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**STRUCTURAL EFFECTS OF NIOBIUM ION
BOMBARDMENT OF NIOBIUM FOILS - A PROGRESS REPORT***

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STRUCTURAL EFFECTS OF NIOBIUM ION BOMBARDMENT ON NIOBIUM FOILS - A PROGRESS REPORT*

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ABSTRACT

This paper deals with the effects which 14 MeV neutron radiation is likely to have on the engineering properties of thermonuclear reactor materials. Other papers^{1,2} have dealt with various aspects of this and related problems. This work enumerates and compares the damage mechanisms associated with fusion neutron irradiations with particular emphasis on their effect on reactor operating temperatures and service lifetimes.

There is some considerable difficulty anticipated in attaining the required fluence of 14 MeV neutrons for radiation effects testing prior to the existence of the reactors themselves. This leads to consideration of other means for experimental simulation of the desired effects. We favor an approach to simulation based on the bombardment and simultaneous testing of thin foils. Such a program of evaluation has been initiated at L.R.L. An assessment is given of the surface physics complications which are believed to be present. The technology for the creation of representative thin foils is discussed and an outline for in-situ creep testing is presented. Electron microscopy, diffraction and X-ray production methods of specimen characterization and experiment control are considered.

INTRODUCTION

In a recent paper¹ one of the authors of this paper reported on the projected effects which fusion neutrons might have on the engineering properties of fusion reactor structural materials. At that time it was also proposed that heavy (self) ion bombardment of thin foils be considered as a means of simulating certain of these effects at an earlier time and at considerably less expense than would be possible by direct neutron bombardment. A modest program to evaluate the usefulness of this proposal has been undertaken as a research project of the Mechanical Engineering Department at Lawrence Radiation Laboratory. In this paper, we would like to enumerate the reasons which we feel make the original proposal a viable and worthwhile one and to report on the progress which we have made toward its implementation.

* This work performed under the auspices of the U. S. Atomic Energy Commission.

RADIATIONS IN FUSION REACTORS

A number of investigators²⁻⁶ have pointed out that the structure of thermonuclear reactors will be subject to a considerable variety of possible forms of radiation bombardment. These radiations will originate both within the reacting plasma and the surrounding fertile or structural materials. Both long and short range forms of wave energy and charged and neutral particles can be anticipated. A schematic of some of these radiation forms and their means of production is shown in Figure 1.⁶

We can conveniently divide these radiation forms first into two categories; those which tend to be affected by the magnetic field confining the plasma, i.e., the charged particles, and those which are not, i.e., the wave form energies and neutrals. The effect which the former will have on their surroundings is strongly dependent on the nature of the magnetic confinement; while the latter would tend to be independent of the mode of confinement. Both would, of course, be dependent on the nature of the fusion reaction or fuel cycle employed.

In a similar manner, we can characterize the charged particle radiation as tending to be short ranged and to be of concern principally with regard to possible surface interactions. Photon and neutron radiation on the other hand may be either short or long ranged. It is a further convenience therefore to make a further distinction as to whether the radiation interactions take place principally at the surfaces or possibly deep within the structural or fertile materials of the reactor. Reactions of either kind may give rise to secondary radiations although those which are of greatest interest to the engineer tend to be associated with neutron events involving displacements and transmutations. (Fig. 2)

Early thermonuclear reactor systems are expected to rely principally on the deuterium - tritium (D-T) reaction for which the order of 80% of the reaction energy is released in the form of energetic (14 MeV) neutrons. These energetic

FIG. 1 RADIATION DAMAGE MECHANISMS IN FUSION REACTORS

Fig. 2 Transmutation Scheme for Niobium
(From A.E.R.E. R-5837)

two orders of magnitude. This is a conclusion which may prove of considerable consequence both to the design and more especially the lifetime of fusion reactors.

Perhaps equally significant is the observation that the predicted values for combined neutron energy and dose are well beyond either our present experiences or ability to duplicate.

As a consequence of the referenced calculations¹ it was proposed that one of the principal results of neutron interaction, namely production of lattice atom displacements, might at least in principle be simulated by self-ion bombardment of thin foils to produce direct excitation of secondary or target atoms. (Fig. 4) These excited secondary atoms would then be expected to produce displacement cascades of the same nature as those which have been observed to be created by neutron bombardment.

THE SIMULATION OF NEUTRON DAMAGE IN FOILS

We direct our attention at least initially toward those refractory metals which appear to be attractive candidates as fusion reactor structural materials. In this instance, we have singled out niobium for first attention since it is a tractable engineering material, has proven to be relatively compatible with most prospective heat transfer and fertile materials and is likely relatively resistant to damage by surface bombardments. The predicted range of the displacement cascade due to a 14 MeV incident neutron in niobium is of the order a few hundred to one thousand angstroms (Å), depending on the orientation and degree of order of the target. This range effectively limits the maximum thickness of specimen which can be bombarded with reasonable uniformity of displacement at a single time. The first order remaining question therefore is whether foils of this order of thickness can be prepared, bombarded under representative service conditions and the results interpreted in terms of the behavior of bulk specimen under service conditions.

Energy of Primary Neutron Resulting from Fusion (fu) and Fission (fi) Neutrons

	A	Z	E _n (MeV)	fu	fi	E _n (MeV)	fu	fi
He	4	2	14.1	155	15.5	2540	254	
C	6	12	1.1	8.6	91	9.1	3060	306
Al	13	27	1.1	11.7	19.7	2.0	978	97
Ti	22	48	1.1	5.4	6.4	0.64	565	56.5
Ni	28	58	1.2	4.3	4.3	0.43	466	46.6
Nb	41	93	1.1	5.2	3.7	0.17	296	29.6
Ta	73	181	1.1	5.3	0.47	0.95	155	15.5

First-Neutron-Only Displaced Atoms (D)							
	Be	C	Al	Ti	Ni	Nb	Ta
Fusion	230	230	530	1050	2050	2730	1050
Fission	100	100	400	950	400	230	150

FIG. 4 SCHEMATIC OF FOIL BOMBARDMENT ASSEMBLY

FIG. 5 FOIL BOMBARDMENT SPECIMEN

THE PRODUCTION AND CHARACTERIZATION OF FOILS

The technology for the production of thin metal foils has been investigated for some time and rapid advances are still being made. Since in this instance, we have addressed ourselves to the effects of bombardment on the engineering properties of the materials, we have chosen to attempt the fabrication of free standing foil creep test specimen. (Fig. 5a) The mechanical properties of thin foils have been reported on by a number of investigators.^{8,9} In general, thin foils of ductile metals have shown short time strengths of the same order as the bulk material down to thicknesses of a few thousand angstroms. For thinner foils where a single grain spans the thickness the measured strengths appear to increase in a

predictable fashion by about 50% for each factor of two reduction in thickness.

Our foils are deposited by condensation of the metal vapor upon a soluble (salt) substrate; roughly sectioned and floated free upon the surface of a water bath from which they are lifted using the actual test frame. (Fig. 5b) Electron microscopy and diffraction (Fig. 6) has shown the as-deposited structure to consist mainly of small disordered grains with fairly frequent tear like defects. Subsequent heating (annealing) results in rapid grain growth, increased orientation and "healing" of the previously observed defects. (Fig. 7)

Fig. 6 ELECTRON MICROGRAPHS OF AS-DEPOSITED NIOBIUM FOILS

Fig. 7 ELECTRON MICROGRAPHS OF ANNEALED NIOBIUM FOIL

BOMBARDMENT AND MECHANICAL TESTING

Our intent is to subject stressed foils of niobium to bombardment while at temperatures up to 1150 or 1450 K for ion doses ranging from 10^{15} to 10^{17} ions per cm^2 which are equivalent to neutron doses of approximately 10^{19} and 10^{23} respectively. The accelerator chosen for this initial work is a direct current type with a nominal beam energy of 100 KV and current of a few microamperes using a sputtered metal source. We have equipped the target region of this accelerator with fixtures suitable for supporting, manipulating and heating the installed specimen. A schematic of the installation was shown above in Figure 4 and some of the associated hardware in Figure 8. During the bombardments, X-rays produced by the bombardments will be monitored both as a means of impurity detection and hopefully as a means of continuous determination of foil thickness.¹⁰

Fig. 8a - Mounting 1000 Å Foil

Fig. 8b - 100 KeV Accelerator (upper)
Fig. 8c - Foil Target Apparatus (lower)

POST BOMBARDMENT EXAMINATION

Exposed specimen will be measured optically for elongation and then compared by means of transmission electron microscopy with both unbombarded and unstressed control specimen run thru the same temperature and atmosphere history. Aside from direct measurement of the foil elongation or rupture strength vs. dose and temperature, the principal recorded quantity will be the density of resolvable bombardment produced displacement structures. Of particular interest will be any evidence of equilibrium or saturation levels of defects with increasing temperature and dose.

SPUTTERING, TRAPPING AND IMPURITIES

There are some aspects in which self ion bombardment is unlikely to simulate fusion neutron irradiation. Some of the more significant would appear to be the following:

1. Sputtering of the foils by incident ions: The data available at this time would indicate that at low temperatures, the sputtering coefficient of niobium for niobium at the chosen bombardment energy is of the order 3 to 4 sputtered atoms per incident ion.¹¹ At this rate, the recession of the surface of the foil should proceed at a rate of roughly one angstrom for each 10^{14} ions per cm^2 dose (~ 100 micro amu/cm^2). Such a rate of recession may well preclude reaching the largest doses of interest (above say 10^{16} per cm^2) without simultaneous redeposition of new material. We presently consider the possibility of backside sputtering unlikely since incident angles are unlikely to be oriented with channeling directions and highly ordered structures are likely to be disordered early in the bombardment sequence.
2. The trapping coefficient of the foil for the incident ions is assumed to be unity. We believe this value is justified both because of the thermodynamic conditions required for escape are unlikely to occur and because the highest doses anticipated are below those for which this effect is likely to be activated.¹²
3. Probably the greatest tendency for stabilization of radiation induced defect structures in both the neutron and ion bombarded materials will come from impurity atoms, especially gasses either born within the solid thru transmutation reactions or induced thru entrapment by bombardment, diffusion or chemical reactions. In the case of the simulated irradiation thru ion bombardment the potential problem will be attacked thru careful control of the chemistry of both the specimen and environment. In the case of the prototype neutron irradiation, the effect will depend not only on chemical impurity considerations but also on the tendency for production of mobile or reactive species thru transmutation reactions. Present estimates place the probability that the latter effect will be limiting at a relatively low level.^{13,6}

CLOSURE

We have chosen to address ourselves to the question of kinetic interactions of fusion neutrons with reactor materials because we feel that this is the area of radiation damage related to fusion damage in which the most valuable contribution can be made at this time. We base this first on the likelihood that the D-T reaction will be the first successfully exploited and on the relative uncertainty which questions of containment and high energy neutron capture reactions extend over other enumerable radiation problems. Finally, present estimations¹ indicate that the kinetic damage problem is the most likely to be limiting in terms of engineering design and economic lifetime of early fusion reactors, while transmutation reactions are more likely to govern the efficiency and operating economy.^{1,5}

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