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TENSILE PROPERTIES OF NEUTRON IRRADIATED TZM AND TUNGSTEN *

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The effect of neutron irradiation on the elevated temperature tensile properties of TZM and tungsten has been experimentally determined. Specimens were irradiated at a temperature of approximately 385°C to fluences of 0.4 and 0.9×10^{22} n/cm^2 ($E > 0.1$ MeV). Test parameters for both control and irradiated specimens included strain rates from 3×10^{-4} to 1 sec^{-1} and temperatures from 22 to 927°C. The results of these tests were correlated with a rate-temperature parameter ($T \ln A/\dot{\epsilon}$) to provide a concise description of material behavior over the range of deformation conditions of this study. The yield strength of the subject materials was significantly increased by decreasing temperature, increasing strain rate, and increasing fluence. Ductility was significantly reduced at any temperature or strain rate by increasing fluence. Cleavage fractures occurred in both unirradiated and irradiated specimens when the yield strength was elevated to the effective cleavage stress by temperature and/or strain rate. Neutron irradiation for the conditions of this study increased the ductile-to-brittle transition temperature of TZM by about 215°C and tungsten by 150°C.

L'effet de l'irradiation aux neutrons sur les propriétés en traction à température élevée du TZM et du tungstène a été déterminé expérimentalement. Les échantillons étaient irradiés à une température de 385°C environ et sous des flux de 0,4 et $0{,}9 \times 10^{22}$ n/cm^2 ($E > 0{,}1$ MeV). Les paramètres d'essais pour les échantillons témoins et irradiés étaient la vitesse de déformation comprise entre 3×10^4 et 1 sec^{-1} et la température comprise entre 22 et 927°C. Les résultats de ces essais ont été corrélés avec un paramètre température-vitesse ($T \log A/\dot{\epsilon}$) pour obtenir une description concise du comportement du matériau sur tout le domaine des conditions de déformation étudiées. La limite élastique des matériaux augmentaient d'une façon significative avec la température décroissante, avec la vitesse de déformation croissante et avec des flux coissants. La ductilité était sensiblement réduite à toute température ou à toute vitesse de déformation avec le flux croissant. Des fractures par clivage se produisaient à la fois dans les échantillons non irradiés et irradiés quand la limite élastique était élevée à la contrainte effective de clivage par la température et/ou par la vitesse de déformation. L'irradiation aux neutrons pour les conditions étudiées augmentait la température de transition ductile-fragile du TZM de 215°C environ et du tungstène de 150°C

Der Einfluss der Neutronenbestrahlung auf die Festigkeitseigenschaften von TZM und Wolfram bei höheren Temperaturen wurde experimentell untersucht. Die Proben wurden bei etwa 385°C mit einer Dosis von 0,4 und $0{,}9 \times 10^{22}$ n/cm^2 ($E > 0{,}1$ MeV) bestrahlt. Die Versuchsparameter für die Kontroll- und bestrahlten Proben waren Dehnungsgeschwindigkeiten zwischen 3×10^{-4} und 1 s^{-1} und Temperaturen zwischen 22 und 927°C. Die Ergebnisse werden mit einem Geschwindigkeits-Temperatur-Parameter $T \ln A/\dot{\epsilon}$ korreliert. Dadurch wird eine prägnante Beschreibung des Materialverhaltens im Bereich der Verformungsbedingungen dieser Arbeit erzielt. Die Streckgrenze des Materials steigt mit abnehmender Temperatur, zunehmender Dehungsgeschwindigkeit und zunehmender Dosis stark an. Durch zunehmende Dosis wird die Zähigkeit bei jeder Temperatur oder Dehnungsgeschwindigkeit erniedrigt. Spaltbrüche treten in unbestrahlten und bestrahlten Proben auf, wenn die Streckgrenze bis zur effektiven Spaltbruchspannung durch die Temperatur und/oder Dehnungsgeschwindigkeit erhöht wird. Durch die Neutronenbestrahlung unter den Bedingungen dieser Arbeit wird die Übergangstemperatur vom zähen zum spröden Bereich um etwa 215°C für TZM und 150°C für Wolfram erhöht.

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1. Introduction

Design of liquid metal fast reactor systems has resulted in the selection of TZM and tungsten for unique applications in certain components. As such, these materials will be exposed to neutron irradiation at elevated temperatures for extended lengths of time. Since this exposure could significantly alter the structural behavior of these materials it is essential that the mechanical properties and associated ductile-to-brittle transition temperature be known for anticipated service conditions.

The mechanical properties of unirradiated TZM and tungsten have been extensively characterized [1–6], and limited information is available on the effects of neutron irradiation on these properties [7–9]. Kangilaski [7] has summarized early irradiation effects data and has shown that mechanical properties, particularly the ductile-to-brittle transition temperature, are significantly influenced by neutron irradiation. Unfortunately, these data were obtained from specimens irradiated in thermal reactors (irradiation temperatures ranged from ~49 to 104°C) and are therefore not directly applicable to liquid metal fast reactor systems. Wiffen [9] recently performed an EBR-II irradiation experiment on several refractory metals and alloys including molybdenum and Mo-0.5 Ti. Irradiation conditions for his study included a temperature of 454°C and a fluence of 3.0×10^{22} n/cm^2 ($E > 0.1$ MeV). Irradiation at this temperature and fluence significantly increased the strength and decreased the ductility of molybdenum and Mo-0.5 Ti and resulted in a ductile-to-brittle transition temperature of at least 487°C. No fast reactor irradiation effects data on the mechanical properties of TZM or tungsten were found in the literature.

In the present study the effects of fast reactor irradiation on the tensile properties and ductile-to-brittle transition temperature of TZM and tungsten were determined. Irradiation conditions included temperatures from 371–388°C and fluences from 0.4 to 0.9×10^{22} n/cm^2 ($E > 0.1$ MeV). Test parameters on unirradiated and irradiated materials included temperatures from room temperature to 871°C and strain rates from 3×10^{-4} to 1 sec^{-1}. Results of the completed tests were correlated with a rate-temperature parameter ($T \ln A/\dot{\epsilon}$) to provide a detailed description of irradiation effects.

2. Experimental procedure

2.1. Test material and specimens

The TZM used in this study was obtained from 10.5 cm diameter wrought bar which had been stress-relived at 1315°C for $2\frac{1}{2}$ hour and the tungsten from 0.63 cm diameter centerless ground wrought rod which had been stress-relieved at 1000°C for 30 minutes. Originally, the TZM was made by consumable electrode vacuum arc melting and the tungsten by isostatic cold pressing and sintering. The grain size was ASTM 7–9 for the TZM and 11–12 for the tungsten. The chemical compositions for the test materials are given in table 1.

Miniature buttonhead tensile specimens were fabricated from the longitudinal forming direction of the rod materials and subsequently used for control testing and for the irradiation experiment. All specimens had gage lengths that were 2.86 cm long and 0.32 cm diameter.

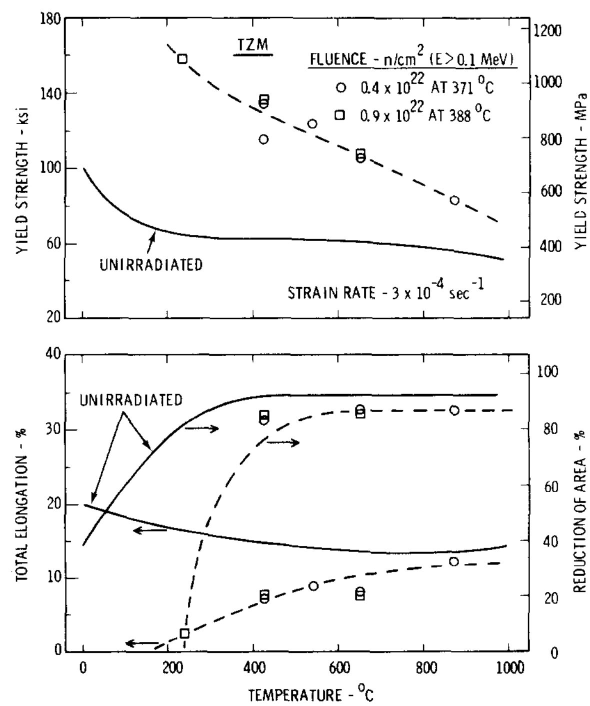

Fig. 1. Temperature dependence of the strength and ductility of unirradiated and irradiated TZM.

Table 1
Chemical composition of TZM and tungsten

TZM-heat 8967 (wt%)

Ti	Zr	C	O_2	H_2	N_2	Fe	Ni	Si	Mo
0.45	0.090	0.013	0.0007	$<10^{-4}$	0.0002	0.003	$<10^{-3}$	0.002	Bal.

Tungsten-heat B-434 (ppm)

Al	Ca	Cu	Cr	Fe	Mg	Mn	Ni	Si	Sn	Mo	Na	K	W
<1.0	<0.5	<0.1	<1	13	<0.5	<1	<1	1.1	<1	16	6	<15	Bal.

2.2. Specimen irradiation

The Experimental Breeder Reactor-II (EBR-II) at the Idaho National Engineering Laboratory was used for the irradiation experiment. This experiment was conducted in a Row 2 core position in structural materials subassembly designated X-195. The test specimens were irradiated in EBR-II sodium coolant at reactor ambient temperature (371–388°C) to fluences from 0.4 to 0.9 × 10^{22} n/cm^2 ($E > 0.1$ MeV). The approximate location of the specimens with respect to reactor midplane was –24 and 0 cm.

2.3. Test procedure

All tensile and high strain rate tests were performed on hard beam machines. An Instron Tensile Machine was utilized for all tests at a strain rate to 3×10^{-4} sec^{-1}, and an MTS Electro-Hydraulic test machine for strain rates from 3×10^{-2} to 1.0 sec^{-1}. Both machines were operated at constant crosshead velocity and strain rate values given here are based on the crosshead velocity and initial specimen gauge length. Ductility data reported in this paper were obtained from pre- and post-test specimen measurements. Detailed test procedures have been reported previously [10].

3. Results and discussion

Tensile testing of unirradiated and irradiated TZM and tungsten was performed at a variety of test temperatures and strain rates to provide a description of unirradiated behavior and to reveal the effects of neutron irradiation. Property discussions presented in this paper include the upper yield strength, total elongation, and reduction of area because of their significance to the ductile-to-brittle transition temperature. Tabular data for the tests on irradiated material are included in table 2.

The effect of test temperature at a strain rate of 3×10^{-4} sec^{-1} on the yield strength and ductility of irradiated TZM is illustrated in fig. 1. Unirradiated data (solid lines) are included for comparison. For all test temperatures neutron irradiation significantly increases the yield strength and decreases the ductility (total elongation and reduction of area) of the test material. Also, convergence of irradiated behavior with unirradiated behavior is noted with increasing test temperature. No distinct effect of increasing fluence on properties is observed. Of significance is the fact that very low ductility is observed in irradiated material at a test temperature of 232°C while good ductility is observed in unirradiated material. Similar effects of irradiation are observed for the elevated temperature tensile properties of tungsten (fig. 2).

To determine the increase in ductile-to-brittle transition temperature as a result of neutron irradiation the yield strength of TZM and tungsten were plotted versus the rate-temperature parameter, $T \ln A/\dot{\epsilon}$, (where T = absolute temperature, K; A = constant, 10^8; and $\dot{\epsilon}$ = strain rate, sec^{-1}), developed from rate-theory by Bennett and Sinclair [11]. The resulting correlations (figs. 3 and 4) provide a means to predict the yield strength of the test materials at any temperature or strain rate within the range of this study and thereby

Table 2
Tensile properties of neutron irradiated TZM and tungsten

Material	Irradiation condition		Test	$\dot{\epsilon}$	Strength (Ksi)		Elongation (%)		Reduction of
	Fluence (n/cm^2) *	Temp. (°C)	temp. (°C)	sec^{-1}	Yield	Ultimate	Total	Uniform	area (%)
TZM	0.4×10^{22}	371	427	3×10^{-4}	134.8	134.8	6.8	0.3	83.5
			427		115.4	115.4	8.4	0.6	87.7
			538		123.2	123.2	9.0	0.6	88.5
			649		106.1	106.1	7.8	0.4	86.9
			232	3×10^{-2}	141.1	141.1	0	0	0
			427		120.3	120.3	8.3	0.6	81.3
	0.9×10^{22}	388	871	3×10^{-5}	82.4	82.4	12.2	1.1	86.9
			232	3×10^{-4}	151.2	151.2	2.6	0.2	2.4
			427		135.4	135.4	7.6	0.3	86.1
			649		107.0	107.0	7.6	0.4	86.1
			232	3×10^{-2}	132.7	132.7	5.2	0.1	46.7
			427		118.4	118.4	2.0	0.1	1.6
Tungsten	0.5×10^{22}	371	427	3×10^{-4}	164.2	164.2	6.8	0.9	66.6
			427		174.0	174.0	3.5	0.9	3.2
			649		118.7	118.7	6.4	0.4	77.1
			760		105.6	105.6	8.5	0.7	89.3
			232	3×10^{-3}	221.9	221.9	0.5	0.1	0.4
			427	3×10^{-2}	165.4	165.4	2.0	0.2	34.9
			649		135.4	135.4	8.8	0.8	76.4
	0.9×10^{22}	382	871	3×10^{-5}	80.1	80.1	10.6	0.6	91.1
			232	3×10^{-4}	197.6	197.6	0.8	0.5	0.8
			427		152.7	152.7	7.2	0.2	65.6
			538		136.6	136.6	7.2	0.8	77.0
			649		112.3	112.3	6.2	0.4	75.6
			427	3×10^{-2}	178.7	178.7	3.8	0.3	4.1
			649		133.6	133.6	8.7	0.9	75.6

* $E > 0.1$ MeV

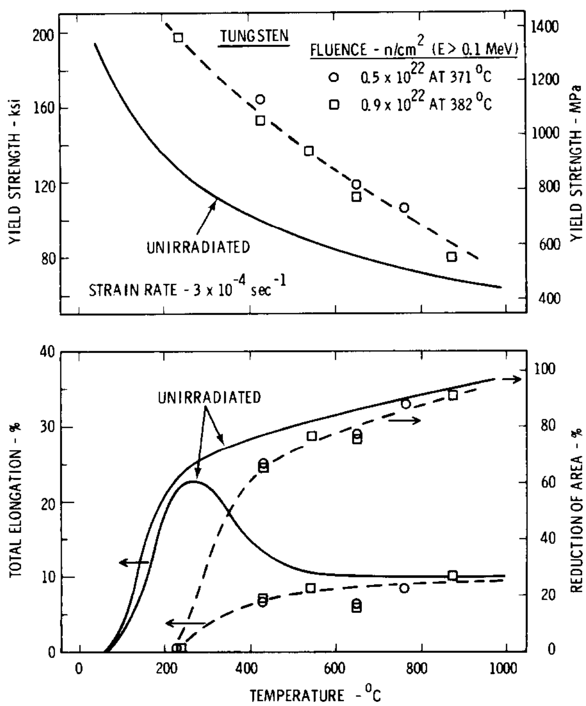

Fig. 2. Temperature dependence of the strength and ductility of unirradiated and irradiated tungsten.

also indicate ductile-to-brittle transition temperatures. The rate-temperature parameter correlation of yield strength for TZM is illustrated in fig. 3. As shown in the figure, the yield strength of unirradiated material increases rapidly as the parameter decreases below ~18 × 10^3 K. Above this parameter value the yield strength remains essentially constant. Neutron irradiation significantly increases the yield strength of TZM for all test conditions (parameter values) of this study. At the higher parameter levels (>28 × 10^3 K) behavior of irradiated material converges with that of unirradiated material. This convergence may result from irradiation damage annealing as a result of the high test temperatures (649–871°C) at these parameter levels. No effect of increasing fluence on yield strength is observed.

The ductile-to-brittle transition temperatures of unirradiated and irradiated TZM can be determined from the correlation in fig. 3. For unirradiated TZM cleavage fractures were observed in tensile specimens when the yield strength was elevated to 130–140 Ksi (895–965 MPa) by increasing strain rate or decreasing test temperature. From the correlation this yield strength is observed at a parameter value of ~5.5 × 10^3 K. Using this parameter value and assuming

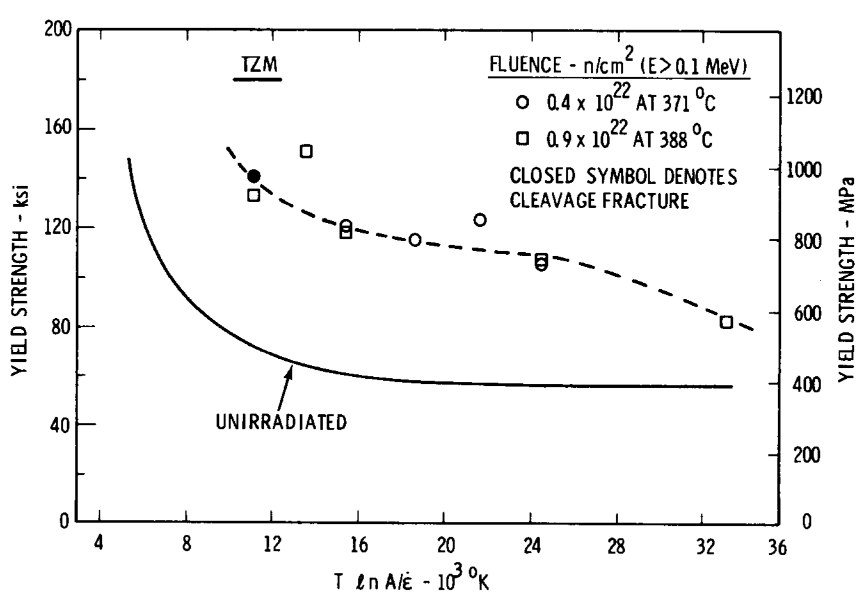

Fig. 3. Rate-temperature parameter representation of the yield strength of unirradiated and irradiated TZM.

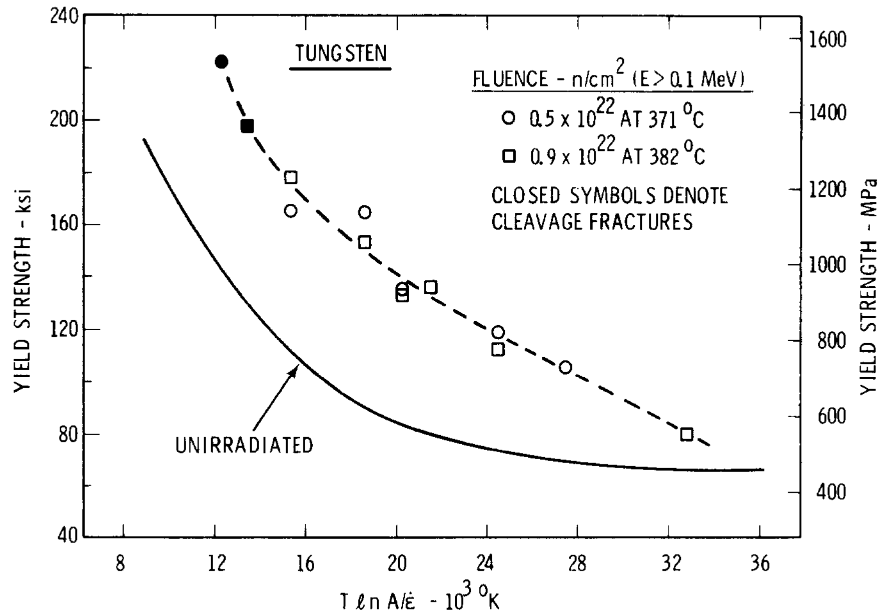

Fig. 4. Rate-temperature parameter representation of the yield strength of unirradiated and irradiated tungsten.

that a conventional tensile test is performed at a strain rate of $\sim 3 \times 10^{-4}\ sec^{-1}$, the test temperature to produce cleavage fracture (ductile-to-brittle transition temperature) can be calculated. For the subject TZM the transition temperature is calculated to be $\sim -85°C$. This temperature is consistent with values in the literature [12] for stress-relieved material.

Similarly, the transition temperature for irradiated TZM can be determined. For the irradiated material cleavage fractures were observed at parameter values to about 11.1×10^3 K which corresponds to a transition temperature of 150°C (at a strain rate of $3 \times 10^{-4}\ sec^{-1}$). Thus, irradiation to the conditions of this study increased the transition temperature by 235°C.

Wiffen [9] has reported tensile properties on EBR-II irradiated Mo-0.5 Ti. Irradiation conditions for his study included a temperature of 454°C and a fluence of $3.0 \times 10^{22}\ n/cm^2$ ($E > 0.1$ MeV). Post irradiation tensile tests on this material indicated that the ductile-to-brittle transition temperature was increased from about −73°C for unirradiated material to at least 480°C for the irradiated material. This increase in transition temperature which is much larger than that observed for TZM in the present study likely results because Wiffen's test material was irradiated in the recrystallized condition and possibly because of the slightly different test material and the higher fluence.

The effect of neutron irradiation on the rate-temperature correlation of the yield strength of tungsten is presented in fig. 4. As observed for TZM, the yield strength of unirradiated and irradiated tungsten increases with decreasing parameter value. Also, at the higher parameter values convergence of irradiated behavior with unirradiated behavior is noted. No effect of increasing fluence is observed. For unirradiated material cleavage fractures occurred at a yield strength of 190 to 200 Ksi (1240 to 1319 MPa) and at a test temperature of 65°C. Irradiation of tungsten to the fluence levels of this study resulted in a transition temperature of 230°C or an increase of 165°C.

Correlation of the total elongation and reduction of area data obtained on the unirradiated and irradiated materials in this study with the rate temperature parameter is precluded by adiabatic heating. Significant abiatic heating occurs during deformation of the test specimens at strain rates above 0.1 sec^{-1} [13]. As such, specimen temperatures are increased and the resulting ultimate strength and ductility values are not an accurate representation of material behavior at the apparent test temperature.

4. Conclusions

The following observations and conclusions may be made regarding the results of this study.

– Tensile properties of TZM and tungsten are strongly strain rate and temperature dependent.

– Neutron irradiation at 371–388°C to a fluence of 0.9×10^{22} n/cm^2 ($E > 0.1$ MeV) significantly increased strength properties and reduced ductility at test temperatures below 649°C.

– Neutron irradiation at the conditions of this study increased the ductile-to-brittle transition temperature of TZM from –85°C to 150°C and of tungsten from ~65°C to 230°C.

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