helium concentrations. Recrystallization at 600°C destroyed the cold work swelling resistance. Cold work increased the cavity concentrations, decreased the cavity sizes, and reduced the swelling, compared with annealed material containing equivalent high helium concentrations. Swelling was greatly increased at irradiation temperatures above 600°C in both

^{4,7}Abstract of pp. 259-88 in *Radiation Effects and Tritium Technology for Fusion Reactors* (Proc. Intern. Conf. Gatlinburg, Tenn., Oct. 1-3, 1975, CONF-750989).

the annealed and cold-worked samples; the increase was due to the formation of very large cavities at the grain boundaries. Calculations to compare the amount of helium measured in the samples with the amount necessary to stabilize equilibrium bubbles have shown most of the cavities to be equilibrium helium bubbles.

2. SURFACE STUDIES

L. C. Emerson L. Heatherly R. E. Clausing

Impurities coming into the plasma from the walls of present-day toroidal plasma confinement devices modify plasma behavior substantially. Small fractions of high- Z ions in the plasma greatly decrease plasma temperatures and increase plasma energy losses. We are attempting to identify and control the sources of impurities from the "first wall" in ORMAK. Auger electron spectroscopy, soft x-ray appearance potential spectroscopy, and other surface sensitive techniques have been used to characterize the surface composition of the first wall and to develop methods to remove carbon and oxygen. Oxygen glow discharge cleaning has been shown, in the laboratory, to be an effective way of removing carbon from gold films (simulated ORMAK liner material), and the use of oxygen discharge cleaning in ORMAK has resulted in a decrease in plasma contamination, a 50% increase in plasma current and an accompanying increase in plasma temperature. In spite of these improvements the walls of ORMAK are far from clean. Substantial amounts of carbon, oxygen, iron, and other elements remain. The results of these initial measurements of the composition of the surface of the ORMAK liner and the effectiveness of oxygen glow discharge cleaning have been published.¹

More recently the results of additional studies were reported.² The studies of glow discharge cleaning were extended to include both gold and stainless steel surfaces and to include direct bombardment with 300-eV hydrogen and oxygen ions. Direct ion bombardment with either hydrogen or oxygen is very effective in reducing carbon surface impurities on both stainless steel and gold. The oxygen bombardment produces a

¹R. 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(1): 437-42 (January-February 1976).

²R. E. Clausing, L. C. Emerson, L. Heatherly, and R. J. Colchin, "Surface Impurities and 'Clean-up' Techniques in ORMAK," paper presented at the International Conference on Surface Effects in Controlled Fusion Devices, San Francisco, California, February 16-20, 1976.

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hyperstoichiometric oxide layer. The hydrogen ion bombardment produced a substoichiometric oxide film but did not remove the oxide. A soft x-ray appearance potential probe (SXAPS) developed specifically for use in ORMAK was used to monitor the changes in surface composition that occurred during hydrogen glow discharge cleaning in ORMAK and the recontamination of the surface by carbon when the discharge was discontinued. Techniques were developed for controlling hydrocarbon contamination of the probe, which plagued its initial use in ORMAK. We have also developed techniques for using an oxygen glow discharge in the probe itself for cleaning away carbonaceous deposits should they develop. Thus far the probe has shown that hydrogen discharge cleaning in ORMAK does reduce surface carbon levels, that the carbon recontamination of surfaces in the ORMAK liner is rapid, and that iron is a major contaminant on gold surfaces in the ORMAK liner.

Studies of the use of oxygen and hydrogen discharges for cleanup purposes are continuing both in ORMAK and in the laboratory. The SXAPS probe is presently in use in ORMAK, and we are developing techniques to permit Auger electron spectroscopy (AES) and other surface analytical techniques to be used for plasma-surface studies in ORMAK and ISX (impurity study experiment³).

³R. J. Colchin and T. C. Jernigan, *ISX - A Tokamak for Surface and Impurities Studies*, ORNL/TM-5434 (May 1976).

3. COMPATIBILITY STUDIES

J. H. DeVan and J. E. Selle

The purpose of this work is to study the chemical compatibility of fusion reactor blanket and first-wall materials. Potential blanket materials include metallic lithium and molten Li_2SeF_4 . First-wall materials include austenitic stainless steels and refractory alloys based on niobium and vanadium. Initial efforts are concentrating on austenitic stainless steels.

3.1 CORROSION OF IRON-BASE ALLOYS BY LITHIUM - J. E. Selle and J. H. DeVan

Two papers^{1,2} have described our literature survey,³ our planned program, and initial work on our loop experiments. The last of these is described by the following paragraph from a published summary.²

A 2 1/4 Cr-1 Mo steel thermal-convection loop was run for 10,000 hr with a 600°C hot leg and 400°C cold leg. It showed grain-boundary grooving in the hot leg and deposition on the cold leg. Similar thermal-convection loops of types 321 and 304L stainless steel showed a higher corrosion rate for hot-leg samples along with mass transport of alloy constituents to the cold leg.

3.1.1 Loop Tests - J. E. Selle

Thermal convection loops constructed of types 304L, 321, and 316 stainless steel were operated with lithium at a hot-leg temperature of 600°C with a 200°C temperature difference. The maximum corrosion rates

¹J. E. Selle, "Corrosion of Iron-Based Alloys by Lithium," paper presented at the International Conference on Liquid Metal Technology in Energy Production, Seven Springs Resort, Champion, Pa., May 3-6, 1976; to be published in the proceedings.

²J. E. Selle and J. H. DeVan, "Lithium-Stainless Steel Corrosion Studies," paper presented at 1975 Winter Meeting, American Nuclear Society, Nov. 16-21, 1975, San Francisco, Calif., summary published in *Trans. Am. Nucl. Soc.* 22(TANSAO 22): 161 (1975).

³J. H. DeVan, J. E. Selle, and A. E. Morris, *Review of Lithium Iron-Base Alloy Corrosion Studies*, ORNL-TM-4927 (January 1976).

are shown in Fig. 3.1 along with data obtained from the literature. These tests will be used as a reference for future comparisons of the effects of lithium purity, temperature, and time and to compare the usefulness of corrosion inhibitors. Weight loss due to thermal-gradient mass transport of chromium and nickel was observed on test specimens at 485°C or higher with resulting depletion of alloying constituents at the reaction interface. Chromium and nickel were deposited at colder portions of the loop. Insert specimens in these loops provided an accurate profile of mass transfer rate as a function of local operating temperatures and loop position.

Fig. 3.1. Maximum Corrosion Rates in Stainless Steel Loops at 600°C Compared with Published Corrosion Rates of Stainless Steel by Lithium. 1 mil/year = 25.4 μ m/year. References: E. E. Hoffman, *Corrosion of Materials by Lithium at Elevated Temperatures*, ORNL-2674 (March 9, 1959). W. N. Gill et al., "Mass Transfer in Liquid-Lithium Systems," *AIChE J.* 6(1): 139-44 (March 1960). I. S. Nihei et al., *Corrosion of Austenitic Stainless Steels in Liquid Lithium*, JAERI Report M-5683 (April 10, 1974).

3.1.2 Static Capsule Tests - J. E. Selle

Static capsule tests of austenitic stainless steels at temperatures between 500 and 700°C were continued. The objectives of the tests are to:

1. determine the effects of C, N, O, and H on corrosion by lithium;
2. determine the effects of soluble (Ca, Al) and solid (Y, Zr, Ti) getters on corrosion of stainless steel by lithium;
3. determine dissimilar-metal mass transfer tendencies between stainless steel and Mo, Nb, and Co;
4. investigate feasibility of coating the stainless steel *in situ*, in order to improve corrosion resistance.

A reference test matrix was completed to establish a basis for comparison of any test. Tests were then run in which additions of various kinds were made to the lithium. The additions are summarized in Table 3.1. Weight changes were recorded on tabular specimens submerged in the lithium, and the results are summarized in Fig. 3.2. Weight gains were recorded for carbon, cobalt, and aluminum, while the remainder of the additives produced no significant weight change over the base matrix (no additive). For those samples showing a weight gain, metallographic analysis revealed reaction layers at the surface of the samples. Work is continuing on this technique to produce and study the effect of coatings on the corrosion resistance of austenitic stainless steels.

3.2 THE CORROSION RESISTANCE OF TYPE 316 STAINLESS STEEL TO Li_2BeF_4 (Ref. 4) - J. R. Keiser and J. H. DeVan

The blanket material for a fusion reactor must effectively breed tritium. It must also permit the efficient recovery of the tritium, and it must be chemically compatible with the fusion reactor components it contacts. The salt Li_2BeF_4 is presently being evaluated with respect to each of these requirements. The purpose of this paper is to report on the corrosion resistance of the fusion reactor containment reference material, type 316 stainless steel, to Li_2BeF_4 .

⁴Paper presented at 1975 Winter Meeting, American Nuclear Society, Nov. 16-21, 1975, San Francisco, Calif., summary published in *Trans. Am. Nucl. Soc.* 22(TANSO 22): 160 (1975).

Table 3.1. Additives in Stainless Steel Lithium Capsule Corrosion Tests

Type of Addition	Form	Concentration (wt %)
Interstitial impurity	C (graphite)	0.9
	Li ₂ O	0.09
	Li ₃ N	4.9
	LiH	7.3
Soluble getter	Ca	9.25
	Al	4.8
Solid getter (powder)	Y	1.7
Dissimilar metal (powder)	Co	12.1
	Mo	8.8
	Nb	8.9
Impurity + getter	Li ₃ N + Y	5.0 + 18.8
	Li ₃ N + Ca	4.9 + 12.6
	Li ₂ O + Y	0.09 + 0.36

Fig. 3.2. Weight Changes in Type 316 Stainless Steel Exposed in Capsules for 2000 hr to Lithium with Various Additives.

Corrosion of stainless steel due to reactions with the major salt constituents will be negligible considering the thermodynamic stability of these constituents. As shown⁵ in Table 3.2, both LiF and BeF₂ are significantly more stable than the fluorides of the constituents of the stainless steel (Cr, Fe, Ni, Mo). However, reactions can occur between the stainless steel and impurities in the Li₂BeF₄. Impurities such as NiF₂ and FeF₂ will react with chromium in the steel to form CrF₂, and HF will react with virtually all the elements constituting the stainless steel. Thus, the TF produced by the breeding reaction

becomes a significant factor in the utilization of Li₂BeF₄ as a fusion reactor blanket material. However, experience gained in the molten-salt reactor program (MSRP) on uranium-containing LiF-BeF₂ mixture indicates that the corrosive effects of chemically active species dissolved in the salt can be controlled through the use of a "redox" buffer. Such a buffer fixes the salt at the oxidation potential needed to maintain chemical compatibility with the container material and neutralizes the effects of oxidizing impurities in the melt. However, the buffer must be carefully selected to avoid the deposition of unwanted corrosion products or undesirable side reactions with the container material.

Prior corrosion data⁶ on type 316 stainless steel in LiF-BeF₂ were obtained under conditions where the concentration ratio between UF₄ and UF₃ established the redox potential of the salt. Under these conditions, the maximum weight loss at 650°C translated to an average wall thickness change of about 25 μm/year (1 mil/year). However, the predominant corrosion reaction was

⁵C. F. Baes, Jr., "The Chemistry and Thermodynamics of Molten Salt Reactor Fuels," *Nucl. Met.* 15: 624, P. Chiotti, ed., CONF-690801 (1969).

⁶J. W. Koger, *Alloy Compatibility with LiF-BeF₂ Salts Containing ThF₄ and UF₄*, ORNL-TM-4286 (December 1972), p. 36.

Table 3.2. Formation Free Energy of Fluorides at 1000 K

Compound	Free Energy per Gram-Atom of Fluoride	
	(kJ)	(kcal)
LiF	-523	-125
CeF ₃	-490	-117
BeF ₂	-448	-107
UF ₃	-410	-98
CrF ₂	-314	-75
FeF ₂	-280	-67
HF	-276	-66
NiF ₂	-230	-55
MoF ₆	-209	-50

and because of the selective removal of chromium, subsurface voids developed in the loop wall at 650°C to a depth of 25 μ m (1 mil) in a 4300-hr test.

Clearly, more reducing conditions are required to achieve long-term compatibility with austenitic stainless steels. We are presently operating a type 316 stainless steel loop test in which beryllium metal is being introduced as the redox buffer. The oxidation potential of the salt is constantly monitored and controlled by the use of voltammetry. The tentative conclusion from this loop test is that the corrosion rate of type 316 stainless steel can be held to an acceptable level by additions of a strong reductant such as beryllium metal.

3.3 COMPATIBILITY OF STRUCTURAL MATERIALS WITH BOILING POTASSIUM⁷ - J. H. DeVan

The suitability of iron-, nickel-, and cobalt-base alloys for use as structural materials in boiling-potassium systems is examined with

⁷Abstract to paper presented at the International Conference on Liquid Metal Technology in Energy Production, Seven Springs Resort, Champion, Pa., May 3-6, 1976; to be published in the proceedings of the conference.

particular attention to corrosion and mass transfer. Corrosion results for high-temperature sodium and potassium loops are summarized and related to the corrosion requirements of a boiling-potassium Rankine-cycle system. This analysis indicates that, in a recirculating boiler with an efficient vapor separator, mass transfer effects will be counter to those in an all-liquid system. Solution will be restricted to the cooler zones and deposition to the hottest zone (i.e., the boiler) as potassium is distilled off and dissolved material is left behind. This hypothesis is evaluated against the data available for boiling-potassium systems, and the results observed are consistent with the hypothesis. Mass transfer rates are notably lower than in all-liquid systems, and in the absence of contamination effects recirculating boilers have shown negligible corrosion.

3.4 CHEMICAL COMPATIBILITY PROBLEMS AMONG BLANKET, COOLANT, AND FIRST-WALL MATERIALS⁹ — J. E. Selle and J. H. DeVan

Potential coolants for fusion reactors include liquid lithium, molten LiF-BeF₂ mixtures (FLIBE), and helium. Blanket materials such as liquid lithium, FLIBE, or solid compounds such as LiAlO₂ will also be required for breeding tritium. Each combination of blanket, coolant, and first wall has its own special set of compatibility problems, and this paper will summarize the fundamental nature of these problems. Recent data on the corrosion of austenitic stainless steels by lithium indicate that intergranular penetration and thermal-gradient mass transfer will pose stringent limitations on operating temperature. However, there are indications that this temperature limitation can be offset through alloy modification and lithium purification. Molten FLIBE will preferentially oxidize chromium in type 316 stainless steel unless the oxidation potential of the salt is controlled by additions of reductants such as beryllium or chromium. Experimental data on lithium and FLIBE compatibility with stainless steels will be reviewed in the context of

⁹Abstract of paper presented at the 105th AIME Annual Meeting, February 22-26, 1976, Las Vegas, Nev., two-session symposium: Controlled Thermonuclear Reactors — Materials Research and Development.

fusion reactor requirements. Using helium as a coolant implies higher operating temperatures than for lithium or FLIBE, and consideration must be given to the thermodynamics of potential reactions between impurities in helium and structural materials. Experimental verification of this requirement will be presented and discussed with respect to vanadium and niobium alloys.

3.5 PROBLEMS IN SPECIFIC ENERGY SYSTEMS 5: FUSION REACTORS⁹ -

J. H. DeVan

1. The development of fusion reactors has only recently entered the conceptual design phase. Hence the identification of engineering parameters and material choices is still highly tentative.

2. Four different classes of stress-corrosion cracking problems are embodied in current fusion reactor concepts.

a. The use of liquid lithium as a tritium breeding material portends caustic stress-corrosion cracking problems analogous to those of liquid sodium in the Liquid Metal Fast Breeder Reactor (LMFBR).

b. Water is being considered as the first-wall coolant for early demonstration reactor experiments. Because of the free oxygen released by radiolytic decomposition, cracking of sensitized austenitic stainless steel weldments will be of definite concern unless reductants can be added to the water.

c. In the presence of certain impurities, liquid lithium can penetrate the grain boundaries of structural steels and pure niobium. The rate of penetration in some cases may be stress-enhanced.

d. Fusion devices are fueled by hydrogen isotopes, which can give rise to stress-cracking problems in the case of refractory metals.

⁹Summary and Conclusions from pp. 45-52 in *Stress Corrosion Cracking Problems and Research in Energy Systems* (Summary of Workshop held at ERDA-Germantown, Feb. 24-25, 1976), ERDA-76-98 (February 1976).

4. MAGNET DEVELOPMENT

C. C. Koch

4.1 STRESS EFFECTS IN SUPERCONDUCTORS - D. S. Easton and C. C. Koch

The development of toroidal superconducting magnets is the main goal of the Superconducting Magnet Development Program (SMDP). Our portion of this program is to study the effect of stress on the mechanical and superconducting properties of commercially available composite conductors. These data will be utilized in both the selection of a final conductor and the operation of any large magnetic device. Work has continued on previously reported¹⁻³ studies of mechanical properties of various commercial conductors. Tensile data at 4.2 K were measured on 20 different composites representing a variety of conductors from Supercon, Airco, IGA, MCA, and Alcoa. Results of one test each for these materials are summarized in Table 4.1 for Young's modulus, 0.2% yield strength, and ultimate strength. Little correlation was found between modulus and yield strength versus the physical characteristics of the conductors. Ultimate tensile strength values were generally higher for samples with low normal-to-superconductor ratios and higher number of filaments.

In situ J_c versus H measurements under stress at 4.2 K of a number of Nb-Ti and Nb₃Sn multifilamentary (MF) composites have been

¹D. S. Easton and C. C. Koch, "Tensile Properties of Superconducting Composite Conductors and Nb-Ti Alloys at 4.2 K," pp. 431-44 in *Shape Memory Effects in Alloys*, ed. by J. Perkins, Plenum Press, New York, 1976.

²D. S. Easton and C. C. Koch, "Mechanical Properties of Superconducting Nb-Ti Composites," presented at the 1975 International Cryogenic Materials Conference, Queens University, Kingston, Ontario, Canada, July 22-25, 1975. To be published in *Advances in Cryogenic Engineering*, Vol. 22, National Bureau of Standards, Boulder, Colorado.

³C. C. Koch, D. S. Easton, and S. B. Waters, "Stress Effects in Superconductors," *Controlled Thermonuclear Materials Technology Program Annu. Prog. Rep. June 30, 1975*, ORNL-5082, pp. 67-71.

Table 4.1. Tensile Properties at 4.2 K for a Variety of Composite Conductors^a

Sample	Cross Section		Pitch		Volume Ratio ^b	Number of Filaments	Young's Modulus		Strength, MPa (ksi)	
	(mm)	(in.)	(m ⁻¹)	(in. ⁻¹)			(GPa)	(psi)	0.2% Yield	Ultimate
75-1-A	1.37 × 2.9	0.057 × 0.114	40	1	2	160	108	15.6 × 10 ⁶	183 (26.6)	788 (114.2)
75-1-A-1	1.37 × 2.9	0.057 × 0.114	40	1	2	160	108	15.7	184 (26.7)	892 (129.3)
75-3-S	1.27 × 2.54	0.05 × 0.10	40	1	2.7	18	98	14.2	168 (24.2)	757 (109.8)
75-4-S	1.27 × 2.54	0.05 × 0.10	80	2	2.7	18	120	17.4	218 (34.5)	803 (116.5)
75-5-S	1.27 × 2.54	0.05 × 0.10	40	1	2.7	18	115	16.6	216 (31.3)	689 (99.9)
75-6-S	0.89 × 2.67	0.035 × 0.105	80	2	2.7	18	109	15.8	152 (22.1)	674 (97.7)
75-7-M	1.4 × 2.54	0.055 × 0.10	40	1	2.6	132	97	14.1	194 (28.1)	779 (111.7)
75-8-M	1.4 × 2.54	0.055 × 0.10	80	2	2.6	132	128	18.5	177 (25.6)	805 (116.0)
75-9-M	1.4 × 2.54	0.055 × 0.10	40	1	2.6	234	111	16.1	159 (23.0)	781 (113.2)
75-10-M	1.4 × 2.54	0.055 × 0.10	80	2	2.6	234	97	14.1	209 (30.3)	840 (121.8)
75-11-M	1.04 × 3.15	0.041 × 0.124	40	1	2.6	132	99	14.4	166 (24.1)	794 (115.1)
75-12-M	1.04 × 3.15	0.041 × 0.124	40	1	2.6	234	115	16.7	157 (22.7)	799 (115.8)
75-13-M	1.27 × 2.54	0.05 × 0.10	40	1	1.6	1,530	114	16.6	193 (28.0)	1099 (159.3)
75-14-I	1.27 × 2.54	0.05 × 0.10	80	2	1.6	1,530	135	19.6	257 (37.3)	981 (142.3)
75-15-I	1.02 × 3.05	0.04 × 0.12	40	1	1.6	1,530	92	13.4	244 (35.4)	1034 (150.0)
75-16-I	1.27 × 2.54	0.05 × 0.10	40	1	1.6	1,530	109	15.8	274 (39.8)	1187 (172.1)
75-18-S	1.27 × 2.54	0.05 × 0.10			c	1,591	74	10.7	662 (96.0)	1226 (177.7)
75-19-A ^d	0.81 × 2.54	0.032 × 0.10	40	1		25,000	161	23.4	207 (30.0)	424 (61.5)
75-21-L ^e	1.6 diam	0.063 diam	40	1		54	63	9.1	668 (96.9)	1107 (160.5)
75-22-I ^f	1.52 × 4.32	0.06 × 0.17	40	1		3,036	94	13.7	549 (79.6)	1309 (189.8)

^aThe filament material is niobium-titanium alloy unless otherwise specified.

^bRatio of copper matrix to superconductor.

^cCu, Cu-Ni alloy, and superconductor in proportions 0.5:1:1.

^dNb₃Sn superconductor.

^eAluminum matrix material.

^fCable.

completed.⁴⁻⁶ In this test the sample is mounted in a tensile testing machine so that a tensile load can be applied while the sample is submerged in liquid helium. Current leads are attached, and the sample is placed under load in a magnetic field. Strain is measured with a clip-on extensometer previously calibrated at 4.2 K, and Lorentz forces in the sample are supported by a Micarta backing block. For comparison, J_c in these tests is defined as the current density at an equivalent sample resistivity of $1 \times 10^{-13} \Omega\text{-m}$ and $5 \times 10^{-14} \Omega\text{-m}$ for the niobium-titanium and Nb₃Sn conductors, respectively. Values of J_c are referred to the entire conductor cross-sectional area.

In the case of the niobium-titanium conductors, J_c was reduced with stress or strain; however, with the exception of an aluminum alloy matrix composite, the degradation was less than 15% at strains to 2.0%. The aluminum matrix sample showed degradation of about 40% at 1.0% strain. The J_c value at zero strain was almost (within 1%) fully recoverable upon the release of stress in all samples, again with the exception of the aluminum matrix composite. Figure 4.1 shows the values of J_c as a function of strain for a number of the Nb-Ti composites. Sample identifications on curves refer to Table 4.1.

Several different configurations of MF Nb₃Sn conductors are shown in Fig. 4.2. The samples (a) through (e) are cables manufactured by the tin dip process.⁷ The basic 0.60-mm-diam strand contains 230 filaments (6 μm diam). Sample (f) is a 1 by 2 mm monolithic conductor

⁴D. S. Easton, R. E. Schwall, and W. A. Fietz, "Degradation of Multifilament Nb₃Sn Superconductor by Tensile Strain," paper presented at the Sixth Symposium on Engineering Problems of Fusion Research, San Diego, Ca., Nov. 18-21, 1975.

⁵D. S. Easton, "Stress Effects on Mechanical and Superconducting Properties of Commercial Conductors at 4.2 K," paper presented at the Symposium on Stress Effects in Superconductors, Vail, Colo., April 5, 1976.

⁶D. S. Easton and R. E. Schwall, "Performance of Multifilamentary Nb₃Sn Under Mechanical Load," to be published in *Applied Physics Letters*.

⁷R. M. Scanlan and W. A. Fietz, "Multifilamentary Nb₃Sn for Superconducting Generator Applications," *IEEE Trans. Magn.* **11**: 287 (1975). Samples tested were obtained from Intermagnetics General Corporation, Guilderland, New York.

Fig. 4.1. Critical Current Density Versus Strain for a Number of Niobium-Titanium Multifilament Conductors.

Fig. 4.2. Multifilamentary Nb₃Sn Conductors.

containing 24,781 filaments (4 μm diam) produced by the bronze diffusion technique.⁸

Figure 4.3 presents typical plots of J_c versus strain for the Nb_3Sn MF conductors. The letters in the legend refer to Fig. 4.2. The two curves shown for the monolithic conductor represent the limits of the behavior observed. Although the sample-to-sample repeatability for the cable conductor and for previously tested NbTi conductors has been about 5%, five of the adjacent lengths of the monolithic Nb_3Sn displayed widely varying characteristics. At present the source of the scatter is not known, and we can only set limits on J_c .

In Fig. 4.4 we illustrate the normalized J_c as a function of apparent strain. Most conductors show an initial increase in J_c when stress is applied and then a steady decrease. The approximate recovery of J_c when stress is released is indicated by the dashed line. With the exception of the low J_c monolithic material, all conductors show a substantial recovery of J_c when stress is released. The mechanism responsible for these strain-induced changes is still not understood, although similar results were obtained as early as 1955 by Buehler and Levinstein.⁹ Their work indicates that J_c is highest in unstrained Nb_3Sn and decreases under both tension and compression. Hence the initial increase of J_c with applied stress is attributed to relieving the compressive stresses due to differential thermal contraction of the surrounding bronze. Recently, Luhman and Suenaga¹⁰ have suggested that stress-related changes in T_c and J_c in bronze-clad Nb_3Sn wires were possibly

⁸E. Gregory et al., "Multifilamentary Nb_3Sn Conductor for Large Fusion Magnets," pp. 301-03 in *Fifth Intern. Magnet Conf., Rome, Italy, April 1975*, ed. by N. Sacchetti, M. Spadore, and S. Stripchi, Laboratori Nazionali del CNEN, Servizio Documentazione, Frascati, Italy. Sample tested was purchased from Airco, Inc., Murray Hill, N. J.

⁹E. Buehler and H. J. Levinstein, "Effect of Tensile Stress on the Transition Temperature and Current-Carrying Capacity of Nb_3Sn ," *J. Appl. Phys.* 36: 3856 (1965).

¹⁰T. Luhman and M. Suenaga, "Effects of Stresses Induced by Thermal Contraction of a Bronze Matrix on the Superconductivity Properties of Nb_3Sn Wires," *Bull. Am. Phys. Soc.* 21: 289 (March 1976).

Fig. 4.3. Critical Current Density as a Function of Apparent Strain for Nb₃Sn Conductors.

Fig. 4.4. Critical Current Normalized to Zero Strain Versus Apparent Strain for Nb₃Sn Conductors. Dashed line indicates approximate values obtained upon release of load.

due to a martensitic phase transformation in the Nb_3Sn . Sweedler et al.¹¹ have seen an increase in J_c following neutron irradiation of MF Nb_3Sn . These authors attributed the increase to an enhanced H_{c2} resulting from an increase in the normal state resistivity. A similar resistivity increase could result from strain. In any case the behavior of J_c under strain is likely to depend not only on the geometry of the conductor but also on the stress state of the surrounding matrix and on the strength of the bond between Nb_3Sn and the matrix.

Perhaps the most important result of these measurements is the rapid decrease in J_c with tensile strain. A uniform tensile strain (ϵ) of only 0.3% can cause as much as a 12 to 30% decrease. This would indicate that for reliable design of MF Nb_3Sn magnets the conventional H - T plot must be superceded by an H - T - ϵ characteristic.

4.2 PREPARATION OF SPECIFICATIONS FOR SUPERCONDUCTIVE WIRE — R. J. Beaver

We assisted the MFE Program in the preparation of specifications for rectangular, copper-base, superconductive wire containing filaments consisting of a niobium alloy with a titanium concentration, at the discretion of the supplier, in the range of 45 to 55 wt %. Because of the cancellation of the Eccentric Coil Test Project, the first specification for wire with the capability of 3700 A minimum at 4.2 K, measured in a transverse 8.0-T field at any equivalent resistivity of not more than 10^{-13} $\Omega\text{-m}$, was not used. It was revised to reflect a change to 10,000 A and will be used for procurement of superconductive wire for the Large Coil Segment Test Program.

4.3 ANALYSIS OF EDDY-CURRENT PHENOMENA IN THERMONUCLEAR EXPERIMENTS — C. V. Dodd and W. E. Deeds¹²

Work was continued on possible eddy-current problems involved in the design of thermonuclear experiments. When the large magnets are

¹¹A. R. Sweedler et al., "High-Energy Neutron Irradiation of Superconducting Compounds," pp. 422-35 in *Radiation Effects and Tritium Technology for Fusion Reactors* (Proc. Intern. Conf., Gatlinburg, Tenn. Oct. 1-3, 1975), Vol. II, ed. by J. S. Watson and F. W. Wiffen, CONF-750989.

¹²Consultant from The University of Tennessee, Knoxville.

turned on or off in a full-size thermonuclear experiment, important and possibly destructive eddy-current effects can occur.

Scaling laws were derived¹³ to permit accurate predictions of the important effects in large-scale apparatus from measurements made on small and relatively inexpensive models.

A two-dimensional relaxation program¹⁴ was developed to calculate various electromagnetic quantities in axially symmetric configurations with inhomogeneous permeability. Among the innovations in the program were the use of extra-fine lattices at the boundaries between media and the immediate replacement of the field component at a lattice point with its relaxed value, to save computer memory storage. The relaxation algorithms required for a three-dimensional relaxation program have also been derived, but the computer storage and running time problems appear to make accurate three-dimensional relaxations impractical at present.

A paper¹⁵ presenting the results of these investigations was presented at the Sixth Symposium on Engineering Problems of Fusion Research in San Diego, California.

For the complicated configurations of coils and conductors contemplated for thermonuclear experiments, it seems that the best combination of accuracy and economy at present is obtained by making measurements on small-scale models and using the scaling laws to predict the values for the large-scale apparatus.

¹³C. V. Dodd and W. E. Deeds, *Scaling Relations for Eddy Current Phenomena*, ORNL-5077 (November 1975).

¹⁴W. E. Deeds and C. V. Dodd, *A Two-Dimensional Relaxation Program for Systems with Inhomogeneous Permeability*, ORNL/TM-5333 (Jan. 1976).

¹⁵W. E. Deeds and C. V. Dodd, "Analysis of Induction Phenomena in Thermonuclear Experiments," pp. 961-63 in the *Proc. 6th Symp. Engineering Problems of Fusion Research, San Diego, Calif., Nov. 18-21, 1975*, IEEE Nuclear and Plasma Sciences Society, IEEE Pub. No. 75CH1097-5-NP.