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REVIEW OF IRRADIATION HARDENING AND EMBRITTLEMENT EFFECTS IN REFRACTORY METALS RELEVANT TO NUCLEAR THERMAL PROPULSION APPLICATIONS

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Nuclear thermal propulsion (NTP) is advantageous for future crewed interplanetary missions because of its capability for high specific impulse, thrust, large abort windows, and good cargo capacity. A fuel under consideration for use in NTP systems is a ceramic metallic (cermet) consisting of fissile fuel particles, such as uranium dioxide (UO_2) or uranium nitride (UN), suspended in a structural refractory metal matrix, such as molybdenum (Mo) or tungsten (W). When structural materials are irradiated at low temperatures (below ~0.35 times the melting point) to low doses (0.001 to 0.1 displacements per atom), irradiation hardening and embrittlement may occur. This phenomenon increases the yield strength of materials, but also causes a decline in ductility and an increase in the ductile to brittle transition temperature (DBTT). During operation, large temperature gradients are present throughout the core, causing regions of the fuel element to operate at relatively low temperatures and receive neutron doses conducive to irradiation hardening. A comprehensive literature review was conducted to determine the effects of low-fluence neutron irradiation of pure and alloyed Mo and W. Irradiation hardening occurs in Mo and W up to 1070 and 1100 K respectively, with significant changes in the mechanical properties even at very low neutron doses. The reviewed literature relevant to NTP applications are summarized, knowledge gaps identified, and implications of mechanical property evolution on fuel performance and operating margins discussed.

I. INTRODUCTION

Nuclear thermal propulsion (NTP) offers significant advantages to alternative in-space propulsion methods for crewed interplanetary missions due to its capability for high specific impulse (850 – 1000 s) and thrust (25 – 250 klbf), as well as the potential to enable large abort windows and increase mission payload capacity. NTP systems utilize a nuclear reactor to heat a propellant, typically hydrogen (H_2), which is expanded through a nozzle to produce thrust. Ceramic metallic (cermet) nuclear fuels are a leading option for NTP applications. Cermet fuels of interest consist of ceramic nuclear fuel particles, typically uranium dioxide (UO_2) or uranium nitride (UN), suspended in a refractory metal matrix[1,2]. The structural material properties of the cermet depend primarily on the properties of the metal matrix. Molybdenum (Mo) and tungsten (W) are the two refractory metals of interest due to their high melting temperatures, 2900 and 3700 K respectively, as well as their chemical compatibility with the propellant.

Current studies have been focused on cermet fuel fabrication and the non-nuclear testing of subscale Mo and W cermet samples in hot flowing hydrogen[3,4,5,6,7,8]. However, during operation in a prototypic NTP system, the fuel must be able to withstand both the demanding conditions stemming from high temperature operation (high temperature stability, corrosive interaction with the propellant, large thermomechanical stresses, thermal shock incurred under multiple burns, etc.) in addition to those stemming from the intense irradiation intrinsic to the fission process. In order to verify fuel performance and define appropriate operating margins required for NTP fuel down selection/screening and engine design activities, both thermal and irradiation effects must be well characterized to more accurately predict engine performance. In order to quantify the effects stemming from neutron irradiation, a comprehensive literature review of radiation hardening and embrittlement of Mo and W was performed and is presented in this report.

I.A. Background: Radiation Degradation Mechanisms

Radiation can affect material properties in different ways, depending on the dose and the irradiation temperature. Dose is typically measured in terms of displacements per atom (dpa), representing the number of instances each atom of a material is knocked out of its crystal lattice site to a new site[9]. Irradiation hardening and embrittlement occurs in materials irradiated at temperatures below 0.4 T_M (absolute melting temperature) at low doses typically below 1 dpa[10]. The mechanisms of irradiation hardening and embrittlement will be discussed in section I.C. Irradiation of materials between 0.3 to 0.6 T_M to doses above 10 dpa can lead to radiation induced precipitation[10]. Irradiation creep is another adverse radiation effect, occurring at temperatures above 0.45 T_M and becoming pronounced at doses above 10 dpa[9]. At high neutron doses, typically >10 dpa, and around 0.3 to 0.6 T_M, volumetric void swelling is a major concern in materials[9]. Finally, at high temperatures (above 0.5 T_M) helium embrittlement typically becomes a problem if concentrations of transmutant He of ~10 atomic parts per million or higher occur[11]. Since an NTP system will only operate for relatively short times, attaining low neutron doses, only irradiation hardening and embrittlement are expected to affect the fuel element. Additionally, alternative sources of irradiation damage phenomena to in-reactor fuel and structural materials including fission product generation and attenuation, and nuclear transmutation and decay are not considered in this review.

I.B. Anticipated NTP Reactor Operating Conditions

For NTP missions under investigation, the propulsion system would operate for multiple burns, however total cumulative operating time would be only ~85 – 102 minutes for crewed missions to a nearby planet such as Mars.[12] This short operation lifetime produces low total neutron fluences compared to terrestrial power reactors (multiyear operation) and minimal irradiation effects are predicted for most structural reactor materials.

Irradiation effects due to neutron damage typically exhibit a temperature and fluence dependence. In order to assess the relative fluence and temperature gradients expected during material system operation within the engine, historic test data was evaluated. In 1970, the Pewee-1 rocket (epithermal neutron spectrum reactor) was tested at the Nevada test site and data was collected during the operation. During the test, the reactor ran for 40 min at peak power[13]. The Pewee-1 was a similar sized reactor to one proposed for a crewed mission to Mars[14]. The neutron flux and temperature profiles collected from the Pewee-1 test are expected to be similar during the testing of future NTP systems. Figure 1 summarizes the average operating temperature and the total fluence of the fuel unit as a function of axial position in the reactor[15].

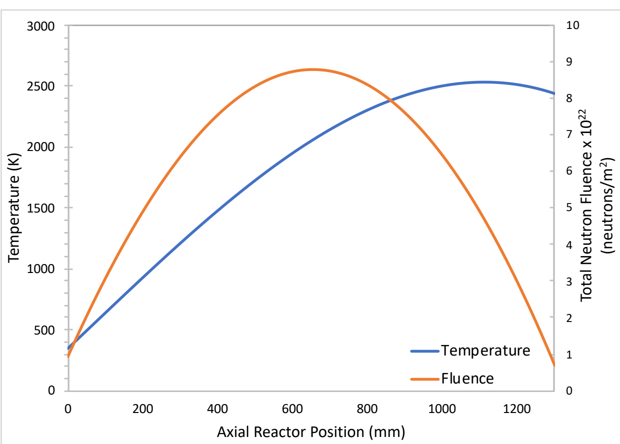

Fig. 1. Temperature and Fluence profile for a 1300 mm NTP fuel element modeled after the Pewee-1 test.

This test provides a good benchmark for the average temperature and fluence profile within future tested NTP reactors. From Pewee-1 test data, it is expected average maximum operating temperatures of ~2500 K, at the hot end of the fuel (highest axial position) with minimum temperatures below 500 K near the inlet region (lowest axial position). Reactor fluences, after 40 minutes of operation time, ranged from 0.5 to 8.5 x 10^{22} neutrons/m^2 dependent upon axial position. Since these values only represents the axial centerline of the cylindrical reactor core, greater variation in fluences and temperatures are anticipated at the radial periphery. However, this analysis still provides a useful approximation of maximum experienced dose for initial comparison to literature data.

In order to compare data from past irradiation studies, approximate damage levels and temperatures of a future NTP system were modeled. Since the Pewee-1 test represents a single 40-minute burn, to estimate the damage attained on a crewed interplanetary mission, this fluence can be scaled to be representative of the multiple burns required for crewed missions. Using fast reactor estimations from Gabriel et al., 3.43 x 10^{-26} dpa/fluence for Mo and 1.24 x 10^{-26} dpa/fluence (fluence in units of neutron/m^2)[16], a maximum and minimum damage were determined. For W cermet core, damage levels of 4.4 x 10^{-4} – 5.5 x 10^{-3} dpa were predicted for 5 burns. For a Mo core operating under the same conditions, the damage is slightly higher with a minimum and maximum of 1.2 x 10^{-3} and 0.015 dpa, respectively.

I.C. Irradiation Hardening and Embrittlement

Due to the low accumulated neutron doses, the only radiation degradation mechanism likely to occur is radiation hardening and embrittlement[17,18]. This phenomenon has been observed in refractory metals for the last sixty years. Radiation hardening in refractory metals typically becomes evident even at low neutron damage levels around 0.01 dpa and at temperatures below 0.3 to 0.4 T_M[17,18].

Radiation hardening occurs when atoms are knocked out of their crystal lattice. This creates a high density of defect clusters that become obstacles to dislocation motion in the matrix. As temperature increases, the density of these defect clusters decreases and the dislocation motion in the matrix is enhanced[10]. At a certain temperature, dependent on the material, annealing can occur reverting the material back to its unirradiated properties. The primary defect cluster geometries observed in irradiated refractory metals are dislocation loops, aligned "rafts" of small loops, and cavities. Cavity formation is typically not observed until higher damage levels (above 0.1 dpa)[19]. Radiation hardening in refractory alloys produces an increase in the strength of the metal or alloy. While this is potentially a beneficial result, the hardening also produces a dramatic loss in ductility (decrease in uniform and total elongation). This also increases the potential of the material for brittle fracture[18]. As a body centered cubic (BCC) material like Mo and W undergoes radiation hardening, it also experiences a shift in the ductile to brittle transition temperature (DBTT), or the point where the material transitions between ductile and brittle fracture[10]. As hardening occurs, the DBTT shifts upward. If this DBTT shifts too high, parts of an NTP system could be operating in the brittle fracture region and would be prone to prompt fracture when exposed to thermomechanical stress.

II. LITERATURE REVIEW: IRRADIATION HARDENING, EMBRITTLEMENT IN W AND MO

II.A. Radiation Hardening and Embrittlement in pure Mo and Mo Alloys

II.A.1. Molybdenum

There only exist a few studies conducted at low fluences for comparison of effects expected during operation of Mo in an NTP system. A study conducted by Li et al. neutron irradiated Mo samples at 353 K to doses of 7.2 x 10^{-5}, 7.2 x 10^{-4}, 7.2 x 10^{-3}, 7.2 x 10^{-2}, and 0.28 dpa[19]. These samples were then tensile tested at various temperatures between 223 and 373 K. The yield stress from these and other tests are shown in Figure 2.

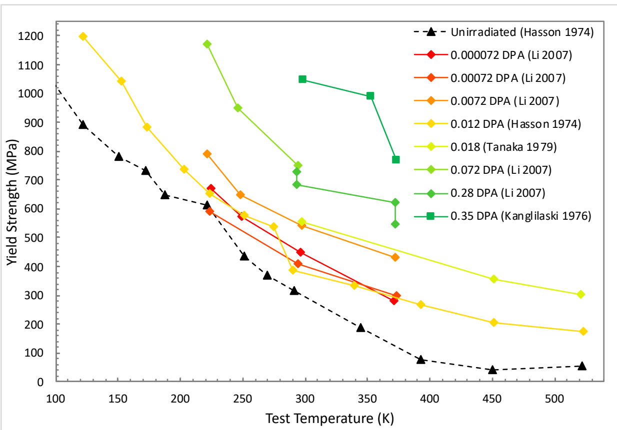

Fig. 2. Yield strength of Mo irradiated between 340 and 370 K to different damage levels (0.000072 – 0.35 dpa).

All of the samples experienced an increase in yield strength, representative of irradiation hardening. The study shows a decreasing yield strength with increasing testing temperature corresponding to the increase in ductility of the material. At dose at and above 7.2 x 10^{-2} the samples fractured during testing, indicating pronounced embrittlement of the tensile specimens.

A study by Hasson et al.[20] tensile tested Mo neutron irradiated between 333 and 353 K to fast fluences similar to those of Li et al.; results are also displayed in Figure 2. They observed similar trends, with only minor differences in yield strengths at similar doses. Yield strength data from tensile tests conducted by Tanaka et al. on samples irradiated to 0.018 dpa match closely with data from Li et al.'s 0.0072 dpa samples[21]. Possible differences in specimen purity (particularly oxygen content), grain size, or testing artifacts might be responsible for these discrepancies. Tanaka et al. also studied the effects of post irradiation annealing. Annealing up to 780 K had little to no effect on the radiation hardening of the samples. However, tensile tests on samples irradiated to 1300 K or higher resulted in a reduction of yield stress to values similar to unirradiated Mo. Nemoto et al. also studied the effects of annealing on radiation hardening and embrittlement testing samples at 681, 874, and 1072 K. The samples tested at 681 K experienced no annealing whereas the sample at 874 K displayed a slight decrease in yield strength. The samples tested at 1072 K experienced a significant decrease in yield strength and recovered ductility[22].

The data from Figure 2 was replotted in Fig. 3 to look at the effects of dose on yield strength. Four curves were generated, representing irradiation and testing at 295, 340, 375, and 520 K [19,20,21]. Once again, as the irradiation temperature increases, the yield strength decreases. Irradiation hardening saturation for the 295 and 375 K irradiation temperatures appears to begin at around 0.072 dpa. For the samples irradiated at 340 K, irradiation hardening saturation appears to begin much earlier at around 0.0072 dpa. More experiments are needed to determine the exact dose where irradiation saturation occurs over a wide range of NTP-relevant temperatures.

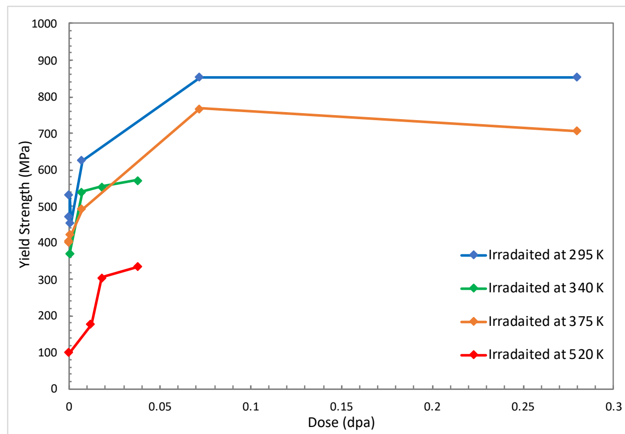

Fig. 3. Effect of dose on irradiation hardening.

Other studies have investigated effects of irradiation hardening on ductility by measuring the decline in uniform and total elongation. In the same study as above, Byun et al. measured the uniform elongation in samples irradiated and tested at 330 K to doses less than 0.1 dpa[23]. After achieving a dose as low as 0.001 dpa, uniform elongation dropped to 0 %. Other studies found decreases in total elongation as high as 10% after irradiation to 0.045 dpa[24].

Mo experiences significant radiation hardening but the magnitude of the effects depends heavily on the irradiation dose, irradiation temperature, and the testing temperature. As irradiation temperature increases above ~850 K, the magnitude of irradiation hardening decreases until around 1100 K where full annealing occurs. Also, as Mo is irradiated, the tensile DBTT will increase from 280 K by up to 80 K[25]. There is very little data on the increase of the DBTT for Mo at doses relevant to NTP.

II.A.2. Mo Alloys

Unirradiated Mo has relatively low ductility compared with other metals. To improve ductility, Mo is typically alloyed with 1-50 weight % (wt%) rhenium (Re). TZM is another common commercial alloy of Mo, using small amounts of titanium and zirconium. Several studies have been conducted on the effects of irradiation hardening in these Mo alloys.

In one study, Singh et al. studied radiation effects for both TZM and Mo-5% Re[26]. The samples were irradiated to 0.16 dpa at 320 and 373 K. The irradiated samples of both the Mo-5wt% Re and TZM saw significant increases in yield and ultimate tensile strengths, to a magnitude greater than in pure Mo. Also, the study found a drastic decrease in ductility as the irradiated samples uniform and total elongations decreased to less than 1%. These samples experienced brittle fracture when tested at 295 K.

II.B. Radiation Hardening and Embrittlement in pure W and W Alloys

II.B.1. Tungsten

Most of the irradiation studies conducted for W have been completed at higher fluences than relevant for NTP applications, since W was being examined for fusion energy structural components. The studies reported irradiation hardening effects up to 9 dpa[27].

Kangilaski et al. compiled early published data on the effects of neutron doses and irradiation temperatures on W[28]. Samples were irradiated to 0.12 dpa at 343 K and tested at 643 K. The yield strength of the samples increased with dose but began to level off around 0.12 dpa. Radiation hardening also introduces an accompanying pronounced reduction of ductility. Figure 4 demonstrates the decline in ductility (total elongation) with respect to neutron dose. After about 0.04 dpa, the total elongation drops below 1 % (irradiation tests were not performed for doses between ~0.006 and 0.04 dpa). Tensile specimens exposed to doses higher than 0.04 dpa experienced brittle fracture during the testing.

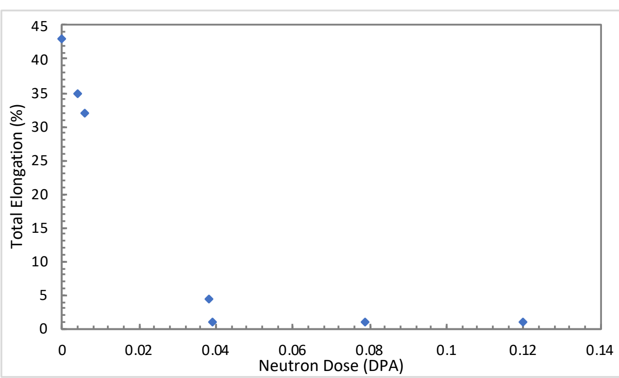

Fig. 4. Decrease in total elongation with respect to neutron dose in W irradiated at 343 K.

In order to determine the temperature dependence of irradiation hardening in W, Hu et al. neutron irradiated samples from 360 to 1123 K to doses of 0.03 - 2.2 dpa[29]. The study reported no significant dependence on irradiation temperature for radiation hardening in irradiated W and W alloys between 360 and 1123 K.

In another study, Barabash et al. tested W neutron irradiated to 4.2 x 10^{23} n/m^2 (E > 0.1 MeV) (0.0084 dpa) at 373 K and from 5.3 x 10^{24} to 5.6 x 10^{25} n/m^2 (E > 0.1 MeV) (0.1 – 1.1 dpa) at 373 and 550 K[30]. These samples were tested by bending and using tensile tests to determine the corresponding DBTT. While inaccurate, these tests can approximate DBTTs and illustrate general trends. There was a significant increase in the DBTT determined by the tensile tests in the W sample irradiated to 0.0084 dpa compared to the unirradiated W, bringing the DBTT to almost 900 K. Also, for the bend test, there was a constant increase in the DBTT until around 0.87 dpa, at which point the measured DBTT remained constant. This suggests that at 550 K, the saturation point for radiation hardening is 0.87 dpa. The increase in DBTT can cause significant issues to NTP reactors as the cooler parts of the fuel element will be operating in the brittle region of the material.

II.B.2. W Alloys

W alloys typically consist of W mixed with up to 50 wt% Re. Several studies were conducted to test the effect of Re content on the effects of radiation hardening. One study compared the Vickers Hardness of W-Re samples containing 0, 3, 5, 10, and 27 wt%[31]. These samples were neutron irradiated to damage levels of 0.15, 0.37, 0.96, 1.0, and 11 dpa. Figure 5 illustrates these results. There is a definite trend of increasing hardness with increasing Re content for all of the samples tested. Similar to the Mo alloys, the alloying of W increases the effects of irradiation hardening.

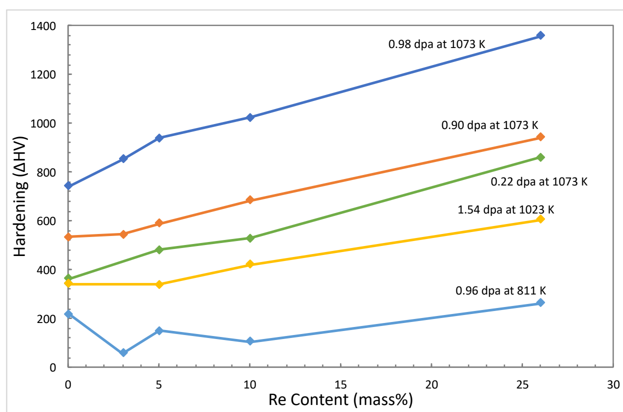

Fig. 5. Increase in hardness for W-Re alloys based on Re mass content.

III. CONCLUSIONS

Radiation hardening will occur in refractory metals such as Mo and W even at very low neutron doses. Maximum hardening will occur in Mo up to irradiation temperatures of ~870 K (~0.3 T_M). Irradiated Mo will begin to recover unirradiated mechanical properties at

1070 K and will experience full annealing at around 1300 K. Hardening saturation appears to occur around 0.1 dpa, although this value varies based on irradiation temperature. The DBTT will increase, but a lack of studies at relevant doses do not allow for a justified approximation. The parts of the fuel element operating below the DBTT will have an increased risk of brittle fracture.

Radiation hardening trends are very similar in W. Radiation hardening occurs up to at least 1100 K (0.3 T_M), a higher irradiation temperature than Mo due to the higher thermal stability of radiation defects in W (which can be approximately correlated with melting temperature). There is a lack of irradiation data for W above 1100 K, so it is difficult to determine the precise upper temperature limit for radiation hardening in W. Radiation hardening saturation appears to occur between 0.1 and 0.4 dpa for W. DBTTs collected from tensile and bend tests suggest a DBTT for W irradiated to NTP relevant doses of 550 K. The expected shift in DBTT for W at a dpa of around 0.0055 was not determined in this review. Annealing in W begins to occur around 1000K.

Radiation hardening has a more drastic effect on alloys of refractory metals than the pure refractory metals. Neutron irradiation causes precipitation of the alloying elements which increases the hardening even at low doses. For Mo-Re and W-Re alloys, hardening increases with Re content (particularly at higher doses). TZM behaved similarly to Mo-Re in that the increase in radiation hardening was more drastic than in pure Mo.

Most of the studies conducted for Mo and W were performed at doses higher than the anticipated lifetime values of 0.015 and 5.5 x 10^{-3} dpa, respectively, for NTP crewed missions to Mars. In order to determine operating parameters for cermet based NTP systems, future studies are needed. Pure Mo and W along with cermets with surrogate fuel particles will be ion irradiated at higher temperatures to determine the cutoff where the effects of irradiation hardening are counterbalanced by thermal annealing. These studies will also compare the irradiation hardening of cermets to the hardening observed in the pure metals. Additionally, the ion irradiated samples will be tested with hot flowing hydrogen and compared to unirradiated samples tested in the same environment. These experiments will serve to fill the knowledge gap determined by the literature review and confirm the viability of a cermet fuel element in an NTP system.

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