THE TENSILE PROPERTIES OF FAST REACTOR NEUTRON
IRRADIATED BCC METALS AND ALLOYS*

F. W. Wiffen
Metals and Ceramics Division, Oak Ridge National Laboratory
Oak Ridge, Tennessee 37830

Abstract

Rod tensile samples of niobium, molybdenum, tantalum, one alloy based on each of these metals, and two vanadium-base alloys have been irradiated in EBR-II at temperatures between 390 and 1140°C to neutron fluences in the range 1.5 to 6.1×10^{22} neutrons/cm² (>0.1 MeV). The effect of neutron irradiation at these fluences and temperatures on the strength and ductility properties was determined in tensile tests at temperatures between 20 and 800°C at strain rates of 0.02 min⁻¹ or slower. The effect of irradiation is most severe in molybdenum base systems. Irradiation near 400°C renders Mo-50% Re brittle to at least 800°C. Similar irradiation of molybdenum and Mo-0.5% Ti produces a high concentration of small dislocation loops in the microstructure and raises the strain-rate dependent tensile DBTT from below room temperature to the range 400 to 550°C. Brittle failures occur with a mixed cleavage and grain boundary separation fracture mode. Specimens of tantalum, T-111, and Nb-1% Zr exhibited zero uniform elongation but had total elongations in the range 4 to 10% with ductile mode fractures for most test conditions. Niobium, V-10% Cr, and VANSTAR-7 had 5 to 17% total elongation and uniform elongations in the range 1.5 to 10%. Marked strength increases over control values were found in all samples. The results are discussed in terms of the microstructures and fracture modes. It is concluded that dislocation channeling is the process responsible for the complete loss of uniform elongation where loop hardening predominates.

Introduction**MASTER**

Body-centered cubic (bcc) refractory metals have been largely ignored in the elevated-temperature, high-fluence radiation damage studies prompted by the needs of the fission breeder reactor programs. However, the progress in plasma physics experiments has been sufficiently encouraging that serious studies of the engineering problems of proposed Controlled Thermonuclear Reactors (CTR's) have begun. These CTR-related studies have renewed the interest in the properties of bcc metals irradiated to high fluences at temperatures typical of future fusion reactor operation, a relatively new environment for these materials.

Some microstructural effects of neutron irradiation in bcc metals have been reported by Wiffen (1) and by Slen et al. (2), with particular emphasis placed on void formation.

*Research sponsored by the U.S. Atomic Energy Commission under contract with the Union Carbide Corporation.

Less complete sets of data have been reported by other authors, and all of the available microstructural data for irradiated bcc metals, including the void data, has recently been reviewed by Eyre (3). Even fewer data are available for mechanical properties of bcc metals irradiated to high fluences at elevated temperatures. Where data are available, irradiation conditions usually reflect the environment of research reactors with temperatures near reactor ambient (about 50°C) and fluences below 10^{21} neutrons/cm². The limited mechanical properties data on irradiated bcc metals have been compiled by Kangilaski (4).

This paper presents tensile data and supporting microstructural and fractographic analyses for refractory bcc metal specimens irradiated in the Experimental Breeder Reactor (EBR-II). Specimens of most materials were made from commercially available alloy rods. They were irradiated in static inert-gas atmospheres at temperatures from 390 to 1140°C to fluences in the range 1.5 to 6.1×10^{22} neutrons/cm² (>0.1 MeV). The specimens discussed here, with the exception of V-10% Cr and VANSTAR-7 alloys, were included by J. Moteff and co-workers, then at GE-NMPO, in a joint ORNL-GE-NMPO irradiation program.

Some data for the molybdenum alloys have been presented previously (5); the irradiation temperatures given then corresponded to original design objectives. The irradiation temperatures used in this report are more accurate. The microstructures discussed in the earlier reports were for tab samples irradiated under similar conditions to those of the tensile samples. Microstructural results reported here were obtained from the tensile specimens proper. These observations replace the previous speculations.

Experimental

The specimens of Mo, Nb, Ta and their alloys used in this work were made from rod stock supplied by commercial vendors. They represent state-of-the-art refractory metal production, not research grade material production. Since no commercial supplier was available for vanadium alloys, a 1-lb ingot of V-10% Cr and a 40-lb ingot of VANSTAR-7 were produced in alloy development programs. The suppliers and primary consolidation methods for the specimen materials are listed in Table 1. All rod stock was reduced from ingots by hot breakdown followed by cold forming to final diameter near 0.25 in. Tensile specimens were machined with a gage section 0.125 in. in diameter and 1.125 in. long. They were given a final vacuum anneal at the temperatures given in Table 1 to produce fully recrystallized microstructures. Chemical analyses are given in Table 2.

All irradiations reported here were performed in EBR-II. Since irradiation experiments in the core of this reactor are not instrumented, irradiation at temperatures above the reactor coolant temperature is accomplished in sealed capsules in a static-gas atmosphere. Four tensile specimens were loaded in each holder to form a nearly solid specimen and holder assembly (Fig. 1). The heat generation rate in this assembly was calculated

TABLE 1
Material Sources and Preirradiation Specimen Anneal

Alloy	Supplier	Ingot Consolidation	Specimen Anneal ^a Temperature, °C
Molybdenum	Climax Molybdenum Company of Michigan	Climelt Arc Cast	1350
Mo-0.5% Ti	Climax Molybdenum Company of Michigan	Climelt Arc Cast	1450
Mo-50% Re	Chase Brass and Copper Company, Rhenium Division	Powder Metallurgy	1400
Niobium	Union Carbide Corporation	Electron-Beam Melt	1150
Nb-1% Zr	Union Carbide Corporation	Electron-Beam Melt	1275
Tantalum	National Research Corporation	Double-Arc Melt	1250
T-111	National Research Corporation	Double-Arc Melt	1400
V-10% Cr	Oak Ridge National Laboratory	Arc Melt	1100
VANSTAR-7	Westinghouse-Astronuclear Laboratory	Arc Melt	1200

^aAll anneals for 1 hr in vacuum.

TABLE 2
Chemical Composition of Specimen Materials

Composition	Specimen Material								
	Mo	Mo-0.5% Ti	Mo-50% Re	Nb	Nb-1% Zr	Ta	T-111	V-10% Cr	VANSTAR 7
Alloy Base	Mo	Mo	Mo	Nb	Nb	Ta	Ta	V	V
Alloy Addition, %		0.52 Ti	50 Re		0.94 Zr		7.9 W 2.3 Hf	9.9 Cr	9.1 Cr 3.3 Fe 1.2 Zr
Impurities, ppm, weight									
C	30	400	<20	30	30	20	24	140	500
H	0.2	0.4	0.1	2	2	1	1		
N	1	2	0.1	10	17	10	20	550	90
O	7	10	2	198	80	20	19	77	760
Cu	3	<1	<1	<20	<20	25	40		
Fe	15	28	32	<20	20	35	23		M ^a
Mo	M	M	M	43	750	60	60		
Nb			<1	M	M	250	280		<1000
Si	15	77	30	35	120	<20	<20		
Ta	<1000			1000	1400	M	M		<2000
W	<100	<100	<40	<100	1220	380	M		

^aM = major alloying agent.

FIG. 1

Schematic of Tensile Specimens and Specimen Holder Used in EBR-II Materials Irradiation Experiments.

for the known nuclear heating rate in the intended reactor position. This heat production and the heat transfer rates were used to calculate the gas gap between the specimen holder and the containment tube that gave the desired irradiation temperature. The greatest uncertainty in the calculated temperatures arise from uncertainties in the nuclear heating rates. Since most of the samples were irradiated between 390 and 500°C, the thermal gradients between reactor coolant and specimens were small, and calculated irradiation temperatures are believed to be accurate to $\pm 10^\circ\text{C}$ or less. The uncertainty increases as the irradiation temperature increases. For temperatures in the range 630 to 700°C, the estimated uncertainty is $\pm 30^\circ\text{C}$. Two capsules were initially at $850 \pm 30^\circ\text{C}$ but the stainless steel containment tube swelled during irradiation, increasing the gas gap and raising the end-of-life irradiation temperature about 200 to 300°C above the initial value. Checks on the irradiation temperatures were provided by the postirradiation annealing responses of passive SiC temperature monitors included in similar capsules; they confirm these temperature limits for the lower irradiation temperatures.

Neutron fluences were calculated from the reactor power-time history and the detailed reactor flux vs position characterization (6). Fluences are quoted for neutron energies $E > 0.1$ MeV. For comparison with damage from other reactor irradiations and from ion bombardment, the damage in terms of displacements per atom (dpa) have been calculated for the Lindhard model using a threshold displacement energy of 25 eV and the position-dependent neutron spectra in EBR-II (7). The resulting dpa numbers for each set of samples are given in Table 3. There is no proportionality constant relating dpa values

TABLE 3
Displacements and Helium Produced During Reactor Irradiation

Alloy ^a	Fluence, >0.1 MeV (n/cm ²)	Displacements ^b Per Atom	Helium Content ^c (ppm, atomic)
	$\times 10^{22}$		
Molybdenum	6.1	74	0.95
Mo (50% Re)	5.3	59	0.83
Molybdenum	4.0	38	0.62
Molybdenum	3.45	38	0.54
Mo (0.5% Ti)	3.0	33	0.47
Mo (50% Re)	1.5	15	0.23
Nb, Nb (1% Zr)	3.7	34	2.0
Niobium	3.0	32	1.6
Nb (1% Zr)	1.9	15	1.0
Ta, Ta (T-111)	1.9	7.3	0.2
Ta (T-111)	1.9	9.2	0.2
Tantalum	1.5	6.4	0.15
V (10% Cr, VANSTAR)	1.5	17	3

^aAlloys are treated as if 100% base metal for these calculations.

^bDpa calculated from Linhard model with effective threshold displacement energy of 25 eV. Calculations reflect the neutron spectrum at the sample location. Ref: J. D. Jenkins, H. T. Kerr, and R. Q. Wright, Oak Ridge National Laboratory, personal communication, May 28, 1971 and May 17, 1973.

^cHelium content in molybdenum and niobium calculated by fluence ratio from measured contents at 2.5×10^{22} n/cm², ref: H. Farrar IV, Atomics International, personal communication, Aug. 31, 1972. Spectral differences have not been considered. Values for tantalum and vanadium taken from H. Alter and C. E. Weber, J. Nucl. Mater. 16, 68 (1965).

to the fluence values because of the dependence of the neutron spectrum on the reactor position. Approximate transmutation helium contents from (n,α) reactions are also given in Table 3. These values do not include effects of the position-dependence of the reactor spectrum. Helium contents were calculated for molybdenum, niobium, and their alloys from measured helium contents in molybdenum and niobium irradiated to 2.5×10^{22} neutrons/cm² (>0.1 MeV) in one of these experiments (8). Values for tantalum and vanadium alloys were calculated from the helium production rates given by Alter and Weber (9).

Tensile tests were conducted on an Instron testing machine in a flowing helium environment. Specimens of molybdenum alloys were held 30 min, while specimens of other materials were held for 15 min at temperature before the start of the test. Engineering stresses and strains based on initial specimen dimensions are reported. Elongations are derived from the Instron crosshead travel. Crosshead speeds from 0.0002 to 2.0 in./min

were used, with most tests at 0.02 in./min. As the specimens have an effective gage length of 1.0 in. nominal strain rates equal the crosshead speeds.

Fracture surfaces were examined by scanning electron microscopy to help establish fracture modes.

The damage state of the specimens was characterized by swelling determined from length measurement, from immersion density measurements, and by transmission electron microscopy. Length measurements were of limited value because of uncertainty in the pre-irradiation length measurements on the specimens. Immersion densities were measured in water with a wetting agent. Transmission electron microscopy provides the best evaluation of the defect microstructure, but only qualitative data are available and only for some of the materials. Some specimens have been examined as-irradiated and after postirradiation annealing. Microstructures were so complex and the damage cluster density so great in some cases that the high-temperature anneals were of value in simplifying the microstructure. This technique was especially useful in verifying the presence of voids.

Results

Tensile Properties

Tensile properties, test conditions, and irradiation parameters are given in Table 4. All irradiated materials under all test conditions showed large increases in the strength properties and decreases in the ductility properties. In general, the tensile tests yielded three types of stress-strain curves. For purposed of discussion, the three types of stress-strain curves will be defined as follows:

1. Conventional. — The load vs elongation curve deviates from the modulus line at a proportional limit or yield point, then the load increases with increasing elongation until a maximum load is reached, and finally decreases as elongation continues to failure.
2. Brittle. — Failure occurs at the maximum load after less than 0.2% deviation from the modulus line.
3. Unstable. — The maximum load occurs after less than 0.2% deviation from the modulus line. Plastic instability is coincident with yielding but the specimen necks and shows appreciable elongation and reduction in area before failure at a load much below the maximum.

The tensile data for molybdenum and Mo-0.5% Ti were similar (see Table 4). The ultimate tensile strength (UTS) and elongation values for molybdenum are given in Fig. 2. Control material was brittle at -80 and -196°C. The ductile-brittle transition temperature (DBTT) for the fully annealed control material was between -80 and 20°C, with conventional stress-strain behavior at room temperature and above. Irradiation increased the ultimate strengths of molybdenum and Mo-0.5% Ti by a factor of 2 to 3. Molybdenum irradiated near 450°C and tested at a strain rate of 0.02 min⁻¹ showed a DBTT between 550 and 650°C: at 550°C and below the stress-strain behavior was brittle, but at 650°C the

TABLE 4

Tensile Properties of Irradiated and Control
Refractory Metals

Test		Irradiation		Strength, psi		Ductility, %		
Temperature (°C)	Strain Rate (min ⁻¹)	Temperature (°C)	Fluence >0.1 MeV, (n/cm ²)	0.2% Yield	Ultimate Tensile	Uniform Elongation	Total Elongation	Reduction in Area
				$\times 10^3$	$\times 10^3$			
Molybdenum								
-196	0.02			105.3	105.3	0	0	0
-90	0.02			118.6	118.6	0.1	0.1	0
22	0.02			52.0	72.0	22.0	54.8	64
200	0.02			20.8	31.1	39.0	56.1	75
200	0.0002			17.2	43.4	38.0	47.0	80
400	0.02			14.7	37.9	30.9	42.4	87
550	0.02			10.5	33.5	31.7	41.8	90
550	0.0002			9.44	32.1	28.1	37.1	67
650	0.02			10.7	32.4	29.7	42.3	94
22	a	455	4.0	a	a	a	0.1	0
22	0.02	857 to 1136 ^b	6.1	124.3	124.3	0.1	0.2	0
200	0.0002	455	4.0	128.8	128.8	0	0	0
400	0.02	454	3.5	113.3	113.3	0.1	0.1	0
400	0.02	455 + Anneal ^c	4.0	84.8	107.0	6.3	6.3	7
400	0.02	857 to 1136 ^b	6.1	81.7	101.2	5.7	10.5	34
550	0.02	454	3.5	101.0	101.0	0.2	0.2	0
550	0.0002	454	3.5	85.0	85.0	0.1	0.5	42
650	0.02	454	3.5	86.3	86.3	0.1	8.4	71
Mo-0.52 Ti								
-196	0.02			103.7	101.7	0	0	0
-90	0.02			100.2	111.5	3.8	3.8	3
22	0.02			50.9	79.3	21.5	48.1	34
400	0.02			14.4	44.5	34.0	50.1	88
400	0.002			15.0	41.7	32.9	47.5	87
400	0.0002			17.1	40.7	10.9	47.3	91
650	0.02			14.0	40.3	29.3	39.6	85
400	0.02	445	3.0	118.0	118.0	0.1	0.1	0
400	0.002	445	3.0	107.6	107.6	0	0	0
650	0.0002	445	3.0	101.2	101.2	0.1	7.0	68
650	0.02	445	3.0	94.4	94.4	0.1	7.9	70
Mo-50% Re								
-196	0.02			105.4	184.4	5.9	5.9	6
22	0.02			87.6	134.3	19.5	27.6	41
400	0.02			63.3	87.4	20.8	31.7	85
650	0.02			45.0	76.1	12.5	20.3	34
800	0.02			41.9	68.0	12.1	21.1	89
400	0.02	393	1.5	144.3	144.3	0.1	0.1	0
400	0.02	855 to 1066 ^b	5.1	170.7	191.7	0.8	0.8	1
650	0.02	393	1.5	147.3	147.3	0.2	0.2	0
650	0.02	855 to 1066 ^b	5.1	179.3	179.3	0.1	0.1	0
800	0.02	393	1.5	141.4	141.4	0	0	0
Niobium								
22	0.02			15.9	30.5	26.8	60.3	95
400	0.02			10.4	25.3	24.9	39.2	92
550	0.02			8.1	20.1	18.1	32.6	99
650	0.02			8.7	17.5	22.5	40.8	97
25	0.02	460	3.0	62.3	75.6	10.1	17.0	73
400	0.02	460	3.0	49.8	59.8	6.6	15.3	85
400	0.02	460	3.0	57.1	64.9	4.8	13.0	84
650	0.02	460	3.0	47.3	50.4	2.6	12.4	47
Nb-1% Zr								
22	0.02			16.2	32.9	30.4	52.0	94
400	0.02			17.7	25.1	18.2	27.5	95
650	0.02			12.3	27.0	14.7	23.8	80
25	0.0002	394	1.9	82.0	92.0	0.2	10.4	65
25	2.0	394	1.9	81.7	89.1	3.4	12.4	82
25	0.02	448	3.7	92.5	92.5	0.1	6.3	39
400	0.02	394	1.9	72.4	72.4	0.1	8.5	74
400	0.02	448	3.7	74.3	74.3	0.1	8.4	53
650	0.02	448	3.7	66.3	66.3	0.1	8.3	83
Tantalum								
22	0.02			25.0	37.0	33.7	49.0	95
400	0.02			9.5	31.5	22.1	33.6	89
650	0.02			11.4	38.0	9.3	20.1	74
25	0.02	391	1.5	106.3	106.3	0.2	10.1	64
400	0.02	391	1.5	46.9	56.9	0.1	9.4	87
400	0.02	637	1.9	62.8	71.2	6.6	14.9	72
650	0.02	391	1.5	71.2	71.2	0.2	7.9	80
650	0.02	637	1.9	61.5	68.9	4.4	11.3	60
Ta-1% Ti								
22	0.02			80.2	97.7	19.9	37.8	81
400	0.02			51.1	66.3	23.6	31.3	99
550	0.02			51.1	84.3	16.2	23.7	81
25	a	414	1.9	a	a	a	7.6	68
400	0.02	414	1.9	179.3	183.3	0.3	4.5	36
400	0.02	643	1.9	154.6	152.3	4.0	10.8	67
650	0.02	314	1.9	157.0	157.0	0.1	6.0	69
650	0.02	643	1.9	144.6	152.7	1.9	8.3	65
V-10% Cr								
22	0.02			35.7	44.9	19.2	32.5	83
400	0.02			27.4	53.5	15.7	26.5	86
650	0.02			5.0	32.0	18.3	39.4	91
400	0.02	494	1.5	58.3	58.3	4.2	13.1	39
650	0.02	690	1.5	41.1	45.6	3.8	20.3	78
VANSTAR-7								
22	0.02			51.5	73.8	17.1	28.5	72
400	0.02			32.7	61.6	13.4	23.8	73
550	0.02			34.3	61.0	12.1	20.0	71
25	0.02	496	1.5	117.9	147.5	2.5	8.2	40
400	0.02	496	1.5	118.7	130.2	1.5	5.0	27
400	0.02	690	1.5	97.1	102.3	5.4	10.1	70
650	0.02	690	1.5	91.0	97.6	4.6	14.0	40

a Equipment malfunction, data not available.

b Irradiation temperature increased due to swelling of housing tube at high fluences.

c Postirradiation annealed 1 hr at 1050°C in vacuum.

unstable type of curve resulted. A lower DBTT was found for a strain rate of 0.0002 min^{-1} with brittle failure at 200°C but the unstable type of ductile curve resulting at 550°C . Tests at 400°C on the irradiated Mo-0.5% Ti showed brittle failures at strain rates of 0.02 and 0.002 min^{-1} but the unstable ductile behavior at 0.0002 min^{-1} . The observed similarities for molybdenum and Mo-0.5% Ti justify combining these data and concluding that the DBTT of both irradiated materials is between 200 and 400°C for a strain rate of 0.0002 min^{-1} .

Two molybdenum samples irradiated at a temperature that increased from 857 to 1136°C during the reactor exposure were tested at 0.02 min^{-1} . For this condition the molybdenum was brittle at 20°C but ductile at 400°C , setting an upper limit on the DBTT of 400°C . It is important, moreover, that the stress-strain curve at 400°C was of the conventional type, indicating some ability to work harden while straining almost 6% before the onset of plastic instability.

A molybdenum specimen irradiated at 455°C was annealed for 1 hr at 1050°C before testing. This heat treatment was expected to cause recovery of any small dislocation loop structure, but have little effect on the void population (2). The annealed sample was tensile tested at 400°C at 0.02 min^{-1} . Its yield strength was well below that of the as-irradiated sample tested under the same conditions. After yield

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

FIG. 2

The Ultimate Tensile Strength and Total Tensile Elongation for Irradiated and Control Samples of Molybdenum. Tensile strain rate was 0.02 min⁻¹ except as shown. Solid lines connect results differing only in test temperature. Dashed lines connect results where irradiation conditions or strain rates are not held constant.

the sample work hardened and strained uniformly to 6.3%. Failure occurred abruptly at the ultimate load without any plastic instability (necking).

The ultimate tensile stress for brittle failure is equivalent to a cleavage strength; it was above 100,000 psi for all brittle fractures. The data in Fig. 2 suggest that the cleavage stresses of irradiated samples may be slightly higher than those for control samples, and that the cleavage stress in the irradiated samples may be temperature dependent. The data suggest a drop in cleavage stress from 125,000 psi at 20–200°C to 100,000 psi at 550°C. This observation is tentative until more data can be assembled and treated statistically. However, all cleavage stresses, for both irradiated and control samples,

may be expressed as $114,000 \pm 13,000$ psi, which is narrower than the range $97,500 \pm 15,000$ psi given by Rosenfield et al. (10) for the cleavage stress of unirradiated, unnotched molybdenum.

As the tabular data show, the irradiated Mo-0.5% Ti data closely parallel the irradiated molybdenum data, but the strengths of the alloy slightly exceed those for molybdenum tested under the same conditions, even though the fluence received by the alloy was slightly lower. The two samples of Mo-0.5% Ti showing nonbrittle behavior had the unstable type stress-strain curve and failed with elongations less than 10%. The local ductility was high, however, with reduction in area near 70% at fracture.

Samples of Mo-50% Re irradiated at 393°C were brittle at a strain rate of 0.02 min^{-1} when tested at 400, 650, and 800°C (see Table 4). The failure stress was essentially temperature independent at $144,300 \pm 3,000$ psi, a value 1.6 to 2 times the ultimate stress for control samples tested at the same temperatures. Samples of this alloy irradiated at increasing temperatures of 855 to 1066°C were brittle at 650°C but had uniform plastic strain of 0.8% at 400°C , then underwent brittle failure abruptly at the ultimate load. The two samples of Mo-50% Re irradiated at the higher temperature had much higher cleavage stress than did the samples irradiated at 393°C . This result suggests that the behavior of this alloy is too complex to be explained in terms of an irradiation produced shift in the DBTT. Unfortunately, too little work has been completed to explain this behavior.

Stress-strain curves for irradiated niobium and Nb-1% Zr are shown in Figs. 3 and 4 respectively. All tests of irradiated niobium showed the conventional type of stress-strain curve. Figure 3 shows that for niobium samples irradiated at a single condition both the strength and ductility properties decrease with increasing test temperature. The stress-strain curves for irradiated Nb-1% Zr, Fig. 4, are all of the unstable type for tests at a strain rate of 0.02 min^{-1} . As for the niobium, both the strength and total elongation of irradiated Nb-1% Zr decrease with increasing test temperature. The strength and elongation properties of irradiated niobium and Nb-1% Zr are compared in Fig. 5 with control properties. The irradiation-produced hardening is somewhat greater in the alloy than in the niobium. The most important difference between the two materials, however, is the lack of work hardening and uniform strain in Nb-1% Zr.

A few specimens of niobium and Nb-1% Zr irradiated under slightly different conditions were also tested and the data are included in Table 4. Irradiation of niobium at a slightly lower temperature to a 25% higher fluence produced properties at 400°C quite similar to those from the basic set, with no differences large enough to be important. Similarly, a Nb-1% Zr specimen irradiated at a temperature lower by 50°C and a fluence only half that of the specimens represented in Fig. 5 had very similar strength and ductility properties at 400°C .

FIG. 3

Tensile Curves for Irradiated Niobium Showing the Effect of Test Temperature. This type of stress-strain curve is referred to as "conventional" in this paper.

FIG. 4

Tensile Curves for Irradiated Nb-1% Zr Showing the Effect of Test Temperature. This type of stress-strain curve is defined as "unstable," with the maximum load reached within 0.2% deviation from the elastic modulus line.

FIG. 5
Tensile Properties of Niobium and Nb-1% Zr Tested in the Annealed and Irradiated Conditions. All tests at a strain rate of 0.02 min^{-1} .

The effect of strain rate at 25°C was investigated in two specimens of Nb-1% Zr irradiated at 394°C to 1.9×10^{22} neutrons/cm² ($>0.1 \text{ MeV}$). The strain rates differed by 4 orders of magnitude. Figure 6 shows the resulting stress-strain curves. At 2.0 min^{-1} work hardening occurred from the 81,700 psi yield stress to the 89,100 psi ultimate tensile stress while 3.4% uniform elongation occurred. At 0.0002 min^{-1} , on the other hand, an identical specimen had the unstable type of stress-strain curve. The yield stresses were quite close in these two tests but the ultimate stress and total elongation were lower for the slower test.

The tensile properties of irradiated tantalum and T-111 (Ta-8% W-2% Hf) are compared to the properties of the unirradiated materials in Fig. 7. Irradiation markedly increased the strength properties and decreased the ductility. The ratio of ultimate stress for the irradiated sample to that of the control sample varies from nearly 3 at room temperature to less than 2 at 650°C in both these materials. Samples of tantalum and T-111 were each irradiated at two different temperatures, and then tested. At the lower irradiation temperature, 391°C for tantalum and 414°C for T-111, all stress-strain curves were of the unstable type, with the maximum load occurring after less than 0.3% uniform elongation. The samples then necked to failure elongations that ranged from 4.5 to 10.1%. The two materials irradiated to comparable fluences near 640°C behaved quite differently, indicating an important effect of irradiation temperature. All of the samples irradiated at 640°C had conventional stress-strain curves. The uniform elongations of these samples ranged from 1.9 to 6.6% with total elongations in the range 8.5 to 14.9%.

FIG. 6
 Effect of Strain Rate on the Tensile Properties of Irradiated Nb-1% Zr. Test temperature is 25°C .

Only a few results are available on vanadium-base alloys. These are included in Table 4, but are not displayed graphically. Irradiation to 1.5×10^{22} neutrons/cm² ($>0.1 \text{ MeV}$) at 496 and 690°C resulted in an increase in strength properties and decrease in elongation of both V-10% Cr and VANSTAR-7, relative to the respective properties in control tests. Both of these alloys retain an ability to work harden beyond the yield stress in the irradiated state and thus exhibit some uniform elongation under all conditions examined. The uniform elongations ranged from 1.5 to 5.4% and total elongations from 5.0 to 20.3%. In both alloys, reduction in area values indicate high local deformation within the necked region. Two tests at 400°C indicate a marked dependence of the hardening on the irradiation temperature in VANSTAR-7, with the increase in ultimate strength approximately 60% greater for 496°C irradiation than for 690°C irradiation.

Fractography

Selected specimens of the molybdenum and niobium-base alloys were examined by scanning electron microscopy (SEM) to evaluate the effect of neutron irradiation on the fracture mode of these materials. Two types of fracture surfaces were found for the molybdenum and molybdenum alloy specimens, either brittle fractures characterized by mixed cleavage and intergranular separation faces or

FIG. 7

Tensile Properties of Tantalum and T-111 (Ta-8% W-2% Hf). Results for annealed samples and for two irradiation temperatures are shown for each material. Uniform elongations were less than 0.3% for the lowest irradiation temperature for both materials and these values are not shown.

ductile fractures with the entire fracture surface covered with the characteristic, equiaxed dimples of the normal rupture of a ductile material. None of the fracture surfaces of the molybdenum-base specimens were mixed mode failures.

Examples of the types of fracture surfaces found in molybdenum and Mo-0.5% Ti are shown in Fig. 8. All fully brittle failures in molybdenum, with total elongations less than 0.2%, were a mixture of regions of cleavage and intergranular separation with approximately equal areas of each type. The example shown in Fig. 8a is typical, and no morphological difference was observed between brittle fractures in control samples at low temperatures and in irradiated samples at 400°C. The molybdenum sample irradiated at 455°C, annealed for 1 hr at 1050°C and tensile tested at 400°C strained 6.3% before failing at

FIG. 8

Typical Areas on the Fracture Surfaces of Irradiated Molybdenum and Mo-0.5% Ti Tensile Specimens. All Tests at 400°C, strain rate 0.02 min^{-1} , except (d) at 0.0002 min^{-1} . Scanning electron microscope magnification $1000\times$. (a) Molybdenum, irradiated to $3.5 \times 10^{22} \text{ n/cm}^2$ at 454°C. Elongation 0.1%. (b) Molybdenum, irradiated to $4.0 \times 10^{22} \text{ n/cm}^2$ at 455°C, annealed 1 hr at 1050°C. Elongation 6.3%. (c) Mo-0.5% Ti irradiated to $3.0 \times 10^{22} \text{ n/cm}^2$ at 455°C. Elongation 0.1%. (d) Mo-0.5% Ti irradiated to $3.0 \times 10^{22} \text{ n/cm}^2$ at 445°C. Elongation 7.0%.

the ultimate stress. This fracture surface, Fig. 8b, showed no major differences from the fracture surfaces of fully brittle tests. Although the sample irradiated between 857 and 1136°C and tested at 400°C exhibited 5.7% uniform elongation and then necked to a total elongation of 10.5% and a reduction in area of 36%, the fracture surface was basically similar to those of fully brittle samples. The main observable difference was a greater degree of secondary cracking in this sample than in the fully brittle samples.

Brittle fractures in Mo-0.5% Ti, Fig. 8c, differed from those in molybdenum in that only 5 to 10% of the fracture surface was intergranular with the remainder cleavage surfaces. Brittle fractures in the Mo-50% Re samples irradiated at 393°C were almost completely made up of cleavage surfaces. Fractures in the Mo-50% Re irradiated at the higher temperature have not been examined.

Unstable stress-strain curves occurred for all other irradiated molybdenum and Mo-0.5% Ti tests. These specimens, and unirradiated samples of molybdenum, Mo-0.5% Ti and Mo-50% Re that failed in a ductile manner, all had fracture surfaces covered with equiaxed dimples. A typical example, from an irradiated Mo-0.5% Ti sample, is shown in Fig. 8d.

Fracture surfaces of irradiated and control samples of niobium and Nb-1% Zr were also examined by SEM. Fracture surfaces of irradiated niobium and Nb-1% Zr tensile tested at 400°C are shown in Fig. 9. The two fractures appear similar, showing high local strains, and do not reveal any differences due to alloying. A control sample of Nb-1% Zr tested at 22°C and irradiated samples of the same material tested at 25 and 650°C were also found to be similar to the fractures shown in Fig. 9.

Fracture surfaces of the tantalum- and vanadium-base samples have not been examined.

Swelling and Microstructures

The available measurements on irradiation produced swelling are given in Table 5. In most cases the agreement between swelling measured by length changes and by immersion density is reasonably good, within at least a factor of 2. The presence of voids in the microstructure of niobium irradiated at 460°C suggests that some of the length measurements on this specimen set may have been incorrect. The other major discrepancy, for VANSTAR-7 irradiated at 690°C, cannot be resolved since microscopic examination is not yet available. The densification of T-111 is also surprising, and explanation must await microscopic evaluation of the samples. The other available swelling results in Table 5 indicate the presence of voids in samples where appreciable swelling is shown. These results suggest that little or no void formation occurred in Nb-1% Zr irradiated at 394 and 448°C nor in V-10% Cr irradiated at 496°C. Swelling measurements are not available for one of the molybdenum specimen sets, the Mo-0.5% Ti, and the low-temperature Mo-50% Re specimen set.

FIG. 9

Fracture Surfaces of Niobium and Nb-1% Zr Tensile Specimens. Tensile tests at 400°C, at a strain rate of 0.02 min⁻¹. (a,b) Niobium irradiated to 3.0×10^{22} n/cm² at 460°C. (c,d) Nb-1% Zr irradiated to 3.7×10^{22} n/cm² at 448°C. Magnification is 50× for a,c and 200× in b,d.

Microstructures of the tested molybdenum and Mo-0.5% Ti specimens were examined. The microstructure of molybdenum irradiated at 454°C and of Mo-0.5% Ti irradiated at 445°C are shown in Fig. 10a and b, respectively. The visible damage in both cases consists of barely resolvable dislocation loops and unresolved black spots, which are assumed to be loops also. These loops form discontinuous linear arrays, believed to be truly linear and not edge-on views of the "rafts" observed in molybdenum irradiated at lower temperatures (11). Voids were not visible in either of these samples, but the annealing results discussed below suggest that they were present in the as-irradiated molybdenum. The void microstructure in the molybdenum specimen irradiated at 857 to 1136°C is shown in Fig. 10c. This specimen contained a high concentration of small voids on a well-defined void superlattice. The concentration of dislocation segments and loops in this sample was quite low.

The necked region of a Mo-0.5% Ti specimen tested at 650°C was examined. The reduction in area at the sample was approximately 50%. The microstructure (Fig. 10d) was

TABLE 5
Swelling in Irradiated Refractory Metal Specimens

Alloy	Irradiation		Swelling, %		Qualitative Microstructural Description
	Temperature (°C)	Fluence, >0.1 MeV (n/cm ²) × 10 ²²	Geometry, $3\left(\frac{\Delta L}{L}\right)$	Density $\frac{-\Delta\rho}{\rho}$	
Molybdenum	455	3.5			Aligned loops and small voids
	455	4.0	1.1	1.5	Aligned loops and small voids
	857 to 1136	6.1	0.41	0.73	Ordered voids
Mo-0.5% Ti	445	3.0			Aligned loops, no voids
Mo-50% Re	393	1.5			
	855 to 1056	5.3	0.38	0.44	
Niobium	460	3.0	-0.14	0.71	Voids and dislocation networks
	440	3.7	0.96	0.69	
Nb-1% Zr	394	1.9	0.07	0.03	High density of tiny loops? No voids.
	448	3.7	0.04	0.20	
Tantalum	391	1.5	0.37	0.76	
	637	1.9	0.27	0.36	
T-111	414	1.9	-0.11	-0.02	
	643	1.9	-0.36	-0.34	
V-10% Cr	496	1.5	0.13	0.08	
	690	1.5	1.0	0.91	
VANSTAR-7	496	1.5	0.26	0.18	
	690	1.5	0.34	-0.05	

dominated by the dislocation structure generated during the tensile test. The substructure consisted of some dislocations in loose networks and well-developed subgrain boundaries and cell walls. Large areas were free of all defect structures, but some isolated areas or "islands" contained high concentrations of black spots and loops believed to be remnants of the irradiation-produced damage microstructure. There were no voids visible in this sample, nor was there any observable evidence of localized deformation.

In order to further evaluate the damage microstructure in the molybdenum and Mo-0.5% Ti irradiated near 450°C, samples of each were annealed for 1 hr in vacuum at 1050°C. Elen (12) has shown that this treatment will coarsen or remove the dislocation structure,

FIG. 10

Microstructure in Irradiated Samples of Molybdenum and Mo-0.5% Ti. All micrographs at 100,000 $\times$. (a) Molybdenum irradiated to 3.5×10^{22} n/cm 2 at 454°C. (b) Mo-0.5% Ti irradiated to 3.0×10^{22} n/cm 2 at 455°C. (c) Molybdenum irradiated to 6.1×10^{22} n/cm 2 at 857 to 1136°C. (d) Mo-0.5% Ti irradiated to 3.0×10^{22} n/cm 2 at 445°C and tested to failure at 650°C. Sample from neck region where local reduction in area was 50%.

but have little effect on the void microstructure. The microstructure of annealed molybdenum (Fig. 11a) contained a high density of small voids in a "patchy" distribution but with the voids on an ordered superlattice. Observation of voids in the irradiated molybdenum corrects our earlier report of the present data (5) that suggested the samples were void free. The annealed sample also contained a connected network of dislocations. The annealed Mo-0.5% Ti did not contain observable voids, but diffraction contrast (Fig. 11b) showed a highly connected dislocation network and numerous isolated loops. The absence of voids in the Mo-0.5% Ti sample is substantiated by unreported swelling measurements on a set of samples irradiated to similar conditions.

The microstructure in the niobium sample irradiated at 460°C is shown in Fig. 12a. This sample contained the dense dislocation distributions shown as well as a high concentration of small voids. Much of the dislocation structure seen was probably generated during tensile testing at 400°C, since this specimen sustained 4.8% uniform elongation before the onset of necking. The dislocation distribution appeared to be uniform, without any formation of cell structure. The microstructure in Nb-1% Zr irradiated at 448°C is complex and has not as yet been analyzed in detail. Some local deformation during the thinning process has been observed, as shown by the banding in Fig. 12b. This is believed

FIG. 11

Microstructure of Irradiated Samples Postirradiation Annealed 1 hr at 1050°C.

(a) Molybdenum irradiated to 4.0×10^{22} n/cm² at 455°C. Voids imaged in overfocused condition. (b) Mo-0.5% Ti irradiated to 3.0×10^{22} n/cm² at 445°C.

FIG. 12

Microstructures of Niobium and Nb-1% Zr. (a,c) Niobium irradiated to 3.0×10^{22} n/cm² at 460°C. (b,d) Nb-1% Zr irradiated to 3.7×10^{22} n/cm² at 448°C. As-irradiated structures shown in (a,b), structures after postirradiation anneal of 1 hr at 900°C shown in (c,d).

to be a result of dislocation channeling as observed in niobium tested after irradiation at 50°C (13).

Samples of niobium and Nb-1% Zr, cut from the same tested samples in which the as-irradiated microstructure was examined, were annealed in vacuum for 1 hr at 900°C. As for molybdenum, Elen (12) has shown that this treatment has little effect on the void microstructure. In the annealed niobium, the void microstructure is clearly visible (Fig. 12c). The voids again were small and numerous in this sample, with a "patchy" distribution and local variation in the void concentration. There is no apparent void ordering in this specimen. The dislocation content of this sample was greatly reduced by the anneal, leaving a few isolated loops and a large-scale dislocation network. The annealing of Nb-1% Zr reduced the microstructure from the unresolvable as-irradiated structure to easily resolvable features. The annealed structure contained a connected dislocation network with numerous dislocation loops (Fig. 12d). In contrast to the niobium, there were no detectable voids in the Nb-1% Zr.

Microstructural results on the Mo-50% Re, tantalum- and vanadium-base alloys are not available.

Discussion

These results suggest that both voids and dislocation loops make important contributions to irradiation strengthening in the bcc refractory metals. In niobium, in molybdenum irradiated at high temperature, and in molybdenum irradiated at 455°C and then annealed at 1050°C, the dominant microstructural feature is the high concentration of small voids. In these cases the dislocation component of the damage microstructure is much coarser than the void component. Yield strengths of these samples (Fig. 3) were several times those for unirradiated specimens and ultimate tensile strengths were greater than the yield strengths. Work hardening thus occurs together with some uniform elongation of these samples.

The behavior of Nb-1% Zr, Mo-0.5% Ti, and molybdenum irradiated at 455°C is somewhat different. For these materials the maximum load occurs at the yield and there is no ability to work harden. The result is no uniform elongation, even when total elongations approach 10% (Fig. 4). The microstructure in all of these samples was dominated by a very high concentration of small dislocation loops and black spots believed to be unresolved loops. In the Nb-1% Zr and Mo-0.5% Ti, both swelling measurements and microscopy of annealed samples confirmed the lack of voids. In molybdenum voids were present, but their concentration appeared to be lower than the loop concentration. The local yielding or "work softening" behavior of these specimens suggests that the deformation occurs by dislocation channeling, at least at small strains. Direct evidence of channeling during thinning of foils was seen in the Nb-1% Zr (Fig. 12b). Channeling has been

observed in molybdenum (14), niobium (13), and vanadium (15) irradiated at temperatures near room temperature and the effect on tensile properties has been described by Smidt (16). While the present observation of reduced work hardening rate (or work softening) due to channeling is consistent with Smidt's discussion (16), the mechanism of ductility reduction is not. Smidt suggests reduced values of both elongation and reduction in area are due to slip on a very few slip systems. In our results, reduced elongations at least in part reflect the very localized deformation, while the reduction in area was generally quite large. Furthermore, Fig. 10d shows that in the high-deformation region of a Mo-0.5% Ti specimen all evidence of local deformation vanished. Dislocations are mainly in subgrain boundaries and cell wall arrays suggesting that many, interacting slip systems were active in the later stages of deformation.

The deformation behavior of molybdenum and Mo-0.5% Ti is complicated by the competition between plastic deformation and brittle (cleavage) fracture. In these two materials the irradiation hardening frequently raised the yield stress above the cleavage stress. The result, failure in a mixed cleavage and grain boundary separation fracture, raises the DBTT to above 550°C for molybdenum and Mo-0.5% Ti irradiated at approximately 450°C and tested at a strain rate of 0.02 min^{-1} . At lower strain rates, the DBTT of irradiated material was somewhat lower. The behavior of molybdenum samples containing mainly voids is somewhat more complex. In the molybdenum irradiated between 857 and 1136°C, the DBTT at a strain rate of 0.02 min^{-1} was above 20°C but below 400°C. In this sample tested at 400°C and in the sample irradiated at 455°C, annealed at 1050°C and then tested at 400°C, some plastic strain occurred; but, after very little work hardening, the cleavage stress was reached and failure occurred in a brittle mode.

The results of irradiation on the strength and ductility properties of Mo-50% Re cannot be explained without further experimental results. The wide range in ultimate tensile stresses of irradiated samples suggests a behavior more complex than simply a DBTT shift. The data on V-10% Cr and VANSTAR-7 are also best left without attempted explanation until microstructural examination is available.

The results on tantalum and T-111 remain incomplete without microstructural examination, yet here an important effect of irradiation temperature is apparent. Neither the tantalum irradiated at 391°C nor the T-111 irradiated at 414°C was able to strain uniformly, suggesting a channeling-controlled deformation process. For both materials irradiated near 640°C, on the other hand, work hardening with uniform elongations in the range 1.9 to 6.6% occurred. The higher irradiation temperature has resulted in a form of damage more stable during deformation, not susceptible to channeling, but the microstructural damage state has not yet been examined. The swelling results, in Table 5, do not allow a simple voids vs loops conclusion to be drawn as was the case for the niobium and Nb-1% Zr comparison.

Acknowledgments

The author would like to thank the many people who contributed to this work, L. K. Egner, L. G. Rardon, and B. C. Williams for tensile tests, R. K. Bennett, Jr. and T. J. Henson for fractography, J. T. Houston for preparation of microscopy samples and E. E. Bloom and J. O. Stiegler for help and discussion in many areas. Thanks also to R. T. King and J. M. Leitnaker for manuscript review and to the Metals and Ceramics Division Reports Office for final copy preparation.

References

1. F. W. Wiffen, Radiation-Induced Voids in Metals, ed. by J. W. Corbett and L. C. Ianniello, p. 386. AEC Symp. Ser. 26, CONF-710601 (April 1972).
2. J. D. Elen, G. Hamburg, and A. Mastenbroek, J. Nucl. Mater. 39, 194 (1971).
3. B. L. Eyre, Defects in Refractory Metals, ed. by R. DeBatist, J. Nihoul, and L. Stals, p. 311. Studiecentrum voor Kernenergie, Centre d'Etude de l'Energie Nucleaire, Mol, Belgium (1972).
4. M. Kangilaski, Radiation Effects Design Handbook, Sect. 7. Structural Alloys, NASA CR-1873 (1971).
5. F. W. Wiffen, Fuels and Materials Development Program Quart. Progr. Rep. June 30, 1970, ORNL-4600, p. 114 and Fusion Technology Studies Progr. Rep. Dec. 31, 1972 ORNL-TM-4156, p. 27.
6. W. N. McElroy, J. L. Jackson, J. A. Ulseth, and R. L. Simons, EBR-II Dosimetry Test Data Analysis, BNWL-1402 (1970).
7. J. D. Jenkins, H. T. Kerr, and R. Q. Wright, Oak Ridge National Laboratory, personal communication, May 28, 1971 and May 17, 1973.
8. H. Farrar IV, Atomics International, personal communication, August 31, 1972.
9. H. Alter and C. E. Weber, J. Nucl. Mater. 16, 68 (1965).
10. A. R. Rosenfield, E. Votava, and G. T. Hahn, Ductility, p. 63. American Society for Metals, Metals Park, Ohio (1968).
11. J. L. Brimhall and B. Mastel, Radiat. Eff. 3, 203 (1970).
12. J. D. Elen, Voids Formed by Irradiation of Reactor Materials, ed. by S. F. Pugh, M. H. Loretto, and D.I.R. Norris, p. 51, British Nuclear Energy Society, Harwell (1971).
13. R. P. Tucker, M. S. Wechsler, and S. M. Ohr, J. Appl. Phys. 40, 400 (1969).
14. J. L. Brimhall, Trans. AIME 233, 1737 (1965).
15. R. J. Arsenault, K. Kuo, and Y. Huang, Mechanical Behavior of Materials. Vol. I - Deformation and Fracture of Metals, p. 48, The Society of Materials Science, Japan (1972).
16. F. A. Smidt, Jr., Dislocation Channeling in Irradiated Metals, NRL Rep. 7078 (1970).