Effects of Electron Irradiation on Candidate Materials for Target Window in Accelerator- Driven Molybdenum-99 Production

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Experimental Operations and Facilities Division

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EFFECTS OF ELECTRON IRRADIATION ON CANDIDATE MATERIALS FOR TARGET WINDOW IN ACCELERATOR-DRIVEN MO-99 PRODUCTION

1 BACKGROUND

NorthStar Medical technologies is planning to produce Mo-99 through the photo-nuclear reaction $\text{Mo-100}(\gamma, n)\text{Mo-99}$ utilizing multiple high-power electron accelerators. In this approach, the production target consists of multiple thin disks of metal Mo-100 cooled by helium gas at high (300–400 psi) pressure [4]. The cost of enriched Mo-100 is on the order of \$500–\$1000 per gram, which makes a smaller target desirable and requires achieving the highest possible electron flux on the target. The heat deposition in the entrance window limits the maximum flux because the window separates the vacuum in the accelerator beam line from the high-pressure helium in the target and is only cooled on one side by the helium flow. Because the window is continuously under stress from the helium pressure and the heat from the electron beam, it is desirable to estimate a lifetime for the window and develop a replacement schedule for the target holder. Such an estimate requires the ability to predict changes in the window material properties under electron irradiation.

Candidate materials for the Mo-99 production target window are Inconel 718, 250 maraging steel, and beryllium. These materials encompass a wide range of thermal conductivity, thermal expansion coefficients, Young's modulus, tensile strength and tensile ductility. The Mo99 target window is subjected to internal pressure from the helium coolant and thermal stresses. The operating conditions and relative advantages of each material have been described previously [1]. The current work investigates the effects of electron irradiation on the tensile properties of these materials. Axial tensile tests were conducted with electron-irradiated samples and results were compared to baseline results generated for as-fabricated materials.

2 ELECTRON IRRADIATION

Electron irradiation of window material samples was conducted with a 55 MeV electron linear accelerator at the Argonne Low Energy Accelerator Facility [5]. Figure 1 shows drawings of the irradiation set up. Samples of the three window material candidates were irradiated at the same time: Inconel 718, maraging steel, and beryllium were placed one after another, with a 1 mm gap between for water cooling. The main components of the irradiation apparatus depicted in Figure 1 are:

- 1, 2, and 3 – Inconel 718, maraging steel 250, and beryllium samples respectively
- 4 – aluminum pins for sample alignment
- 5 – 1 mm thick aluminum spacers
- 6 – large aluminum spacer
- 7 – front cover plate to hold thermocouples
- 8 – graphite gasket
- 9 – coolant tubing

Electron irradiation was conducted at 40 MeV electron beam energy and at ~2.7 kW beam power. Beam spot at the entrance to the irradiation setup was ~5 mm full width at half maximum (FWHM). Table 1 summarizes the irradiation parameters for the samples.

The beam position was monitored during irradiation using beam position monitors and four thermocouples, located equal distances from the edge of the window (left, right, up, and down), to monitor temperatures. After irradiation, samples were removed from the housing and stored to allow radioactive decay. After two months of storage, samples were submitted to testing.

FIGURE 1 Irradiation Setup Design

TABLE 1 Irradiation Parameters

Samples irradiated	Beam energy, MeV	Average beam power, kW	Time of irradiation, hours	Beam size on the target window, FWHM, mm
300H 301H 302H	40	2.65	59.25	5.1x5.2
300J 301J 302G	40	2.7	52.15	5.1x5.2

3 MATERIALS, SAMPLE DESIGN, AND TEST METHODS

Solution-annealed (SA) Inconel 718 was purchased from Special Metals and axial-test samples were fabricated by Argonne Central Shops (CS). Maraging steel (250) was purchased from Carpenter Technology Corporation and also fabricated by Argonne CS. Beryllium (I-220-H) was purchased from Materion, who also fabricated the tensile samples and provided dimensional measurements.

Figure 2 shows the axial-tensile test sample design for Inconel 718 and 250 maraging steel. Table 2 gives nominal dimensions for gauge length (L_{gi}), gauge width (W_{gi}), gauge thickness (H_{gi}), and total sample length (L_i). The subscript “g” indicates gauge and the subscript “i” indicates initial pre-test values. Figure 3 shows the sample design for Be, which differs only in the increased thickness as indicated in Table 2. All dimensions in the figures are in inches. Table 2 includes dimensions in millimeters, as well as inches. The holes near the top and bottom of each sample were drilled to allow pin loading of the sample. Relative to ASTM axial tensile samples (2-inch [50.8-mm] long gauge), these subsized samples were 0.3-inch long (7.62 mm) gauge. Pins were fabricated from high-strength 17-4PH Stainless Steel, also referred to as tool steel. This subsized sample design (SS3) has been successfully used to study the effects of neutron irradiation and environment on the axial tensile properties of stainless steels and high-nickel alloys for use as nuclear-reactor materials [2]. The SS3 tensile sample has also been used to compare tensile properties between commercial heats and laboratory heats of nuclear materials [3].

Axial tensile properties of interest are the yield stress (YS, 0.2% offset), the ultimate tensile stress (UTS), the uniform elongation (UE), the total elongation (TE), and the local reduction in area (RA). From a design perspective, YS, UTS, and UE are the more important parameters. The values for YS, UTS, UE, and TE represent engineering values because they are derived using the as-fabricated dimensions. These values differ from true stress-strain values, which accounts for the decrease in cross-sectional area and the increase in gauge length with increased axial deformation.

Room-temperature (RT) axial tensile tests were conducted in the displacement-controlled mode with the in-glove box Instron 8511, a servo-hydraulic machine. The data acquisition system was set at 10 Hz (1 data point per 0.1 s) and the reference displacement rate was 0.05 mm/s, which is equivalent to a gauge strain rate of 0.0066 mm/mm/s. The measured load was normalized to the as-fabricated cross-sectional area to determine stress. The controlled displacement was normalized to the as-fabricated gauge length to determine strain.

FIGURE 2 Sample Design and Dimensions for Inconel 718 and 250 Maraging Steel

FIGURE 3 Sample Design and Dimensions for Be

TABLE 2 Summary of Sample Dimensions and Vendor-reported Minimum Tensile Properties for Inconel 718 (IN 718), 250 Maraging Steel (MS) and I-220-H Beryllium (Be)

Parameter	IN 718	MS	Be
Gauge Length, in. (mm)	0.300 (7.62)	0.300 (7.62)	0.300 (7.62)
Gauge Width, in. (mm)	0.060 (1.542)	0.060 (1.542)	0.060 (1.542)
Gauge Thickness, in. (mm)	0.020 (0.508)	0.020 (0.508)	0.060 (1.542)
Total Length, in. (mm)	1.000 (25.40)	1.000 (25.40)	1.000 (25.40)
Yield Stress, ksi (MPa)	61.5 (424)	252 (1738)	50 (345)
Ultimate Tensile Stress, ksi (MPa)	130.5 (900)	257 (1772)	65 (448)
Uniform Elongation, %	---	---	---
Total Elongation, %	51.8	9.0	2.0
Reduction in Area, %	---	63	---

4 BASELINE TENSILE TEST RESULTS FOR AS-FABRICATED SAMPLES

Stress-strain curves are shown in Appendix A for SA Inconel 718, Appendix B for 250 maraging steel, and Appendix C for I-220-H Be.

4.1 Inconel 718

Four baseline tests were conducted with Inconel 718: three at 0.05 mm/s and one at 0.005 mm/s. At the slower displacement rate, the number of data points generated was increased by a factor of 10 to determine the loading slope and the 0.2%-offset YS. However, no significant differences existed between the results from tests at the slower displacement rate and the results at 0.05 mm/s; the latter was chosen as the reference displacement rate.

The first test was terminated after necking and just prior to failure. Post-test measurements included the final sample length (L_f), the final gauge width (W_{gf}) at three axial locations, and the final gauge thickness (H_{gf}) at three axial locations. The change in length ($\Delta L = L_f - L_i$) was normalized to the initial gauge length (L_{gi}) to give an upper-bound estimate of TE (60%), which was in good agreement with TE (62%) as determined from the stress-strain curve. As expected, the change in length was higher than the vendor-quoted minimum of about 52%. The reduction in area at the center of the gauge section, where the minimum post-test cross-sectional area was located, was about 40%. Both measurements supported the vendor-reported high ductility value for TE. Based on stress-strain curves, ductility results of the four tests were $54 \pm 1\%$ for UE and $64 \pm 1\%$ for TE.

The YS values at 0.2% offset strain were in the range of 488 ± 6 MPa, which was relatively consistent and about 15% higher than the vendor-reported minimum YS value (see

Table 2). The UTS was within the range of 901 ± 9 , which was in excellent agreement with the vendor-reported minimum value listed in Table 2.

4.2 250 Maraging Steel

The first maraging steel test was conducted at 0.005 mm/s. However, additional testing was paused because the Instron 8511 exhibited drift and oscillation in the preset displacement due to problems with the hydraulic system. The performance of the machine improved after replacing oil filters, and better performance was observed at the faster 0.05 mm/s displacement rate as compared to the slower 0.005 mm/s displacement rate. Two additional tests were conducted at the reference 0.05 mm displacement rate.

The 0.005 mm/s test was terminated prior to failure and before the load drop associated with localized necking. Post-test examination of the sample indicated no significant change in sample thickness, but localized necking was observed in the sample width: 0.8% decrease near the bottom of the gauge, 1.6% decrease near the gauge center, and 2.5% decrease near the top of the gauge. TE, determined from the measured ΔL normalized to the initial gauge length, was 7.3%, in reasonable agreement with the 5.4% determined from the stress-strain curve and the 9% value reported by the vendor, especially considering that the test was interrupted prior to failure. For the two tests conducted to failure, the average TE was 9%, which agreed with the vendor reported value. Both YS (846 MPa) and UTS (935 MPa) for the interrupted test were considerably lower than the vendor reported values in Table 2. For the three tests conducted, YS was in the range of 882 ± 31 MPa and UTS was in the range of 990 ± 49 MPa. It is not clear if the material tested was actually 250 maraging steel or if the sample design resulted in premature necking near the gauge-fillet transition. Based on previous experience with the SS3 tensile sample [2, 3], it is more likely that the material tested differed in the heat treatment commonly specified for 250 maraging steel.

4.3 I-220-H Be

Beryllium has relatively high values for thermal conductivity and Young's modulus. However, it is a low-ductility, anisotropic material. Isostatic hot-pressed Be tends to behave in a more isotropic manner than rolled Be. The "H" indicates that the Be tested was hot-pressed. Prior to conducting the tensile tests, there was concern that Be samples would fail in the loading and/or support hole locations due to the stress-strain concentrations at these positions. The two baseline samples tested behaved better than expected in that both samples failed near the gauge center. The YS values were determined to be 489 ± 1 MPa and the UTS values were in the range of 546 ± 5 MPa. These strength values are higher than the vendor-reported minimum values in Table 2: 42% higher for YS and 22% higher for UTS. The TE values determined from the stress-strain curves were $2.9 \pm 0.5\%$, which is consistent with the 2% minimum value reported in Table 2. As the vendor (Materion) reported only minimum values with no range, it is not clear if the measured values fall within the range of vendor-measured values for I-220-H Be.

5 TENSILE TEST RESULTS FOR ELECTRON-IRRADIATED SAMPLES

The general trend for neutron-irradiated metal alloys is an increase in strength properties (i.e., YS and UTS) and a decrease in ductility (i.e., UE and TE). The percentage increase in YS is generally higher than the percentage increase in UTS. Tests were conducted with electron-irradiated materials to determine if such hardening occurred in these materials.

5.1 Electron-Irradiated SA Inconel 718

Figures 4 and 5 show the stress-strain curves for electron-irradiated SA Inconel 718. Both samples failed in the fillet region, indicating that the UTS and TE determined from these tests represent lower bounds relative to samples that would have failed in the gauge section. The YS values were in the range of 662 ± 76 indicating a $36 \pm 16\%$ increase in YS relative to the as-fabricated material. The UTS values were in the range of 957 ± 19 MPa indicating a $6 \pm 2\%$ increase in UTS relative to the as-fabricated material. Concurrently, UE decreased from about 54% to 34% and TE decreased from about 64% to 38%. Although it is clear that electron irradiation resulted in hardening, the material retained relatively high ductility.

FIGURE 4 Stress-Strain Results for Electron-Irradiated SA Inconel 718 Sample 300H.

FIGURE 5 Stress-Strain Results for Electron-Irradiated SA Inconel 718 Sample 300J.

5.2 Electron-Irradiated 250 Maraging Steel

Figures 6 and 7 show the stress-strain results for electron-irradiated 250 maraging steel. Both samples failed in the fillet region indicating that the UTS and TE values determined from these tests represent lower bounds on these parameters. The YS values were in the range of $1021 \pm 13 \text{ MPa}$ indicating a $16 \pm 1\%$ increase in YS relative to the as-fabricated material. The UTS values were in the range of $1076 \pm 26 \text{ MPa}$ indicating a $9 \pm 3\%$ increase in UTS relative to the as-fabricated material. Concurrently, UE decreased from about 2% to 0.6% and TE decreased from about 9% to about 6%. Electron irradiation appears to have resulted in hardening of this material.

FIGURE 6 Stress-Strain Results for Electron-Irradiated 250 Maraging Steel Sample 301H.

FIGURE 7 Stress-Strain Results for Electron-Irradiated 250 Maraging Steel sample 301J.

5.3 Electron-Irradiated I-220-H Be

The first test (sample 302G) conducted with electron-irradiated I-220-H Be failed in the bottom hole region at a relatively low load and gauge stress. Figure 8 shows the limited stress-strain curve and a photograph of the failed sample. No useful stress-strain information can be determined from these results. The second test (sample 302H) failed in the gauge section and did yield important tensile results. Figure 9 shows the stress-strain results and a post-test photograph of the failed sample. The YS was 403 MPa, which is 17% lower than the YS for the as-fabricated material. The UTS was 536 MPa, which was only 2% lower than the UTS for the as-fabricated material and likely within expected scatter for non-irradiated Be. Also, TE was 2.1%, which is within the range of expected scatter for non-irradiated Be and close to the vendor-reported minimum value of 2%. Although more data are needed to determine the effects of electron irradiation on the mechanical properties of I-220-H Be, these preliminary results suggest a decrease in YS without any significant increase in TE, as would be expected if electron irradiation resulted in a “softening” of this material.

FIGURE 8 Limited Stress-Strain Results for Electron-Irradiated I-220-H Be Sample 302G, which Failed in the Region of the Bottom Support Hole.

FIGURE 9 Stress-Strain Results for Electron-Irradiated I-220-H Be Sample 302H, which Failed in the Gauge Section

6 DISCUSSION

Baseline tensile test results agreed reasonably well with vendor-reported minimum values for YS, UTS, and TE values for SA Inconel 718 and I-220-H Be. However, YS and UTS values were too low by about a factor of two for vendor-reported minimum values of 250 maraging steel. The Rockwell Hardness of this material should be measured and compared to vendor reported values (50 ± 2 HRC). On the basis of the tensile results generated in the current work, the hardness of the material tested is likely to be much less than 50 HRC. This would indicate that the material tested may not have received the full specified heat treatment to qualify as 250 maraging steel.

Electron irradiation increased the strength (YS and UTS) and decreased the ductility (UE and TE) of the SA Inconel 718 and the maraging steel tested. The electron-induced hardening had a more significant effect on the YS as compared to the UTS. This result is consistent with the effects of neutron-induced irradiation hardening. An interesting and productive experiment would be to subject tensile samples to higher levels of electron irradiation to help evaluate design stresses and lifetime. Guidelines for design stresses change as UE drops below about 4%. For primary loading such as pressure loading, the allowable stress is two-thirds the minimum YS for high ductility materials. For materials with $UE < 4\%$, the allowable stress is the minimum of two-

thirds of the minimum YS and one-fourth the minimum UTS. Studying the effects of annealing on YS, UTS, and UE would also be interesting and relevant.

The effects of electron irradiation on I-220-H Be tensile properties are unclear. Based on one successful test, there is some indication that softening (i.e., decrease in YS and UTS) may have occurred without any noticeable change in TE. Additional tests need to be performed at the current electron exposure level as well as higher exposure levels. Such test results would be needed if Be continued to qualify as a serious candidate for the Mo-99 target window.

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APPENDIX A:
AXIAL TENSILE TEST RESULTS FOR AS-FABRICATED SA INCONEL 718

FIGURE A-1 Stress-Strain Results for Baseline As-fabricated SA Inconel 718 Sample 300A Tested at RT and 0.05 mm/s. The test was terminated just prior to sample failure.

FIGURE A-2 Stress-Strain Results for Baseline As-fabricated SA Inconel 718 Sample 300B Tested at RT and 0.05 mm/s. The sample failed in the gauge section of the sample.

FIGURE A-3 Stress-Strain Results for Baseline As-fabricated SA Inconel 718 Sample 300C Tested at RT and 0.005 mm/s. The sample failed in the gauge section.

FIGURE A-4 Stress-Strain Results for Baseline As-fabricated SA Inconel 718 Sample 300D Tested at RT and 0.05 mm/s. The sample failed in the gauge section.

APPENDIX B:
AXIAL TENSILE TEST RESULTS FOR AS-FABRICATED 250 MARAGING STEEL

FIGURE B-1 Stress-Strain Results for Baseline As-fabricated 250 Maraging Steel Sample 301G Tested at RT and 0.005 mm/s. The test was terminated prior to sample failure.

FIGURE B-2 Stress-Strain Results for Baseline As-fabricated 250 Maraging Steel Sample 301F Tested at RT and 0.005 mm/s. The sample failed in the gauge section.

FIGURE B-3 Stress-Strain Results for Baseline As-fabricated 250 Maraging Steel Sample 301E Tested at RT and 0.05 mm/s. The sample failed in the gauge section.

APPENDIX C:
AXIAL TENSILE TEST RESULTS FOR AS-FABRICATED I-220-H BERYLLIUM

FIGURE C-1 Stress-Strain Results for Baseline As-fabricated I-220-H Be Sample 302A Tested at RT and 0.05 mm/s. The sample failed in the gauge section.

FIGURE C-2 Stress-Strain Results for Baseline As-fabricated I-220-H Be Sample 302F Tested at RT and 0.05 mm/s. As indicated in the photograph, the sample failed in the gauge section.

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