EVALUATION OF MATERIALS USED FOR APT IRRADIATION EXPERIMENT*

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Introduction

Investigators at the Los Alamos National Laboratory (LANL) conducted an irradiation experiment at the LAMPF facility to determine the effect of irradiation on several materials that could potentially be used as structural or window materials for the Accelerator for Production of Tritium (APT) facility that is being designed. Tensile and fracture toughness specimens were irradiated. In this report, the results of chemical analysis, hardness, metallography, and Automated Ball Indentation (ABI) measurements on these materials (unirradiated) are reported.

Materials and Specimens

The following materials were in the irradiation experiment for the APT Program: 316L stainless steel, 304L stainless steel, Inconel 718, 6061-T6 Al, 5052-0 Al, and Modified 9Cr-1Mo steel. Mechanical property samples included in the irradiation were: miniature disk-compact tension (DCT) specimens with thicknesses of 2 and 4 mm and sheet tensile specimens either 0.25- or 0.76-mm thick. The 304L stainless steel, Inconel 718, and 5052 Al tensile specimens were 0.76-mm thick, and the the rest were 0.25-mm thick.

Specimens were machined by Metal Samples Inc., Munford, Alabama. With the exception of the Modified 9Cr-1Mo steel, the material from which the the specimens were machined was obtained by Metals Samples Inc., who provided certified mill test reports for the materials they supplied. The Modified 9Cr-1Mo was obtained from ORNL in the form of 2-mm and 4-mm plate and 0.25-mm sheet.

The DCT specimens (2 and 4 mm) of the materials were to be similarly oriented, and in addition, two orientations of the modified 9Cr-1Mo steel DCT specimens were to be tested. Because questions arose concerning the orientation (relative to the rolling direction) of the materials and whether the same material (same plate) was used to make the various specimens for a given material, chemical analyses, metallography, hardness tests, and ABI tests were conducted on the unirradiated materials.

Chemical Composition

Because there was a question about whether the same heats of material were used to make the DCT specimens and the tensile specimens, it was decided to obtain chemical analyses of the material. The analyses were conducted by ABB C-E Power Products Manufacturing, Chattanooga, Tennessee. Unfortunately, there was a limited amount of material available for analysis. One-half of one of the 2-mm and 4-mm DCT specimens were used for the X-ray Fluorescence (XRF) technique. To obtain a large enough area for the 10-mm spot size for the XRF, each specimen was pressed into a foil. Because of the limited size, carbon and sulphur were not obtained from these specimens. With the exception of the 316L stainless steel, only small tensile specimens were available (not enough material for XRF), so the only sheet material for which an analysis was obtained was for a piece of the 0.76-mm 316L stainless steel. The chemical compositions are given in Table 1 for the 316L and 304L stainless steels; Table 2 contains the data for Inconel 718, 5052-Al, 6061-Al, and modified 9Cr-1Mo steel. Where vendor-supplied specifications were available, the chemical analyses obtained here were similar to supplied by the vendor.

Table 1. Chemical Composition of Type 316L and 304L Stainless Steels

Element	316L SS (2 mm)	316L SS (4 mm)	316 SS (0.76 mm)	304L SS (2 mm)	304L SS (4 mm)
Mn	1.67	1.67	1.83	1.85	1.87
P	0.025	0.026	0.024	0.025	0.026
Si	0.62	0.58	0.45	0.43	0.52
Ni	10.83	10.82	10.11	8.37	8.45
Cr	17.47	17.54	16.15	18.16	18.26
Mo	2.08	2.07	2.06	0.17	0.18
V	0.08	0.07	0.05	0.08	0.09
Nb	<0.01	<0.01	0.03	<0.01	<0.01
Ti	0.02	0.02	0.01	0.01	<0.01
Co	0.21	0.21	0.07	0.16	0.16
C			0.026		
S			<0.001		
Cu	0.21	0.21	0.07	0.22	0.23
Fe	Balance	Balance	Balance	Balance	Balance

The results indicate that the same heat (plate) of material was used for the 2- and 4-mm DCT specimens. For the only sheet material that was analyzedóthe 0.76-mm 316L stainless steel sheet from which tensile specimens were machinedóit appears that there is a slight difference in chemistry for a few elements, in particular, the manganese, chromium, and nickel. This different heat of the steel is still within specifications for type 316L stainless steel.

Table 2. Chemical Composition of Inconel 718, Aluminum Alloys, and Modified 9Cr-1Mo Steel

Element	Inconel 718		5052 Aluminum		6061 Aluminum		Modified 9Cr-1Mo	
	2 mm	4mm	2 mm	4 mm	2 mm	4 mm	2 mm	4 mm
Mn	0.11	0.11	0.01	0.01	0.07	0.07	0.45	0.45
P							0.018	0.019
Si	0.14	0.14	0.32	0.34	0.66	0.68	0.30	0.28
Ni	54.4	54.5	<0.01	<0.01	<0.01	<0.01	0.16	0.18
Cr	18.03	18.04	0.22	0.21	0.20	0.20	9.25	9.28
V			0.02	0.02	0.01	0.01	0.25	0.24
Mo	2.89	2.89					0.99	0.99
Nb	5.03	5.00					0.06	0.06
Ti	0.98	0.97	0.01	0.01	0.02	0.02	0.07	0.07
Co	0.20	0.20						
Cu	0.17	0.17						
Al	0.43	0.43	Balance	Balance	Balance	Balance		
Mg			2.56	2.68	1.03	1.06		
Zn			<0.01	<0.01	0.04	0.04		
Fe	17.57	17.54	0.13	0.14	0.45	0.46	Balance	Balance

Hardness

Rockwell A hardness values (the materials were too soft to use Rockwell B or C) for the surface of the 2-mm and 4-mm plate material are given in Table 3 for all but the two aluminum alloys, which were too soft to measure by this technique. No Rockwell hardness measurements were made on the sheet tensile specimens because of the limited thickness. Two measurements were made on each of the specimen surfaces, and the average is given in the table. Hardnesses of the 2- and 4-mm plate specimens appeared similar in all cases.

After the specimens were mounted for metallography, three microhardness measurements were made on the polished cross sections of each type of specimen; average DPH hardness values are given in Table 3. The microhardness values of the 2- and 4-mm specimens were generally the same for all materials. The important observation from the microhardness measurements is that for the 6061 aluminum there appears to be a definite hardness difference between the plate used for the 2- and 4-mm DCT specimens and the sheet used for the tensile specimens. There also appears to be a difference between the 2- and 4-mm plate and the sheet specimen for the 304L stainless steel, although the difference is not as great as it was for the 6061 aluminum. Microhardness values for the other materials appear to be the same for the plate and sheet material.

Table 3 Rockwell and DPH Hardness Measurements of Test Materials

Material	Specimen Thickness	Rockwell A	Microhardness, DPH
316L Stainless Steel	2 mm	46.5	157
	4 mm	45.5	159
	0.25 mm		157
304L Stainless Steel	2 mm	47.8	175
	4 mm	48	171
	0.25 mm		154
Inconel 718	2 mm	71.5	473
	4 mm	71.8	486
	0.25 mm		491
Al-5052	2 mm		70
	4 mm		65
	0.76 mm		57
Al-6061	2 mm		121
	4 mm		121
	0.25 mm		57
Modified 9Cr-1Mo[a]	2 mm (O1)	62	295
	4 mm (O1)	61.8	293
	2 mm (O2)	62.3	293
	4 mm (O2)	59	
	0.25 mm		291

[a] Two orientations (O1 and O2) of the specimens were examined.

Metallography

One-half of a broken 2- and 4-mm disk compact tension specimen from each material was sectioned and mounted for metallography to reveal the microstructure of two of the surfaces (relative to the rolling direction). Two views of each of the sheet tensile specimens were also examined. The materials were examined for microstructure and to determine the orientation of the specimens relative to the rolling direction. An estimation of grain size was also obtained. The results on the orientation and grain size are given in Table 4. Results for the individual materials will be discussed, and for each a low- and high-magnification (100 and 500X) optical photomicrograph of the microstructure of the 2-mm plate from each material will be presented. These microstructures are representative of material of each specimen geometry.

Type 316L Stainless Steel

The microstructures of the 2- and 4-mm DCT fracture toughness specimens and the sheet tensile specimens were typical for this type of annealed austenitic stainless steel (Fig. 1). They contained a small amount of ferrite, but this is usual for this grade of stainless steel. The rolling directions were determined by examining the shape of the grains and the elongated ferrite and inclusion particles. It was concluded that the crack-growth direction of the 2- and 4-mm-thick specimens was parallel to the rolling direction. The overall microstructures of the 2- and 4-mm specimens was similar.

There was a difference in the microstructure of the tensile specimens and the disk compact tension specimens, the major difference being the smaller grain size of the 0.25-mm sheet used for the tensile specimens. Tensile specimens were taken with the axis parallel to the rolling direction, which means the crack growth during the tensile test is perpendicular to crack growth in the fracture toughness tests, but this should not be of much consequence for this tough material.

Type 304L Stainless Steel

The 304L stainless steel was typical of an annealed austenitic stainless steel with a cleaner microstructure than that of the type 316L (Fig. 2). Nevertheless, it was possible to determine that the crack-growth direction of the 2- and 4-mm-thick specimens was parallel to the rolling direction. As was the case with the Type 316 SS, the microstructure of the 0.25-mm sheet tensile specimen was different than that of the 2- and 4-mm thick disk compact tension specimens, as differentiated by the smaller grain size of the tensile specimens. The rolling direction on the tensile specimen was very difficult to discern, but it was tentatively concluded that the rolling direction is parallel to the tensile axis.

Inconel 718

The microstructure of the Inconel 718 under light microscopy is characterized primarily by inclusions, which were used to determine that the rolling direction and the crack growth direction were parallel in the 2- and 4-mm specimens (Fig. 3). For the tensile specimen, there was again a much finer grain size than for the DCT specimens. The specimen axis of the tensile specimens was parallel to the rolling direction.

Table 4 Grain Sizes of the Test Material Orientation

Material	Specimen Thickness	Orientation	Grain Size, µm (ASTM No.)
316L Stainless Steel	2 mm	CG parallel to RD	32 (7)
	4 mm	CG parallel to RD	32 (7)
	0.25 mm	CG 90∞ to RD	16 (9)
304L Stainless Steel	2 mm	CG parallel to RD	65 (5)
	4 mm	CG parallel to RD	55 (5.5)
	0.25 mm	CG 90∞ to RD	22 (8)
Inconel 718	2 mm	CG parallel to RD	55 (5.5)

	4 mm	CG parallel to RD	65 (5)
	0.25 mm	CG 90∞ to RD	45 (6)
Al-5052	2 mm	CG 90∞ to RD	32 (7)
	4 mm	CG 90∞ to RD	27 (7.5)
	0. 76 mm	CG 90∞ to RD	45 (6)
Al-6061	2 mm	CG 90∞ to RD	65 (5)
	4 mm	CG parallel to RD	90 (4)
	0.25 mm	CG 90∞ to RD	45 (6)
Modified 9Cr-1Mo[a]	2 mm (O1)	CG parallel to RD	22 (8)
	4 mm (O1)	CG 90∞ to RD	27 (7.5)
	2 mm (O2)	CG parallel to RD	27 (7.5)
	4 mm (O2)		
	0.25 mm	CG 90∞ to RD	19 (8.5)

[a] Two orientations (O1 and O2) of the specimens were examined.

<u>Aluminum-6061</u>

The microstructure of the 6061-Al is shown in Fig. 4. There were no obvious differences in the 2- and 4-mm specimens. For both specimens, the crack growth direction was perpendicular to the rolling direction. The tensile specimen again had a smaller grain size than did the DCT specimens. On this specimen, it was again difficult to discern the rolling direction, but was tentatively concluded to be parallel to the rolling direction (emphasis on tentative).

<u>Aluminum-5052</u>

Figure 5 shows the microstructure of the 5052-Al. There were no obvious differences between the 2- and 4-mm specimens. For the 4-mm specimen, the crack growth direction was perpendicular to the rolling direction. It was much more difficult to determine this for the 2-mm specimen; it was tentatively concluded to have the same orientation as the 4-mm specimen. For the tensile specimen, it was again difficult to detect the rolling direction, but it was very tentatively concluded that the crack growth was perpendicular to the rolling direction.

<u>Modified 9Cr-1Mo</u>

The microstructure of the modified 9Cr-1Mo steel (Fig. 6) was typical of a tempered martensite microstructure that is developed by the normalizing-and-tempering heat treatment of this steel. Because the microstructure is quite clean, it was difficult to discern the rolling direction. Tentatively, it was concluded that the 4-mm specimen had the crack growth direction perpendicular to the rolling direction. Specimens from the 2-mm plate were to be made in both orientationsóparallel and perpendicular. It appears, however, that both of the 2-mm specimens had the same orientation with the crack growth direction parallel to the rolling direction. The rolling direction for the tensile specimen is parallel to the specimen axis. The microstructures of the 2- and 4-mm compact tension specimens and the tensile specimens appear similar. The sheet material again had a smaller grain size than the plate material.

Automated Ball Indentation Tests

The Automated Ball Indentation (ABI) test is a test technique designed to obtain tensile properties from indentation (hardness-type) measurements; hardness as a function penetration depth is determined and the data are analyzed to estimate the yield stress (YS), ultimate tensile strength (UTS), and strain-hardening exponent. To determine the properties of the 2- and 4-mm plate material, ABI tests were made at room temperature with a 0.030-in diameter tungsten carbide indenter on the surface of untested DCT specimens. To test the sheet material, untested tensile specimens were obtained from LANL. A 0.010-diameter indenter was used on 0.25-mm sheet-tensile specimens, and a 0.030-in indenter was used on 0.76-mm sheet-tensile specimens. Estimates of YS, UTS, and strain-hardening coefficient are given in Table 5 for each material.

It can be observed in Table 5 that the YS and UTS of the 316 stainless steel was essentially the same for the two plate materials (2- and 4-mm) and the sheet material. For the 304L stainless steel, the YS for the two plate materials were similar, with a slightly higher value for the sheet. However, the UTS of the plate materials were greater than for the sheet material. This observation on UTS (Table 5) agrees with observations on the microhardness for this steel (Table 3). Hardness is known to correlate better with the UTS than the YS, so the ABI observations are consistent with the observations on hardness. The Inconel 718 2- and 4-mm plate specimens had a slightly lower YS and a slightly higher UTS than the sheet. There was less variation for the hardness measurements (Table 3).

Table 5. Estimated Tensile Properties Obtained From ABI Tests

Material	Thickness	YS MPa	Strain Hard Exponent	UTS MPa
316L Stainless Steel	2 mm	282	0.192	573
	4 mm	276	0.189	551
	0.25 mm	288	0.185	550
304L Stainless Steel	2 mm	278	0.187	538
	4 mm	272	0.185	551
	0.76 mm	301	0.131	451
Inconel 718	2 mm	823	0.143	1356
	4 mm	861	0.137	1368
	0.76 mm	963	0.097	1282
Al-5052	2 mm	113	0.151	187
	4 mm	114	0.145	184
	0.51 mm	128	0.157	218
Al-6061	2 mm	255	0.053	294

	4 mm	265	0.062	318
	0.76 mm	296	0.031	310
Al-6061-T6	0.25 mm	174	0.084	219
Modified 9Cr-1Mo[a]	2 mm (O1)	493	0.112	702
	4 mm (O1)	499	0.106	688
	2 mm (O2)	492	0.110	690
	4 mm (O2)	446	0.105	613
	0.25 mm	476	0.130	717

[a] Two orientations (O1 and O2) of the specimens were examined.

The YS and UTS for the Al-5052 plate and sheet material were similar, which is consistent with the hardness measurements. When the tensile specimens of Al-6061 were obtained from LANL, two types of 6061-Al specimens were present, one labeled Al-6061 and the other labeled Al-6061-T6. Both of these specimens were tested. Table 3 indicates that for the Al-6061 material, there was little difference in the YS and UTS of the plate and sheet material. On the other hand, for the estimated YS and UTS of the Al-6061-T6 specimen, the strength of the sheet tensile specimen was considerably less than for the plate specimens. This latter result coincides with the hardness observations in Table 3. The T6 aluminum is aged to increase the strength. This information may indicate that the specimen labeled T6 was mixed up with the unlabeled one. This should be investigated. The results indicate that the DCT specimens and the tensile specimen labeled 6061-Al are similar.

Finally, the YS and UTS for the modified 9Cr-1Mo steel specimens showed relatively little variation between the plate and sheet material, which agrees with the hardness measurements.

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