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RADIATION DAMAGE TO REFRACTORY METALS AS RELATED TO THERMIONIC APPLICATIONS

F. W. Wiffen

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RADIATION DAMAGE TO REFRACTORY METALS AS RELATED TO THERMIONIC APPLICATIONS

F. W. Wiffen

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CONTENTS

Page

Abstract 1

Introduction 1

Production and Accumulation of Radiation Damage 2

Low-Temperature Damage 3

Intermediate-Temperature Damage 7

High-Temperature Damage 15

The Effect of Radiation Damage on Properties 16

Swelling of bcc Metals 16

Electrical Resistivity of Irradiated Metals 19

Mechanical Properties of Irradiated bcc Metals 20

Summary — The Effect of Irradiation on Thermionic Reactor Components 22

Emitters 22

Collectors and Electrical Conductors 23

Fuel Element Sheath 23

Acknowledgments 24

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F. W. Wiffen

ABSTRACT

The effects of fast neutron irradiation on the body-centered cubic refractory metals are reviewed with emphasis on materials and conditions typical of thermionic applications in space power systems. Changes produced in the microstructure are characterized as a function of irradiation temperature. The effects of fluence, alloying, and purity are examined where data are available. Swelling and changes in the mechanical and electrical properties produced by neutron irradiation are briefly reviewed. The major effects expected are in the collector and cooler portions of the emitter in a thermionic diode where swelling, electrical resistivity increases, and ductility losses are anticipated during reactor operation.

INTRODUCTION

This paper will give a brief review of the effects of fast neutron irradiation on the properties of the body-centered cubic (bcc) refractory metals, with emphasis on irradiation effects found under conditions similar to those expected in thermionic elements of space reactor systems. Data on bcc metals irradiated at temperatures of interest are sparse and very often the effects of irradiation and material parameters must be predicted from the more extensively studied face-centered cubic (fcc) metals with only a very few bcc data points as reference. Such theoretical and empirical models as have been developed to explain effects, mainly in stainless steels, have not been tested for bcc systems, so are not yet of help in predicting reactor behavior of these metals.

Radiation damage will be reviewed by first examining the atomic scale processes that occur during irradiation. This will develop a composite picture of the behavior of irradiated bcc metals as a function of

the irradiation temperature. The temperature range 0°K to the metal melting point will be divided into low, intermediate, and high regions. Most of the description of the damage state relies on the results of postirradiation examination of the microstructure by transmission electron microscopy (TEM). The changes produced in physical and mechanical properties by the radiation damage will then be examined and discussed in terms of the microstructural information. Where possible, these results will be related to the expected behavior of thermionic diode and structural components of a space power reactor.

The reference reactor parameters used in this paper were taken from the 100 kW(e) Thermionic Power System described by Gietzen.[1] The reactor, designed for a 5-year lifetime, will have a maximum integrated neutron fluence in the core that is calculated to be 2.5×10^{21} neutrons/cm^2 (> 1.0 MeV). This corresponds to at least 5.0×10^{21} neutrons/cm^2 (> 0.1 MeV). The thermionic diode emitter is tungsten, operating at an average temperature of 1660°C. The collector, at 700°C, is niobium or molybdenum. The outer fuel element sheath is Nb–1% Zr and will operate over the range of coolant temperatures 600 to 700°C. These operating conditions put the components into the intermediate- and high-temperature regions of radiation damage.

PRODUCTION AND ACCUMULATION OF RADIATION DAMAGE

Under neutron bombardment two basic processes can occur. An atom struck directly is displaced from its lattice site if the energy transferred to the struck atom exceeds approximately 25 eV. The unoccupied position is called a vacancy. The struck atom moves through the lattice and gradually loses energy until it comes to rest between equilibrium sites in the lattice. This atom is now referred to as an interstitial. If the energy transferred to the atom originally displaced by the neutron bombardment is high, secondary atom displacements are produced as this atom strikes other lattice atoms. The result is a damage-rich zone

[1]A. J. Gietzen, pp. 151–158 in IEEE Conference Record of 1970 Thermionic Conversion Specialist Conference, October 26–29, 1970, The Institute of Electrical and Electronics Engineers, New York, 1970. (70 C 53–ED)

called a displacement cascade, consisting of a central volume with a high vacancy concentration surrounded by a shell with a high interstitial concentration. The second basic process in neutron irradiation is capture of a neutron by a lattice atom. This capture can result in nuclear transmutations of the type (n,α), (n,β), and (n,p) that produce new chemical species in the lattice. Important effects have been attributed to products of these transmutations, with helium from (n,α) reactions believed to be especially important in affecting swelling and ductility.

The basic neutron interaction processes are temperature independent. The behavior of the vacancies, interstitials, and transmutation products once created is very strongly temperature dependent and the damage state produced in metals is governed primarily by the irradiation temperature. The rest of this section is devoted to a review of the microscopic scale effects of irradiation. For convenience, the discussion will be divided in terms of irradiation temperature. Three temperature regions are considered: (1) Low temperatures, where the irradiation temperature, T, is in the range $T < 0.3\ T_m$ (T_m is the absolute melting temperature of the metal considered). (2) Intermediate temperatures, $0.3 < T/T_m < 0.5$. (3) High temperatures, $T > 0.5\ T_m$.

Low-Temperature Damage

The effect of irradiation at temperatures below $0.3\ T_m$ has been investigated in much more detail than effects in the other two temperature ranges. At the lowest temperatures in this region, say 0 to 20°K, no component of the irradiation-produced damage is able to migrate under the influence of thermal energy alone. The damage produced is "frozen-in" except for limited rearrangement that takes place within displacement cascades immediately following their creation. Observation by TEM confirms that cascade rearrangement does occur at 20°K or lower.[2] The amount of damage produced by irradiation has been shown to saturate at relatively low concentrations, approximately 0.2% vacancy-interstitial

[2] L. M. Howe, J. F. McGurn, and R. W. Gilbert, Acta Met. 14, 801–820 (1966).

pairs in several fcc metals.[3] In tantalum[4] electron irradiation results show that saturation of damage production occurs at approximately the same defect concentration, 0.2%. The saturation occurs when the defect concentration is great enough that newly created defects all form within the spontaneous recombination volume of existing defects.[5]

For irradiation at slightly higher temperatures, by 100°K for most of the bcc metals,[6] interstitial atoms can migrate freely in the lattice. The migrating interstitials will annihilate by recombination with vacancies, will be removed at sinks or traps (such as impurities, precipitates, grain boundaries, or dislocations), or will react with other interstitials to form stable defect clusters. The rate of damage accumulation will be much reduced relative to irradiation at the very low temperatures. The damage that does accumulate is now of a different form, consisting mainly of isolated single vacancies and interstitial clusters. The interstitial clusters are two dimensional; they form disks or partial extra atom planes bounded by dislocations. These are visible as dislocation loops when sizes exceed the resolution limit of the microscope.

As the irradiation temperature is further increased, vacancies, too, become mobile. The exact temperature range in which vacancy migration occurs has not been conclusively established, with cases having been made for vacancy migration starting at about 0.15 T_m or at about 0.22 T_m. The arguments and data are reviewed by Nihoul[6] and by Schultz,[7] who both conclude that the most recent data in tungsten and molybdenum are best explained by vacancy migration occurring near 0.22 T_m. Mobile vacancies will suffer the same fates as interstitials – recombination, trapping, loss at sinks, and clustering with other vacancies. As with interstitials, the vacancies in this temperature range form two-dimensional clusters that are identified by TEM as dislocation loops.

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[3]G. Burger, H. Meissner, and W. Schilling, Phys. Status Solidi 4, 281–288 (1964).

[4]D. Meissner and W. Schilling, Z. Naturforsch. 26a, 502–511 (1971).

[5]H. J. Wollenberger, pp. 215–254 in Vacancies and Interstitials in Metals, edited by A. Seeger et al., North-Holland, Amsterdam, 1970.

[6]J. Nihoul, ibid., pp. 839–888.

[7]H. Schultz, Mater. Sci. Eng. 3, 189–219 (1968–1969).

Damage clusters formed by precipitation of either vacancies or interstitials have been reported on for a great variety of material and irradiation conditions. While these clusters have often been identified as "black spots," these spots are believed to be dislocation loops too small for the loop features to be observed by TEM. Analysis of the nature of the clusters is difficult and has in some cases been contradictory but it is clear that, at least in the upper part of this temperature range, both vacancy and interstitial loops form. Where both types of clusters have been identified in the same sample, the vacancy loops are much smaller and more numerous than the interstitial loops.[8] The number of vacancies and interstitials contained in the two types of loops are approximately equal. Irradiation temperature has a strong effect on the damage. For samples irradiated to a constant fluence, the number of defect clusters decreases and the average cluster size increases as the irradiation temperature is increased.[8] At a fixed temperature the cluster concentration increases with increasing fluence, in the low fluence range. Saturation of the amount of damage accumulated in the lattice has been demonstrated in at least one material. In molybdenum irradiated at 70°C, saturation was observed in length change and a maximum in lattice parameter change.[9] A similar behavior was found for the electrical resistivity increase produced by irradiation. Keys et al.[10] found a maximum in the resistivity increase for irradiation at this temperature at a neutron fluence of 5 to 10×10^{19} neutrons/cm^2 (> 1.0 MeV). Only half the maximum increment remained at a fluence of 2×10^{21} neutrons/cm^2. Direct examination of similar specimens by TEM[11] showed a high concentration of small black-spot defect clusters (unresolvable loops) at the lower fluences and a lower concentration of small clusters and larger aggregates at 1×10^{21} neutrons/cm^2, well above the

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[8] J. L. Brimhall and B. Mastel, Radiation Effects 3, 203–215 (1970).

[9] H. E. Kissinger, J. L. Brimhall, B. Mastel, and T. K. Bierlein, pp. 681–683 in International Conference on Vacancies and Interstitials in Metals, Jül-Conf-2 (Vol. II), KFA, Jülich, 1968.

[10] L. K. Keys, J. P. Smith, and J. Moteff, Phys. Rev. Letters 22, 57–60 (1969).

[11] R. C. Rau, J. Moteff, and R. L. Ladd, J. Nucl. Mater. 40, 233–235 (1971).

fluence for maximum damage found by the resistivity measurements. This reduction in the number of clusters suggests that the loops grow and interact (possibly by loop glide) to form dislocation networks or larger loops, resulting in a reduction of the damage in the lattice. This saturation of damage production has not yet been demonstrated for molybdenum at higher temperatures nor has it been demonstrated for other bcc metals.

An example of the damage seen in molybdenum by TEM examination is shown in Fig. 1. In this sample irradiated to 2.5×10^{22} neutrons/cm^2 (> 0.1 MeV) at 425°C (0.25 T_m), there is a very high concentration of small defect clusters. A few of the clusters can be resolved as loops but most are too small to resolve. There is a tendency for the loops to be aligned in "strings," similar to the "rafting" of loops seen in molybdenum at lower fluences.[8]

The effect of alloying on the irradiation response of bcc metals has not been studied in any detail. The comparison of molybdenum and

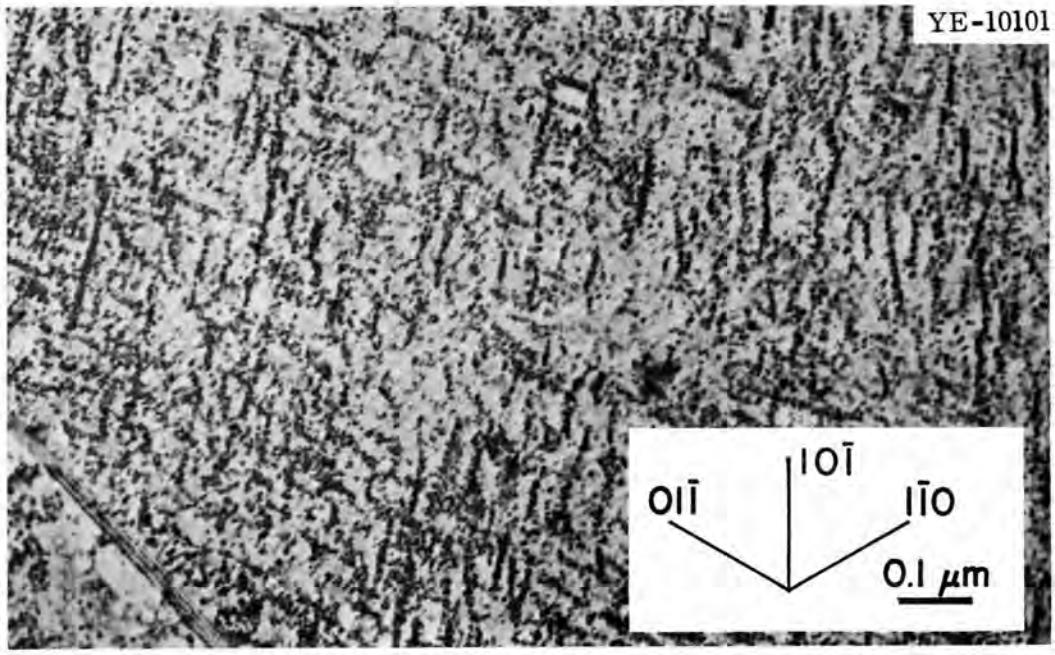

Fig. 1. Microstructure of Molybdenum Irradiated to 2.5×10^{22} neutrons/cm^2 (> 0.1 MeV) at 425°C. The "black spots" are unresolved dislocation loops.

two molybdenum-base alloys irradiated at 70°C showed two extreme effects.[11] Transmission electron microscopy showed very similar cluster concentrations in molybdenum and Mo—0.5% Ti at two fluences while Mo—50% Re irradiated to the same lower fluence contained no defect clusters. There has been no effort made yet to examine in detail the effect of alloying nor to explain the observed extremes.

Intermediate Temperature Damage

This irradiation temperature range, defined by the approximate limits $0.3 < T/T_m < 0.5$, is the range in which most reactor structural components see service. Ambient temperature in research reactors falls below this range for irradiation of bcc refractory metals. This increases the difficulty of experimental irradiation and, as a result, data for bcc metals are limited. Both vacancies and interstitials can migrate freely at these intermediate temperatures and most of the defects produced are lost by recombination. A few of the defects do survive, however, and form very stable defect clusters.

In the lowest temperature range both vacancies and interstitials, when mobile, formed two-dimensional defect clusters — dislocation loops. The intermediate temperature range is defined to cover the range over which a new type of vacancy cluster, the void, is stable. Fast-neutron-produced voids, first identified in stainless steel by Cawthorne and Fulton,[12] are holes or cavities formed by the precipitation of vacancies. The voids form instead of the two-dimensional vacancy loops formed at lower temperature, while the interstitials continue to precipitate as dislocation loops.

Interstitial loops are disks of extra atoms bounded by dislocations. The host lattice is expanded to accommodate them. Large vacancy loops are formed by removal of part of a lattice plane (as the vacancies condense) and can be accommodated by a partial collapse of the metal lattice in the central part of the vacancy loop. As a result of this behavior,

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[12] C. Cawthorne and E. J. Fulton, p. 446 in The Nature of Small Defect Clusters, Vol. 2, ed. by M. J. Makin, AERE-R5269, Her Majesty's Stationery Office, London, 1966.

simultaneous formation of interstitial and vacancy loops at low irradiation temperatures do not result in a large distortion of the overall sample. In the intermediate temperature range, however, vacancies precipitate to form voids and there is no lattice accommodation. The damage state comprised of voids plus interstitial loops results in a net swelling of the metal sample. This swelling can have technologically important results. The void component of this damage has been examined in considerable detail in the last five years and the results have been summarized in two 1971 conferences.[13,14] The interstitial loop component of the damage has been relatively neglected.

Typical void microstructures for refractory metals are shown in Figs. 2 through 4. The voids are imaged in TEM by using absorption

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[13] Voids Formed by Irradiation of Reactor Materials, Proceedings of the British Nuclear Energy Society European Conference held at Reading University on 24 and 25 March 1971, ed. by S. F. Pugh, M. H. Loretto, and D.I.R. Norris, British Nuclear Energy Society, 1971.

[14] Radiation-Induced Voids in Metals, Proceedings of an International Conference held in Albany, New York, June 9–11, 1971; to be published by USAEC.

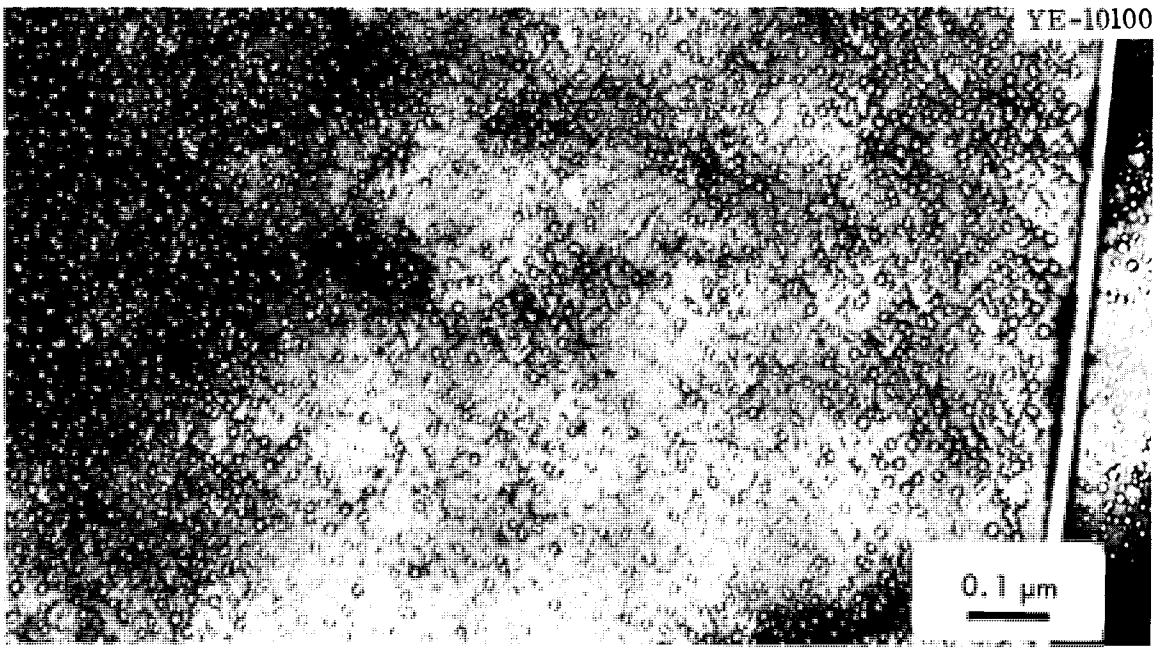

Fig. 2. Voids in Molybdenum Irradiated to 2.5×10^{22} neutrons/cm^2 (> 0.1 MeV) at 790°C. The concentration of voids is 5.4×10^{16} cm^{-3}, with average diameter 72 Å.

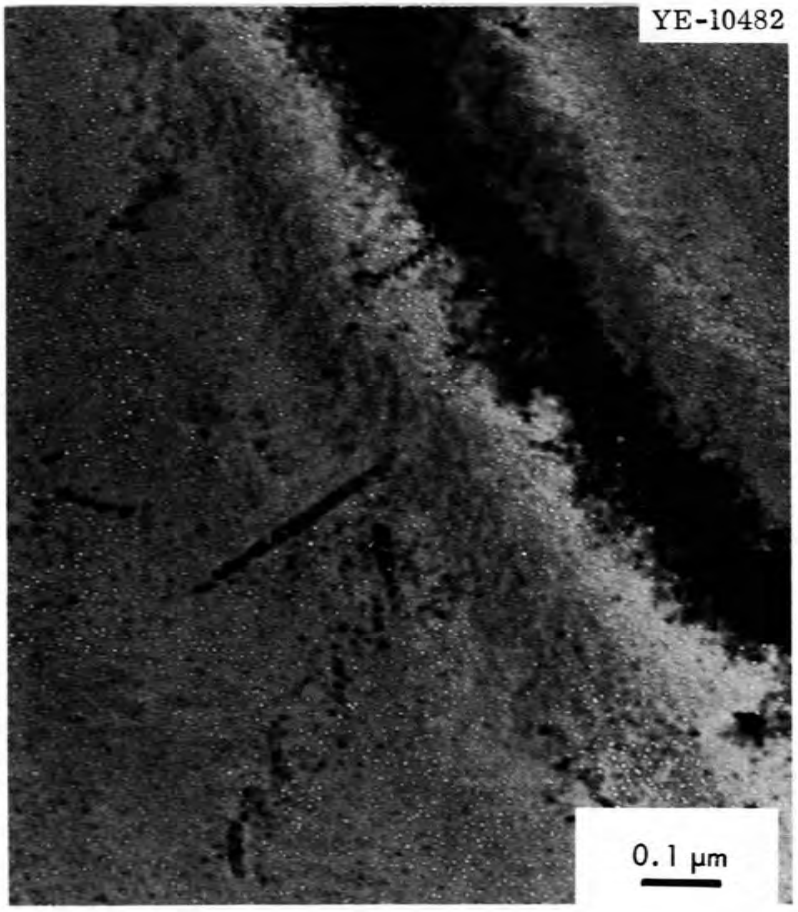

Fig. 3. Niobium Irradiated at 425°C to 2.5×10^{22} neutrons/cm^2. Void parameters given in Table 1.

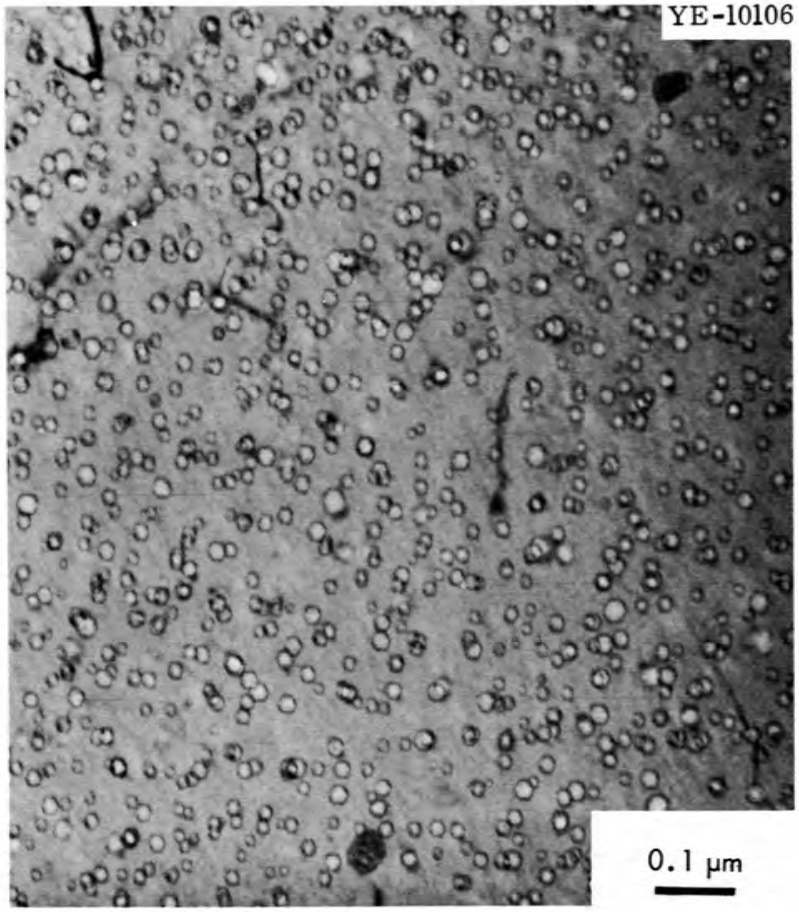

Fig. 4. Niobium Irradiated at 790°C to 2.5×10^{22} neutrons/cm^2. Void parameters given in Table 1.

Table 1. Void and Swelling Data for Neutron-Irradiated Niobium and Tungsten

Material	Irradiation		Void Parameters			Reference
	Temperature (°C)	Fluence (neutrons/cm^2)	Concentration (voids/cm^3)	Average Diameter (Å)	Volume Fraction (%)	
Niobium[a]	425	2.5×10^{22}	1.6×10^{17}	70	3.1	b
	585	2.5×10^{22}	2.1×10^{17}	71	4.8	b
	790	2.5×10^{22}	2.8×10^{15}	186	1.04	c
Niobium[a]	470	3.9×10^{20}	3×10^{16}	20	0.01	d
	650	5.5×10^{20}	8×10^{15}	60	0.09	d
	750	4.1×10^{20}	5×10^{14}	125	0.05	d
Niobium[a]	600	5×10^{20}	1×10^{16}	50–60	0.1–0.2	e
	900	5×10^{20}	0			e
Nb–1% Zr[a]	790	2.5×10^{22}	$< 2 \times 10^{14}$	510	< 1.4	c
Tungsten[f]	1000	1.5×10^{20}	2×10^{16}	37	0.04	g
	1300	1.6×10^{20}	1×10^{15}	100	0.05	g
Tungsten[a]	900	5×10^{20}	1×10^{16}	50–60	0.1–0.2	e

[a]Fluence for neutrons > 0.1 MeV.

[b]F. W. Wiffen, unpublished data.

[c]F. W. Wiffen, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

[d]J. D. Elen, G. Hamburg, and A. Mastenbroek, J. Nucl. Mater. 39, 194–202 (1971).

[e]Y. Adda, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

[f]Fluence for neutrons > 1.0 MeV.

[g]R. C. Rau, R. L. Ladd, and J. Moteff, J. Nucl. Mater. 33, 325–327 (1969).

contrast conditions; as a result the voids show up as white spots on the micrographs. Comparison of Figs. 1 and 2 shows the basic difference between the microstructural damage in the low- and intermediate-temperature regions. Only dislocation loops and unresolved "black spots" are seen for the molybdenum irradiated at 0.25 T_m (Fig. 1) while in the molybdenum irradiated to the same fluence at 0.37 T_m (Fig. 2) the most obvious

component of damage is the void. In Figs. 2 through 4 the void population is accompanied by a dislocation loop structure, on a much larger scale than the void structure, but the dislocation structure is not visible for the TEM conditions in these figures.

The phenomenon of void formation is not well understood. Void nucleation, in particular, has not been adequately modeled theoretically. Nucleation seems to require the presence of some gas atoms in the embryo void to stabilize the three-dimensional void against collapse into a two-dimensional loop.[15] This view is supported by the demonstration in several systems that accelerator-injected helium can increase the void nucleation rate. In reactor irradiation, helium from (n,α) reactions is believed to assist in nucleation, and it is suspected, but not proven, that other gases are instrumental in void nucleation.

The excess of vacancies over interstitials nceessary for both nucleation and growth of voids is established and maintained during irradiation by a preferential attraction of dislocation loops for interstitials over the attraction for vacancies.[15]

The general features of void formation as a function of irradiation parameters have been established in several systems. Within the range of temperatures in which voids form, the void concentration decreases with increasing irradiation temperature at a fixed neutron fluence. This has been confirmed for several bcc metals by Wiffen[16] and Elen et al.[17] Void concentrations can be very high in bcc metals, with concentrations as high as 2×10^{17} voids/cm^3 observed. Void sizes are also dependent on irradiation temperature, with sizes increasing as temperatures increase. The influence of irradiation temperature on voids in niobium is illustrated by Figs. 3 and 4.

The opposing temperature dependence of void concentration and void size results in a temperature dependence of swelling with a peak

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[15]R. Bullough and R. C. Perrin, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

[16]F. W. Wiffen, ibid.

[17]J. D. Elen, G. Hamburg, and A. Mastenbroek, J. Nucl. Mater. 39, 194–202 (1971).

intermediate in the void formation range. The dependence of swelling on irradiation temperature is shown schematically in Fig. 5. In a real system this curve may be less symmetric and the curve location does not occur at the same homologous temperature (T/T_m) for all bcc materials. Available data suggest that voids form at lower homologous temperatures in V, Nb, and Ta than in Mo, although this conclusion remains tentative. The shape and location of the swelling versus irradiation temperature curve are probably also dependent on both neutron flux and fluence. The comparison of damage produced by neutron irradiation and by ion bombardment at several different atom displacement rates demonstrates that the whole swelling curve shifts to higher temperatures as the damage rate (flux) is increased.[18] At a constant flux, data on stainless steel[19] show that, in that system at least, the upper temperature for void

[18]G. L. Kulcinski, J. L. Brimhall, and H. E. Kissinger, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

[19]E. E. Bloom, An Investigation of Fast Neutron Radiation Damage in an Austenitic Stainless Steel, ORNL-4580 (August 1970).

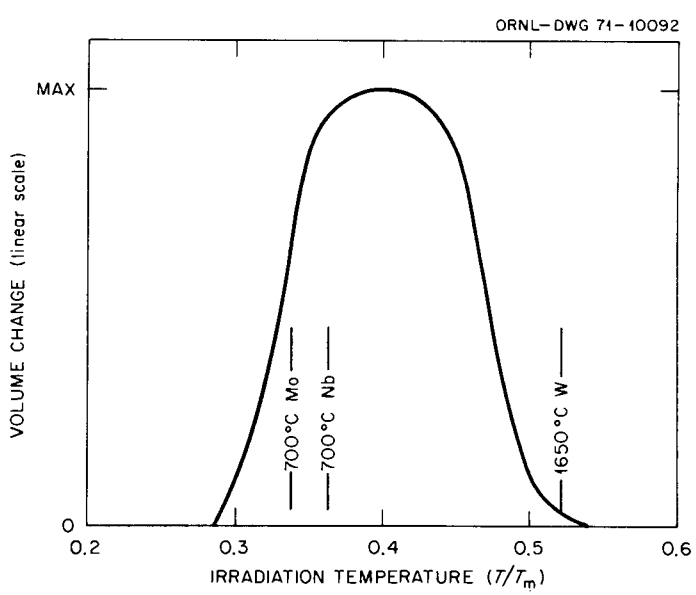

Fig. 5. Idealized Dependence of Irradiation-Produced Swelling on Irradiation Temperature. Operating temperatures for typical thermionic diode materials are shown.

formation increases with increasing fluence. No metal has been studied in sufficient detail to more fully determine these flux and fluence effects.

The effect of fluence at a fixed irradiation temperature has not been determined for any bcc metal. In fcc metals three different types of fluence dependence have been identified. In one grade of nickel[20] the void concentration was found to be independent of fluence, but the average void size to increase with fluence. In stainless steel[21] the concentration increased with fluence, but the void size increased only very slowly with fluence; in aluminum[22] both the void concentration and size increased with increasing fluence. The aluminum void concentration showed a tendency toward saturation at less than 10^{15} voids/cm^3 at a fluence of 1×10^{22} neutrons/cm^2. The very high void concentration observed in bcc metals[16,17] suggests possible saturation at low fluences, but this has not been substantiated. All of the available void parameters for neutron-irradiated niobium, tungsten, and molybdenum are collected in Tables 1 and 2; examination of this data shows the values typically observed in irradiated bcc metals. The swelling data will be discussed further in a following section.

The effect of alloying on void formation, examined in very few bcc metals, is not the same in all bcc systems. For a restricted range of irradiation conditions, it has been shown that high concentrations of small voids form in V, Nb, and Mo while no voids are found in V–3 to 20% Ti (refs. 16,23). Niobium–1% zirconium contained fewer and larger voids than niobium, while total void volumes remained nearly equal.[16] In a comparison of molybdenum-base alloys, one condition was found for which similar void populations formed in molybdenum and Mo–0.5% Ti while at a lower temperature the Mo–0.5% Ti contained more voids than did the

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[20]J. O. Stiegler and E. E. Bloom, Radiat. Eff. 8, 33–41 (1971).

[21]E. E. Bloom, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

[22]N. H. Packan, J. Nucl. Mater. 40, 1–16 (1971).

[23]J. D. Elen, pp. 351–352 in Septième Congrès International de Microscopie Electronique, Grenoble (1970), Vol. II. Société Français de Microscopie Electronique, Paris, 1970.

Table 2. Void and Swelling Data for Neutron-Irradiated Molybdenum

Material	Irradiation		Void Parameters			Reference
	Temperature (°C)	Fluence (neutrons/cm^2)	Concentration (voids/cm^3)	Average Diameter (Å)	Volume Fraction (%)	
Molybdenum[a]	425	2.5×10^{22}	0			b
	585	2.5×10^{22}	7.2×10^{16}	64	1.1	b
	790	2.5×10^{22}	5.4×10^{16}	72	1.2	b
Molybdenum[a]	440	3.5×10^{20}	5×10^{16}	25	0.04	c
	600	5.2×10^{20}	2×10^{16}	40	0.07	c
	760	4.4×10^{20}	5×10^{15}	50	0.03	c
Molybdenum[g]	700	1.4×10^{20}	3×10^{16}	40	0.10	d,e
	1000	1.3×10^{20}	5×10^{14}	200	0.19	d,e
	800	5.7×10^{19}	1×10^{16}	65	0.12	f
Molybdenum[a]	1050	1×10^{21}	1.2×10^{14}	290	0.17	h
	1150	1×10^{21}	5×10^{13}	470	0.28	h
Molybdenum[a]	600	5×10^{20}	8×10^{16}	20–30	0.1	i
	800	5×10^{20}	1×10^{16}	80	0.2	i
	900	5×10^{20}	3×10^{15}	80–200	0.2–0.4	i
	1200	5×10^{20}	1×10^{14}	300–600	0.2	i
Mo–0.5% Ti[a]	425	2.5×10^{22}	0			b
	585	2.5×10^{22}	2.4×10^{17}	69	4.5	b
	790	2.5×10^{22}	6×10^{16}	72	1.3	b

[a]Fluence for neutrons > 0.1 MeV.

[b]F. W. Wiffen, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

[c]J. D. Elen, G. Hamburg, and A. Mastenbroek, J. Nucl. Mater. 39, 194–202 (1971).

[d]R. C. Rau, R. L. Ladd, and J. Moteff, J. Nucl. Mater. 33, 325–327 (1969).

[e]R. C. Rau, F. Secco d'Aragona, and R. L. Ladd, Phil. Mag. 21, 441–452 (1970).

[f]J. L. Brimhall and B. Mastel, J. Nucl. Mater. 33, 186–194 (1969).

[g]Fluence for neutrons > 1.0 MeV.

[h]G. L. Kulcinski, B. Mastel, and J. L. Brimhall, Trans. Am. Nucl. Soc. 13, 162–163 (1970).

[i]Y. Adda, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

molybdenum.[16] In another comparison at lower neutron fluences, TZM was found to be void-free, while molybdenum irradiated under the same conditions contained voids.[24] Impurity content has also been shown to effect void formation in bcc metals. Results on vanadium[16,23] and molybdenum[24] suggest that the void concentration increases as the interstitial impurity content of the sample is increased. These results on impurity and alloying effects are too limited to allow general conclusions to be drawn. The results suggest, however, that interstitial impurities may be influential in the void nucleation step, probably as stabilizing agents in embryo voids. The main effect of alloying is probably related to the effect on the precipitation and distribution of these impurities.

High-Temperature Damage

High irradiation temperatures, $T > 0.5\ T_m$, have received very little attention for bcc metals. At these temperatures both defect species, vacancies and interstitials, have sufficient mobility to migrate rapidly through the lattice. Any defect clusters that do form, as for example in displacement cascades, redissolve as the thermal energy is sufficient to overcome the cluster binding energy. The temperature transition from the intermediate range, where voids form, to the high range, where there is no damage accumulation, is only approximately the $0.5\ T_m$ given above. The exact point depends on the system being studied and probably also shifts upward in temperature as neutron flux or fluence increases.

One important form of radiation damage does accumulate in this temperature region. Helium produced by (n,α) reactions is mobile and precipitates to form bubbles. The bubbles nucleate preferentially on dislocations, precipitates, and grain boundaries. Bubbles can be distinguished from voids by this tendency to nucleate on line or plane defects in the metal. The intrinsic difference between a void and a bubble is that voids are either gas-free or contain gases at low pressures.

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[24] J. L. Brimhall, H. E. Kissinger, and G. L. Kulcinski, to be published in proceedings of International Conference on Radiation-Induced Voids in Metals, held in Albany, New York, June 9–11, 1971.

Bubbles, on the other hand, contain gases at pressures in equilibrium with surface tension, satisfying

$$P = \frac{2\gamma}{r} ,$$

where P = gas pressure in bubble, γ = surface tension, and r = bubble radius. As a result of the internal pressure, voids and bubbles can further be distinguished by their postirradiation annealing behavior, with bubbles growing and voids shrinking on high-temperature annealing. The behavior and properties of helium bubbles have been studied in several fcc metal systems – for example, in stainless steels[25] and copper alloys[26] – but no data are available for bcc refractory metals.

Precipitation processes other than helium may also be of importance in both the intermediate- and high-temperature regions and may be enhanced by the irradiation. Their importance will vary from one metal to another, and in general have not been investigated in detail. The helium alone can be treated as a universal process as helium is insoluble in all metals.

THE EFFECT OF RADIATION DAMAGE ON PROPERTIES

The description of irradiation damage in terms of the changes produced in the microstructure will be related in this section to the changes produced in the physical and mechanical properties of irradiated bcc metals. Data on property changes produced by irradiation at conditions of interest to space reactor application are usually scarce.

Swelling of bcc Metals

Radiation-produced swelling can occur at all irradiation temperatures but is important only in the intermediate temperature region. Measurements of length change produced by 40°C irradiation of molybdenum

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[25] D. R. Harries, J. Brit. Nucl. Energy Soc. 5, 74–87 (1966).

[26] G.J.C. Carpenter and R. B. Nicholson, pp. 383–400 in Radiation Damage in Reactor Materials, Vol. II, International Atomic Energy Agency, Vienna, 1969.

showed the length increase[27] to saturate at a value near 0.05% at an approximate fluence of 5×10^{19} neutrons/cm^2. Data at slightly higher temperatures show that this low-temperature type of volume increase occurs in molybdenum for irradiation temperatures below 500°C (0.27 T_m) (ref. 24). The amount of swelling at constant fluence decreases as the temperature increases from 40 to 500°C. This swelling, which is a measure of the different amount of lattice relaxation around interstitial loops and vacancy defects (small loops or single vacancies), is too small to be of importance to the operation of reactor components.

Swelling in the intermediate-temperature range results from the precipitation of vacancies to form voids, accompanied by the precipitation of interstitials in dislocation loops. The general features of swelling due to void formation were discussed in the earlier section on intermediate-temperature damage. This swelling can interfere with reactor coolant channels and clearance between components. Differential swelling due to fluence or temperature gradients can result in bowing of components. The greatest amount of swelling observed to date in a reactor-irradiated metal is 11% in stainless steel irradiated to 1.45×10^{23} neutrons/cm^2 (> 0.1 MeV) at 410°C. The more restricted data on unalloyed bcc metals at lower fluences show swelling slightly greater than in stainless steel but less than in pure aluminum. The maximum amount of swelling reported in a bcc metal is 4.8% volume increase in niobium at 585°C (see Table 1).

The temperature dependence of swelling can be partially defined for one bcc metal. The niobium data from Table 1 have been extrapolated to a common fluence of 5.0×10^{21} neutrons/cm^2 (> 0.1 MeV), based on the assumption that the volume increase is linearly proportional to fluence. These data are plotted against irradiation temperature in Fig. 6. The data are not consistent at the lower temperatures but do suggest a maximum swelling temperature near 600°C (0.325 T_m) and an upper cutoff temperature near 900°C (0.436 T_m). The lower temperature limit for void formation in niobium is not defined by this set of data.

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[27]H. E. Kissinger, J. L. Brimhall, B. Mastel, and T. K. Bierlein, pp. 681–683 in International Conference on Vacancies and Interstitials in Metals, Jül-Conf-2 (Vol. II), KFA, Jülich, 1968.

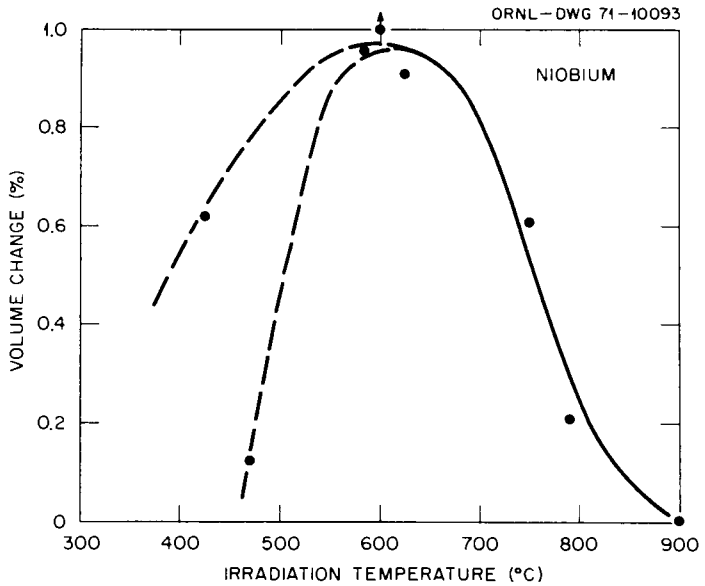

Fig. 6. Swelling of Niobium as a Function of Irradiation Temperature. Normalized to neutron fluence of 5.0×10^{21} neutrons/cm^2 (> 0.1 MeV) assuming linear dependence of swelling on fluence. Original data in Table 1.

If swelling is assumed to be linearly dependent on fluence, the assumption made in plotting Fig. 6, the maximum swelling of niobium at 5×10^{21} neutrons/cm^2 is near 1.0% and occurs near 600°C. If a lower than first-order dependence of swelling on fluence is assumed, as the approximately 0.4 dependence found by Bates and Pitner for tantalum,[28] the maximum niobium swelling determined by back-extrapolation from the higher fluence data could be 2 to 3%. Similar swelling values have been found for molybdenum, with the peak swelling temperature probably in the 800 to 900°C range. Available data in tungsten have demonstrated swelling at 900, 1000, and 1300°C but are too limited to show either the temperature range of maximum swelling or the upper temperature limit for swelling. Based only on comparison with the other bcc metals, especially with molybdenum as the most closely similar material, the temperature of maximum swelling in tungsten is expected to be near 1200°C and the upper cutoff temperature to be near 1600°C.

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[28] J. F. Bates and A. L. Pitner, Trans. Am. Nucl. Soc. 14, 603–604 (1971).

Swelling during high-temperature irradiation can be produced by the precipitation of transmutation-produced helium into equilibrium pressure bubbles. The amount of helium produced in refractory bcc metals in the lifetime fluence of 5×10^{21} neutrons/cm^2 (> 0.1 MeV) will be too small for the accompanying swelling due to helium bubble formation to be of any importance.

Electrical Resistivity of Irradiated Metals

Many experiments have been conducted in which electrical resistivity measured at cryogenic temperatures was used as the measure of the damage state produced by irradiation. Work on fcc metals has shown that the resistivity increase saturates during neutron, ion, or electron bombardment at temperatures below 0.3 T_m with a saturation resistivity increase that is 20 to 30% of the room-temperature resistivity of the metal under test.[29] A similar effect was demonstrated for tantalum electron irradiated at 4°K (ref. 30), where the saturation resistivity was again between 20 and 30% of the 20°C resistivity. In molybdenum neutron irradiated at 70°C the maximum resistivity increase was 25% of the room-temperature value,[31] but in this experiment further irradiation resulted in a reduction in resistivity. The maximum in resistivity is in qualitative agreement with the TEM observation[32] of a maximum in the concentration of observable defect clusters in the microstructure.

Refractory metals irradiated at temperatures between approximately 0.3 and 0.5 T_m form high concentrations of small voids, with void concentrations higher than 10^{17} voids/cm^3 observed. Under these conditions, the intermediate temperatures and the high void concentrations, the intervoid spacing approaches the mean free path of conduction electrons.

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[29] G. Burger, H. Meissner, and W. Schilling, Phys. Status Solidi 4, 281–288 (1964).

[30] D. Meissner and W. Schilling, Z. Naturforsch. 26a, 502–511 (1971).

[31] L. K. Keys, J. P. Smith, and J. Moteff, Phys. Rev. Letters 22, 57–60 (1969).

[32] R. C. Rau, J. Moteff, and R. L. Ladd, J. Nucl. Mater. 40, 233–235 (1971).

There are no measurements of the effect of voids on resistivity. Current models of electrical resistivity cannot predict the changes, but it is expected that enhanced electron scattering may produce large resistivity increases.

Mechanical Properties of Irradiated bcc Metals

As with other properties, the mechanical property data necessary to evaluate the service behavior of bcc metals in a thermionic reactor application are generally unavailable. Most mechanical properties data on bcc metals are on specimens irradiated at temperatures and fluences too low for present requirements. The effect of irradiation at temperatures below those at which voids form, $T < 0.3\ T_m$, has been more extensively studied in molybdenum than in other refractory metals. The general result of tensile tests at or near the irradiation temperature is that the strength properties are increased and the ductility is severely reduced. Data[33] on molybdenum and Mo–0.5% Ti irradiated at 425°C and tested up to 650°C are shown in Figs. 7 and 8. Figure 7 shows load elongation curves for control and irradiated Mo–0.5% Ti. Irradiation has increased the ultimate tensile strength by a factor of 2.5 to 3 but has also reduced the uniform elongation to zero and the total elongation to less than 10%. Examination of fracture modes and tensile curves on these and other specimens establishes that fractures are brittle at test temperatures up to as high as 550°C and allows the behavior to be summarized as shown in Fig. 8. The effect of irradiation has been to shift the tensile ductile-to-brittle transition temperature (DBTT) from below room temperature for the control material to a strain-rate sensitive value in the range 400 to 600°C. These figures also show that the irradiated molybdenum does not work harden and fails at temperatures above the DBTT by local deformation with a much reduced tensile fracture elongation. Data on niobium and Nb–1% Zr (ref. 34) irradiated at 70°C to a

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[33] F. W. Wiffen, unpublished data.

[34] W. J. Stapp and A. R. Begany, Seventh Annual Report – AEC Fuels and Materials Program, GEMP-1004, pp. 137–145 (March 29, 1968).

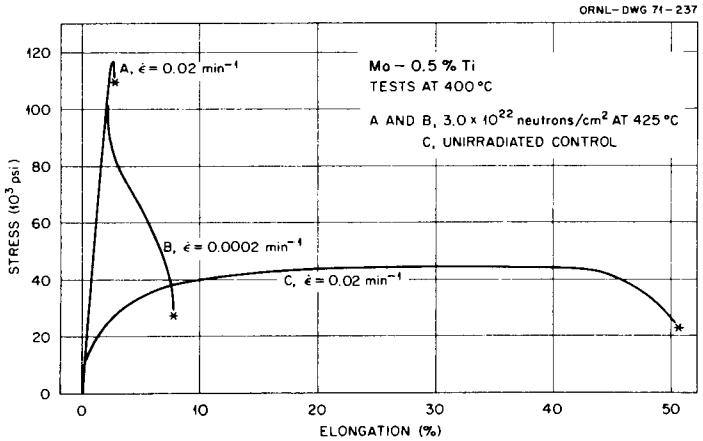

Fig. 7. Load-Elongation Curves for Mo–0.5% Ti Irradiated to 3.0×10^{22} neutrons/cm^2 (> 0.1 MeV) at 425°C.

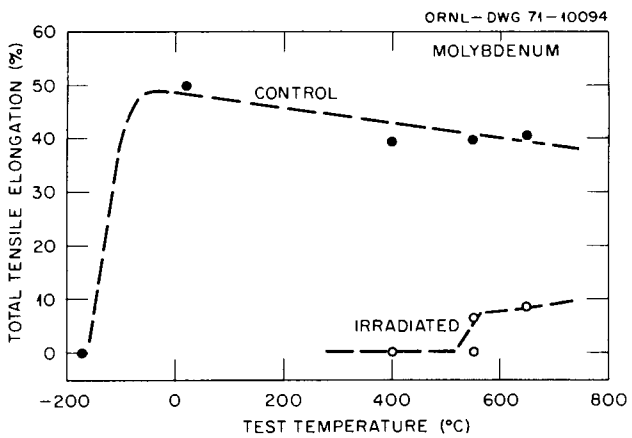

Fig. 8. Effect of Neutron Irradiation on the Tensile Ductility of Molybdenum. Irradiation at 425°C to 3.5×10^{22} neutrons/cm^2 (> 0.1 MeV).

fluence of 2.1×10^{20} neutrons/cm^2 (> 1.0 MeV) show a near-zero uniform elongation at room temperature with a total elongation of 18.6% in the niobium but only 0.4% in the alloy. The behavior of niobium and Nb–1% Zr at higher fluences is not known.

Even less data are available on refractory metals irradiated and tested in the temperature range in which voids form. The only results on specimens known to have contained voids are on molybdenum.[35] Specimens irradiated at 700 and 1000°C to a fluence near 1.4×10^{20} neutrons/cm^2 (> 1.0 MeV) showed creep rupture lives at 750°C that were three to four times that of unirradiated specimens. The

[35] J. Moteff, R. C. Rau, and F. D. Kingsbury, pp. 269–281 in Radiation Damage in Reactor Materials, Vol. II, International Atomic Energy Agency, Vienna, 1969.

ductilities were comparable to that of control specimens. Extrapolation of these results to higher fluences is not possible.

The in-reactor behavior of metal components may also be affected by irradiation-enhanced creep in this intermediate-temperature range. There are no data on the magnitude of this effect in bcc metals.

At higher irradiation temperatures, $T > 0.5\ T_m$, displacement damage does not accumulate in the metal lattice; however, helium produced by transmutation reactions has an effect on reducing the ductility of many metals. In fcc metals the helium generally becomes detrimental to ductility at temperatures above about $0.5\ T_m$ (refs. 36, 37). In the only bcc metal (other than commercial steels) in which the effect of helium was studied, a concentration of 1×10^{-6} atom fraction helium was found to reduce the tensile ductility of vanadium alloys at test temperatures above 800°C (about $0.5\ T_m$) but to have no effect below this temperature.[38] The most severe ductility reduction produced by the helium was in V–20% Ti–10% Nb, where the ductility dropped from 20% to less than 1% in the helium containing specimens. In these vanadium alloys, as in the fcc metals, helium reduces ductility by weakening grain boundaries, resulting in an intergranular fracture mode. The effect of helium on the other bcc metals is unknown, but embrittlement similar to that found in vanadium and in the fcc metals seems to be typical and should be anticipated.

SUMMARY – THE EFFECT OF IRRADIATION ON THERMIONIC REACTOR COMPONENTS

Emitters

Displacement damage effects are not expected in thermionic diode emitters except in low-temperature sections. The average emitter

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[36] D. R. Harries, J. Brit. Nucl. Energy Soc. 5, 74–87 (1966).

[37] G.J.C. Carpenter and R. B. Nicholson, pp. 383–400 in Radiation Damage in Reactor Materials, Vol. II, International Atomic Energy Agency, Vienna, 1969.

[38] K. Ehrlich and H. Böhm, pp. 349–355 in Radiation Damage in Reactor Materials, Vol. II, International Atomic Energy Agency, Vienna, 1969.

operating temperature of 1660°C is 0.525 T_m for tungsten and would be a higher fraction for any substitute material. Vacancies and interstitials do not usually form stable clusters at these temperatures. Ductilities will be reduced by the formation and growth of helium bubbles on grain boundaries and the high vacancy flux produced by the irradiation may aid other insoluble gases trapped in the material to precipitate on grain boundaries and thus weaken the material.

Sections of the emitter operating at lower temperatures will be subject to void formation, with resulting swelling, resistivity increase, and possible degradation of mechanical properties.

Collectors and Electrical Conductors

Niobium or molybdenum collectors at operating temperatures near 700°C will form a high density of small voids during the reactor 5-year lifetime fluence of 5×10^{21} neutrons/cm^2 (> 0.1 MeV). For either material the void concentration is expected to be near 1×10^{17} voids/cm^3 and void volume fraction in the range 1 to 3%. If the collector swells while the emitter does not, the net result will be an increase in the emitter-collector spacing. If the collector diameter is assumed to be 1.0 in., the swelling of 1 to 3% will produce a diameter increase of 1/3 to 1% and thus an increase in diode gap of up to 0.005 in.

Void formation in collectors and conductors could increase the electrical resistivity significantly, resulting in increased power losses due to greater joule heating.

The mechanical properties of the collector will be affected by the irradiation-produced void formation. While the magnitude of changes cannot yet be predicted, it is expected that the ductility will be reduced and the strength properties increased.

Fuel Element Sheath

The Nb–1% Zr containment sheath operating over the temperature range 600 to 700°C will swell due to the formation of voids. The maximum volume increase is expected to be in the range 1 to 3%. Differential

swelling will occur on any fuel sheaths that are exposed to neutron flux gradients or temperature gradients, and some slight component bowing may have to be accommodated in the reactor design. The ductility of this material will also be reduced by the irradiation, but data are not available to determine the magnitude of the reduction.

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