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Effects of Long Term Thermal Exposure on Chemically Pure (CP) Titanium Grade 2 Room Temperature Tensile Properties and Microstructure

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August 2007

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Abstract

Room temperature tensile testing of Chemically Pure (CP) Titanium Grade 2 was conducted for as-received commercially produced sheet and following thermal exposure at 550 and 650 K for times up to 5,000 h. No significant changes in microstructure or failure mechanism were observed. A statistical analysis of the data was performed. Small statistical differences were found, but all properties were well above minimum values for CP Ti Grade 2 as defined by ASTM standards and likely would fall within normal variation of the material.

Introduction

Many portions of the thermal management systems for spacecraft including ducting, recuperators, pumped loops and heat pipes can benefit from the relatively low density and high strength to weight ratio of titanium. These missions can last for years or even decades with the thermal management systems operating at close to peak temperature nearly continuously. Depending on the system, the stresses from internal pressure can range from a few Pascals to nearly 10 MPa. Additional external stresses may be added if the structures are utilized as load bearing members as well. These conditions require knowledge of the mechanical properties of titanium following long term thermal exposures at moderate temperatures (400 to 650 K). However, there is no comprehensive literature data available for the effects of long term thermal exposure on the tensile properties of chemically pure titanium (CP Ti). Therefore this testing was undertaken to determine the effects of thermal exposure alone on room temperature, 550 and 650 K tensile properties.

As shown in Figure 1, Ti could be used in many portions of the power conversion system but mainly would be used for the heat transfer and heat rejection systems. In addition to its low density, its chemical compatibility is a key feature of CP Ti. It is anticipated that CP Ti will not corrode or undergo minimal corrosion when in contact with ultrahigh purity He, NaK or triple distilled H_2O (ref. 1). All three fluids are potential candidates for heat transfer in various portions of the power thermal management systems under consideration by NASA's Prometheus Program depending on the rejection temperature of the energy conversion system and the radiator operating temperature. For Project Prometheus, the current maximum anticipated operating temperature is 550 K (531 °F). There is a possibility that some of the components could be pushed to higher temperatures as the designs evolve or in an over temperature excursion by the system, so properties after exposure to 650 K (711 °F) were also investigated in this study.

Minimum room temperature tensile properties for CP Ti are shown in Table 1. Grade 1 is preferred for power conversion systems due to its lower O content and availability of a low Fe (0.05 wt.%) version. It is felt that lower contamination of the working fluids will occur with the purer Ti. Grade 2 has some large strength advantages with minimal additional trace element levels. It was also observed from examination of available CP Ti Grade 2 sheet that it is possible to purchase CP Ti that both meets the strength requirements of Grade 2 and the chemical requirements of Grade 1. This offers the best option for space missions.

TABLE 1.—CP Ti ROOM TEMPERATURE MINIMUM TENSILE PROPERTIES (REF. 2)

	CP Ti Grade 1	CP Ti Grade 2
0.2% offset yield strength, MPa (ksi)	170 (25)	275 (40)
Ultimate tensile strength, MPa (ksi)	240 (35)	345 (50)
Elongation in 2 in. gauge section, % sheet > 0.025 in. thick	24	20
Reduction in area, bar, %	30	30

Table 2 gives some typical elevated temperature tensile properties. As with the room temperature properties, CP Ti Grade 2 has a significant strength advantage over Grade 1. It also retains strengths at 811 K (1000 °F) that are very usable for many power conversion system applications. The extra temperature capability also adds a safety margin for the systems.

TABLE 2.—TYPICAL CP Ti ELEVATED TEMPERATURE TENSILE PROPERTIES (REF. 3)

	CP Ti Grade 1		CP Ti Grade 2		
	477 K (400 °F)	589 K (600 °F)	477 K (400 °F)	589 K (600 °F)	811 K (1000 °F)
0.2% offset yield strength, MPa (ksi)	103.5 (15)	89.7 (13)	124.2 (18)	103.5 (15)	75.9 MPa (11)
Ultimate tensile strength, MPa (ksi)	193.2 (28)	138.0 (20)	207 (30)	179.4 (26)	131.1 (19)
Elongation in 2 in. gauge section, % sheet > 0.025 in. thick	32	26	37	25	32
Reduction in area, bar, %	70-80	70-80	70-80	70-80	70-80

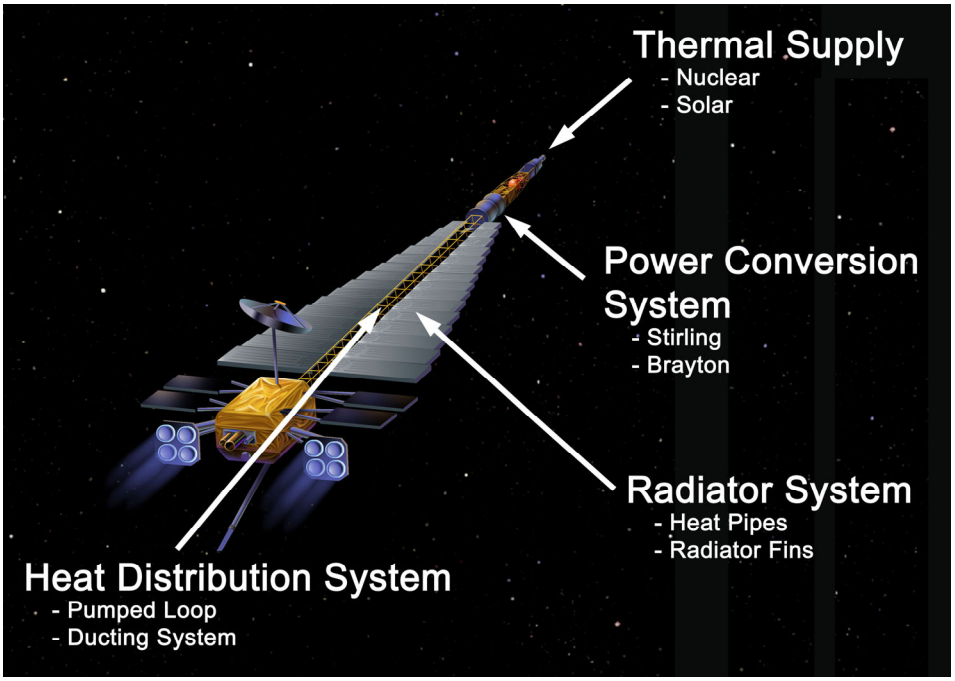


Figure 1.—Typical space power system.

Experimental Procedure

CP Ti was obtained from Titanium Industries[1] as commercially rolled sheet nominally 0.89 mm (0.035 in.) thick. The sheet was provided in the annealed condition in accordance with ASTM Standard B265 (ref. 2).

Samples approximately 76 by 152 mm (3 by 6 in.) were sheared from the larger piece. Each sample was cleaned to remove any grease. The samples were individually placed in Sentry[2] Sen/Pak heat treatment envelopes. The heat treatment envelopes are made from a high Cr stainless steel that acts as both a protective envelope about the samples and an oxygen getter. Alumina rods were placed between the Ti sheet samples and the stainless steel envelopes to prevent diffusion and other interactions. The envelopes were filled with nitrogen and sealed.

Two heat treatment temperatures were selected based upon current designs as a typical operating (550 K/277 °C/531 °F) and a maximum service (650 K/377 °C/711 °F) temperature. Thermal exposures of 1,000 to 5,000 h in 1,000 h increments were selected to assess the changes in the microstructure and mechanical properties with time at these temperatures.

Two furnaces were preheated to the desired temperatures and stabilized prior to loading the furnaces. Thermocouples were placed between the sample envelopes during loading. . Temperatures from these thermocouples during the exposures were recorded using a computer-based data acquisition system.

After exposure the samples were made into tensile test specimens by wire electro discharge machining (EDMing) specimens using the design shown in Figure 2. The design was provided from the Ti-NaK compatibility study (ref. 4) done in conjunction with this work.

The Ti-NaK study generated a considerable number of room temperature tensile tests on as-received material from the same starting sheet of CP Ti used in the current study. Tensile specimens for the Ti-NaK study were machined using water jet cutting rather than wire EDM cutting since the water jet cutting was considerably faster and a large number of samples were required quickly. The water jet cut specimens had their edges hand polished to deburr the edges and remove any thin worked layer of material. To ensure that there were no differences introduced into the results by the machining process and to allow direct comparisons of the Ti-NaK data set and the current data set, five as-received samples were wire EDMed and tested. The two sets of samples were designated As-Received, Water Jet (WJ) and As-Received, Electro Discharge Machining (EDM).

Tensile testing was done using a modified Instron[3] 1125 tensile test load frame. The frame had been upgraded to allow computer control and data acquisition. The upgrades also allowed strain rate control during the testing. The samples were gripped using MTS[4] 647 series hydraulic grips using a gripping force of approximately 3.3 kN (750 lb_f).

Tensile testing was limited to room temperature in this study. Elevated temperature testing is ongoing and will be reported separately.

The room temperature tensile samples were tested using a strain rate of 0.005/min. The sample test order was randomized to minimize the effects of changes in the testing with time. An MTS 632 series general purpose extensometer with a 25.4 mm (1 in.) gauge length was used to measure the strain up to 19.5% or 5.08 mm (0.195 in.) elongation. This point was well past the strain corresponding to the ultimate tensile strength and outside the uniform deformation regime of the samples. After removal of the extensometer, the strain was calculated from the crosshead movement which allowed strain rate control throughout the entire test.

To determine if there were statistically significant differences between the as-received and exposed samples, five repeats were done for all conditions except at 550 K for 1,000 h where only 4 samples were tested due to lack of material. The data from the Ti-NaK as-received samples were included in the

[1]Titanium Industries, 18 Green Pond Road, Rockaway, New Jersey 07866
[2]Sentry Company, 62 Main Street, Foxboro, Massachusetts 02035
[3]Instron Corporation, 825 University Ave., Norwood, Massachusetts 02062-2643
[4]MTS Systems, 14000 Technology Drive, Eden Prairie, Minnesota 55344

analysis as well to determine if there were differences in the tensile properties introduced by the machining method. Systat's[5] SigmaStat Version 3.1 was used to perform a One Way Repeated Measures Analysis of Variance (One Way RM ANOVA) to determine if there were statistically significant differences in the means. If the One Way RM ANOVA indicated that the differences in the means exceeded what could be expected from the variations inherent in each data set, a Student-Neumann-Keuls analysis to rank the average results and to show where differences exist between the various thermal exposures or machining techniques.

To determine if the thermal exposure had any effect on the grain size and morphology of the samples, metallographic mounts were prepared. Each sample was mechanically polished through 0.05 μm silica. An attack polish consisting of 100 ml H_2O – 30 ml H_2O_2 – 30 ml NH_4OH was used in the final step. Care was taken to avoid introducing deformation twins and to remove all worked material. Samples were etched to reveal grain boundaries using Krohl's etchant consisting of 50 ml H_2O – 3 ml 65% HNO_3 acid – 1.5 ml 40% HF acid. Representative micrographs for each condition were obtained using a Reichert ME3 optical microscope.

A semi-quantitative analysis of the grain size was conducted using the line intercept method outlined by Underwood (ref. 5). Only one view was used, so the results lack the statistical significance of a full quantitative analysis using five or more images per condition. SigmaScan Pro Version 5 from Jandel Scientific[6] was used for the image analysis. Prior to measurement each image was calibrated for magnification. A series of parallel lines was electronically overlaid on the image, and the number of intersections between the lines and grain boundaries counted. By summing the total length of the lines and dividing by the number of intersections, the average grain diameter was calculated.

Optical microscopy revealed the presence of many particles within the Ti matrix. A Hitachi S-4700 FESEM was used for Energy Dispersive Spectroscopy (EDS) analysis of the particles to identify their constituents.

Fractography was also conducted on selected samples to determine if any change in fracture mode occurred. In particular, it was desired to see if the thermal exposures affected the strength of the grain boundaries leading to premature failure by decohesion or cleavage at grain boundaries. A JEOL 840A scanning electron microscope (SEM) was selected for the fractography since it had a chamber door large enough to allow insertion of the entire broken sample without cutting or damaging the fractured end.

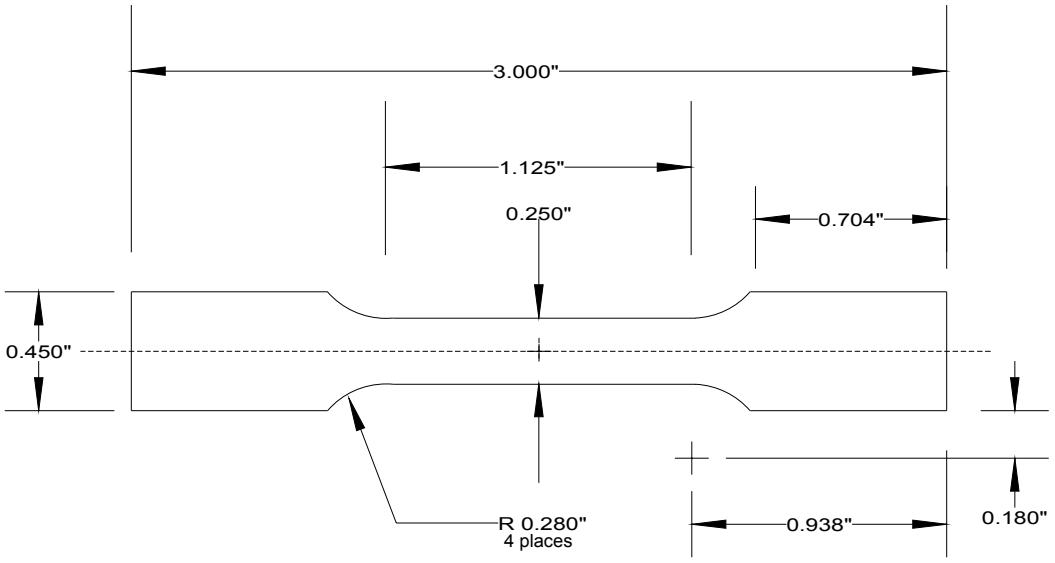


Figure 2.—Room temperature tensile test specimen design.

[5]Systat Software, Inc., 1735, Technology Drive, Ste 430, San Jose, California 95110
[6]Now owned by Systat Software, Inc., 1735, Technology Drive, Ste 430, San Jose, California 95110

Results

Chemistry

The chemistry of the sheet as determined by NASA GRC is presented in Table 3. For comparison the specifications for CP Ti Grades 1 and 2 are presented as well. While the material was designated as Grade 2, it meets the chemical specifications for Grade 1.

TABLE 3.—CP Ti GRADE 2 SHEET CHEMISTRY

Element	NASA GRC	Grade 1 specification (wt. %) (ref. 1)	Grade 2 specification (wt. %) (ref. 1)
Ti	Bal.	Bal.	Bal.
C	0.0140	0.08 max.	0.08 max.
Fe	0.0850	0.20 max.	0.25 max.
N	0.0055	0.03 max.	0.03 max.
O	0.1300	0.18 max.	0.20 max.
H	0.0015	0.015 max.	0.015 max.
Residuals—maximum each	0.0120	0.1	0.1
Residuals—maximum total	0.0299	0.4	0.4

Visual Examination of Samples Following Thermal Exposure

Following exposure at 550 K (531 °F) in a nitrogen atmosphere within a heat treat envelope, the Ti sheet turned a bronze color indicative of surface nitriding (ref. 6). The layer was continuous and very adherent but could be penetrated easily by scratching the surface. No attempt was made to measure the thickness of the layer, but it was not readily apparent during later SEM observations of the fractured surfaces. That meant that the layer was much less than 1 μm thick.

After exposure at 650 K (711 °F) in a nitrogen atmosphere within a heat treat envelope, the Ti sheet dulled and darkened indicating the formation of a thin oxide layer (ref. 6). In the worst case the sheet became a dark grey but most samples were a medium gray color. Some patchy areas with TiN were also observed though they were infrequent and covered only a small portion of the samples. The layer again could be easily penetrated when the surface was lightly scratched. Based on the SEM images of the fractured surfaces, the layer was again much less than 1 μm thick.

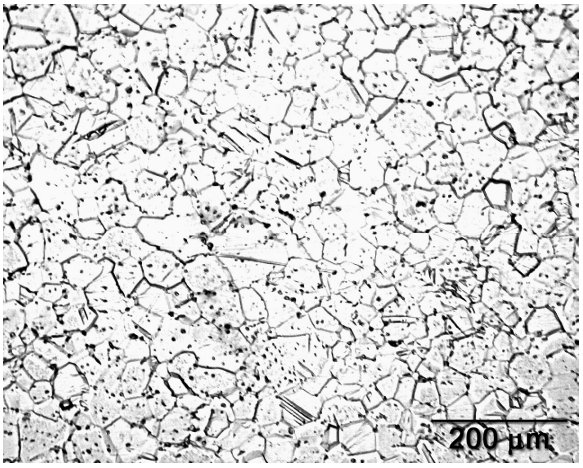
Because the layers are very thin relative to the thickness of the sheet, and there is no indication of intergranular attack, embrittlement or development of notch sensitivity, the layers were ignored during tensile testing.

Microstructure

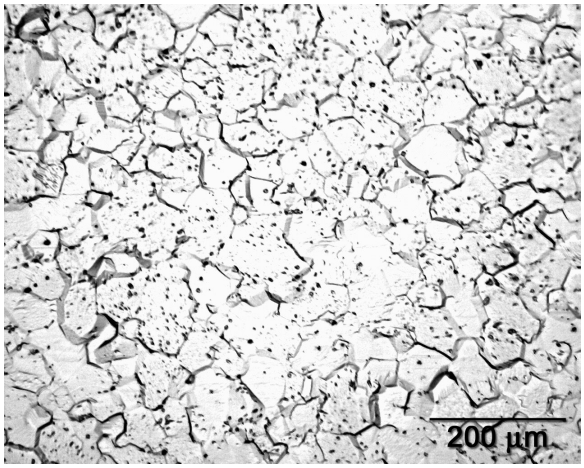
Representative micrographs of the as-received and thermally exposed samples are shown in Figure 3. The exposure temperatures are below the annealing temperature of 973 K (700 °C/1292 °F) and even the stress relief temperature of 773 K (500 °C/932 °F) for CP Ti Grade 2 (ref. 7), so no recrystallization and minimal grain growth were expected even if the mill anneal had not been complete.

While the samples are commercially pure, there are still several trace elements present in measurable quantities as shown by the chemical analysis in Table 3. Many fine precipitates were observed in the polished and etched specimens. The microstructures were consistent with other CP Ti microstructures found in the literature such as those presented by Lathabai et al. (ref. 8). An example of some of the particles can be seen in Figure 4. The EDS spectra such as the one shown in Figure 5 showed that these were Fe containing particles with some trace amounts of Ni and perhaps C as well. The preponderance of the Ti peak relative to the Fe peak is most likely caused by the Ti matrix contained within the excitation volume for the sample. Based upon the Fe-Ti phase diagram (ref. 9), these particles are most likely TiFe with some dissolved Ni.

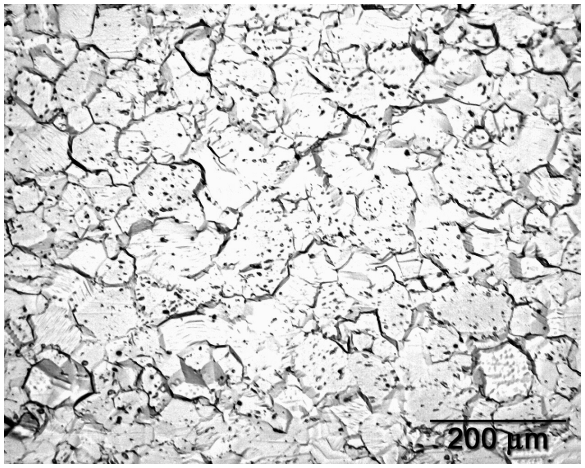
Since there was a concern that the very long thermal exposure could promote grain growth, a semi-quantitative analysis of the grain size was conducted. The results of the semi-quantitative analysis of the images in Figure 5 are presented in Figure 6. No consistent pattern of increased grain size with time or temperature is observed, and most of the average grain sizes are near 40 μm. From this it was concluded that there was no significant grain growth.



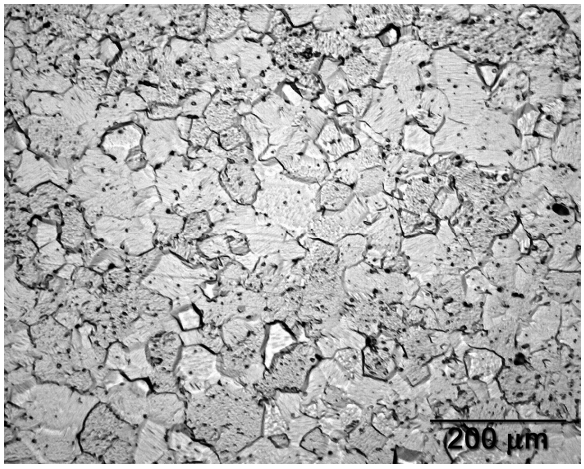
(a) As-Received



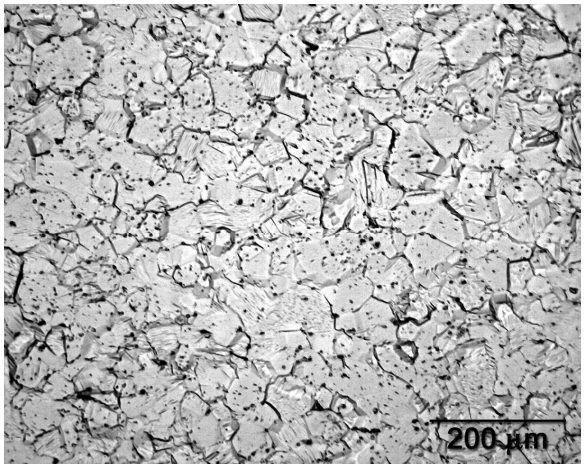
(b) 550 K/1,000 h



(c) 650 K/1,000 h

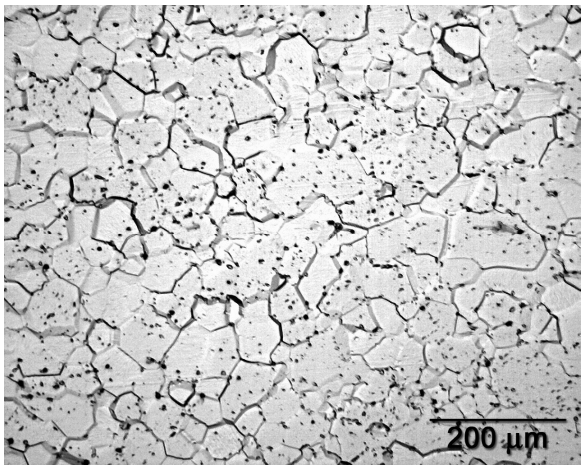


(d) 550 K/2,000 h

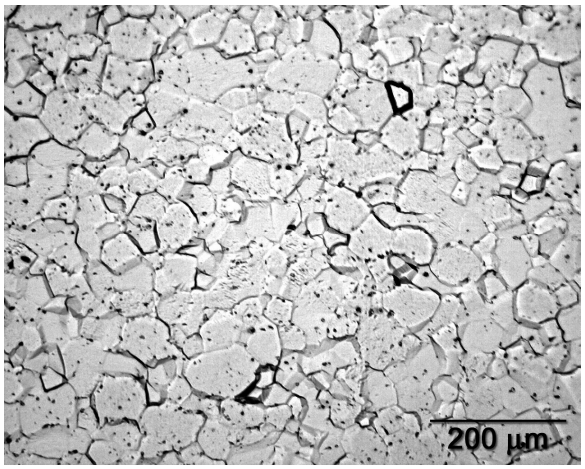


(e) 650 K/2,000 h

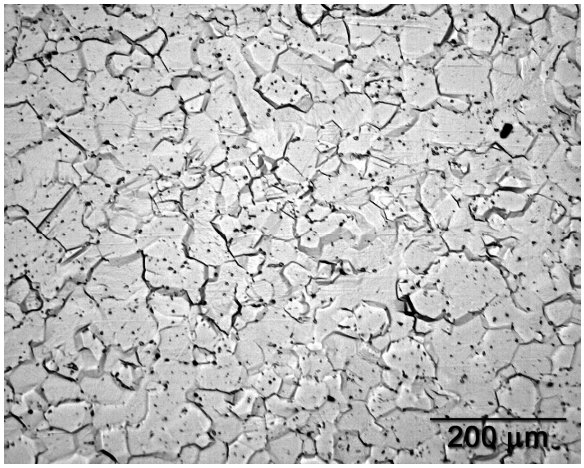
Figure 3.—Optical micrographs of CP Ti grade 2 samples.



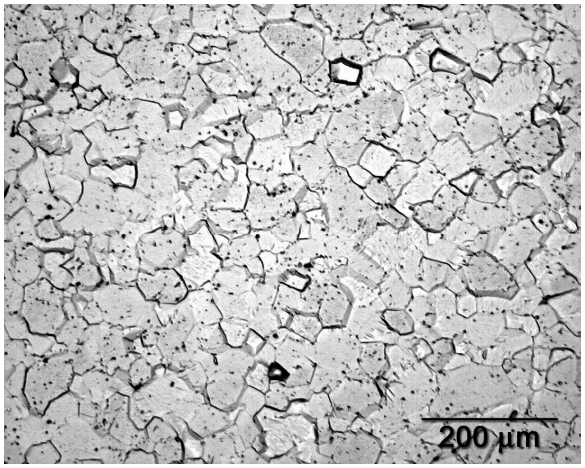
(f) 550 K/3,000 h



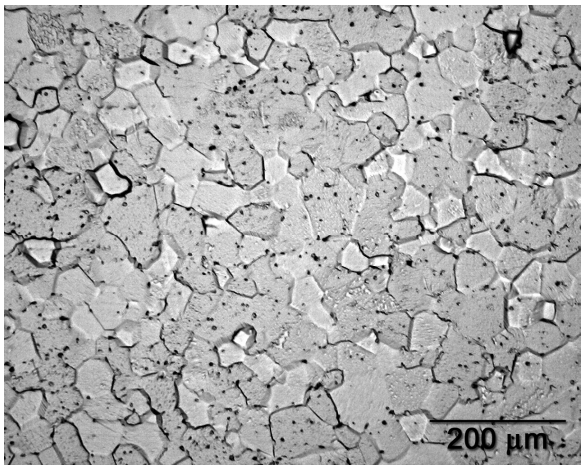
(g) 650 K/3,000 h



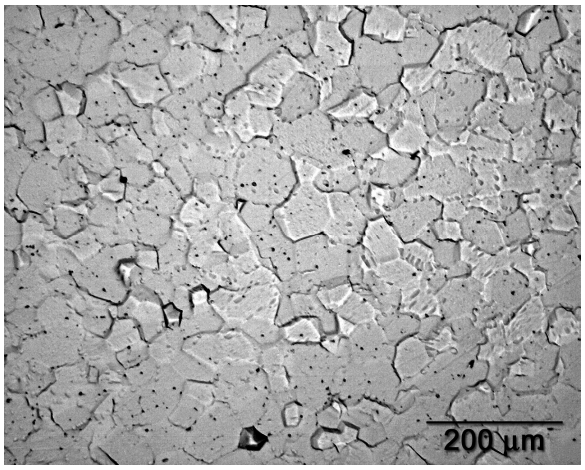
(h) 550 K/4,000 h



(i) 650 K/4,000 h



(j) 550 K/5,000 h



(k) 650 K/5,000 h

Figure 3.—Concluded.

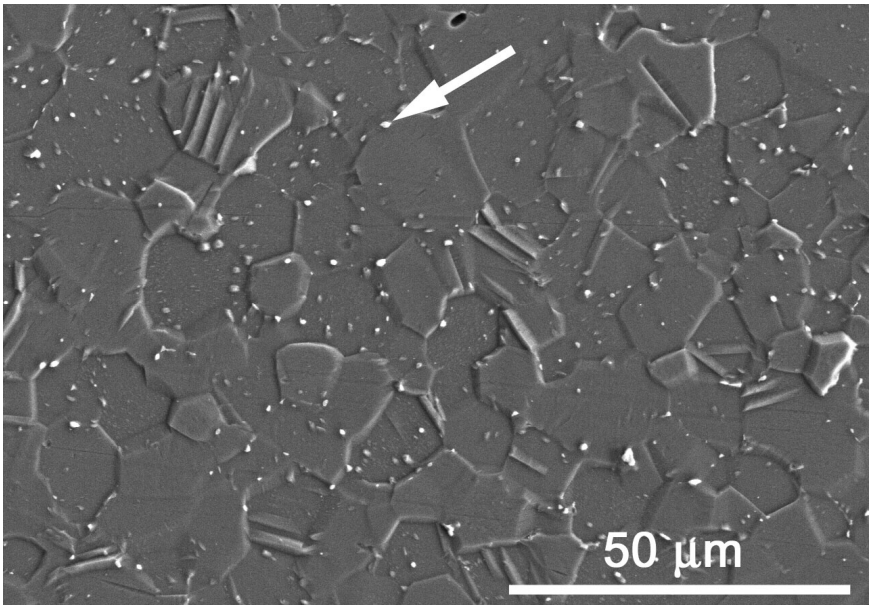


Figure 4.—Secondary electron SEM image showing many fine particles uniformly distributed throughout as-received CP Ti grade 2.

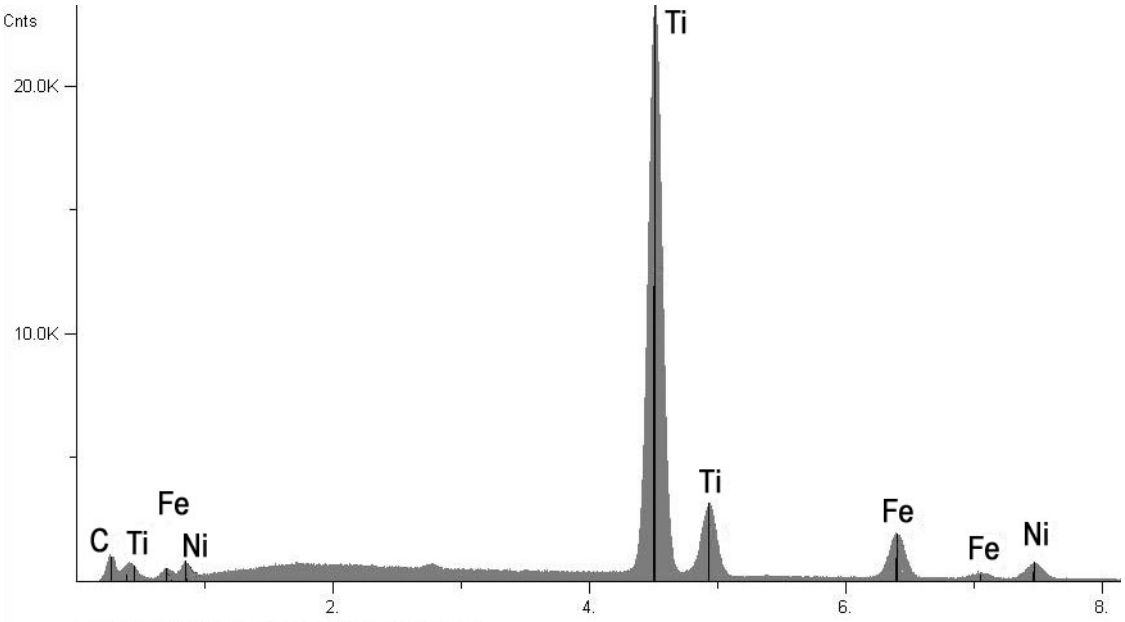


Figure 5.—Typical particle EDS spectra.

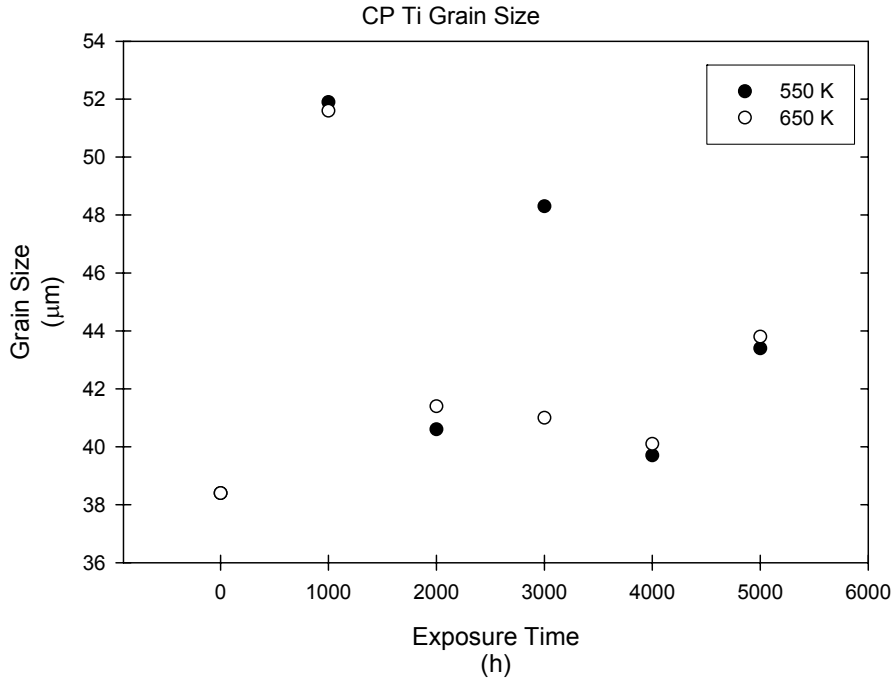


Figure 6.—Average CP Ti grain size as a function of thermal exposure.

Tensile Testing

All specimens exhibited high ductility and considerable necking prior to failure. Failures were consistently located in the middle half of the specimen.

A typical stress-strain curve is shown in Figure 7. There was evidence of a discontinuous yield point for the samples as shown by the small discontinuity in the transition from elastic to plastic deformation. While not observed as a serrated stress-strain curve, the discontinuity is most likely due to the formation of deformation twins (ref. 10). For pure Ti at room temperature $(11\bar{2}2)$ twinning dominates (ref. 11). Paton and Backofen also determined that the stress required for $(11\bar{2}2)$ twinning increases with temperature while the stress for $(10\bar{1}1)$ twinning decreases with temperature until at 400 °C (752 °F) $(10\bar{1}1)$ accompanied by an a+c dislocation slip becomes the dominant twinning mechanism. This change will be important for future elevated temperature tensile testing.

The results of the tensile testing are summarized in Figure 8. Appendix A contains the results for each specimen and the average, standard deviation and the one-sided 95% confidence limit of each data set. The bars in Figure 8 represent the average for each property at a given exposure. The error bars at the ends of each bar represent the two-sided 95% confidence interval for the average based upon a statistical analysis of the results for each data set.

To establish normality for each data set, Normal Scores (NS) testing was used rather than the Kolmogorov-Smirnov test statistic because of the small size of each data set. A probability plot was made for each data set, and the fit of the data as measured by the R^2 value was compared to the critical value, e.g., 0.57 for five data points. During this analysis two data points in the water jet cut as-received data set (RT-1A and RT-1D) were identified as outliers from the normality plot and discarded from the data set. The results listed in Table 4 showed that all data was normally distributed, so standard statistical comparisons and analyses could be conducted.

TABLE 4.—NORMALITY SCORES (R^2 VALUES) FOR EACH DATA SET

Data set	Yield strength	UTS	Elongation
As received, water jet	0.958	0.966	0.958
As received, EDM	0.898	0.991	0.893
550 K/1000 h	0.893	0.676	0.837
550 K/2000 h	0.724	0.729	0.987
550 K/3000 h	0.848	0.914	0.920
550 K/4000 h	0.872	0.991	0.872
550 K/5000 h	0.657	0.732	0.974
650 K/1000 h	0.934	0.895	0.851
650 K/2000 h	0.919	0.930	0.913
650 K/3000 h	0.973	0.718	0.994
650 K/4000 h	0.870	0.971	0.995
650 K/5000 h	0.848	0.906	0.945

A One Way RM ANOVA test was conducted for each tensile property (yield strength, ultimate tensile strength and elongation). The results of the One Way RM ANOVAs appear in Table 5.

In all cases the One Way RM ANOVA indicated that the differences between means were greater than would be expected by random chance, so a Student-Neumann-Keuls (SNK) analysis was conducted to compare the means. The graphical summary of those comparisons is given in Figure 9. In the SNK analysis multiple comparison procedure, the means are ranked from highest to lowest. A $K \times K$ half matrix where K is the number of data sets (12 in this study) is formed. The value for each cell is calculated using the following equation:

$$Cell_{ij} = \frac{\overline{Y}_{(j\bullet)} - \overline{Y}_{(i\bullet)}}{\frac{S_P}{\sqrt{\frac{\sum N_i}{K}}}} \tag{1}$$

where $\overline{Y}_{(j\bullet)}$ = mean ranked value of column j, $\overline{Y}_{(i\bullet)}$ = mean ranked value of row i, S_P = pooled standard deviation, N_i = number of data points in data set i and K = number of data sets. The values of each cell in the half matrix are compared to a critical value which depends on the order difference (j–i), the degrees of freedom ($K\sum_{i=1}^{K} N_i - 1$) and the probability selected ($1-\alpha$). If the value of a cell exceeds the critical value in the pairwise comparison then the two means are statistically different. If the value of the cell is less than the critical value than the two means are statistically equal. All comparisons were done using a 95% probability ($1 - \alpha = 0.95$)

The easiest way to present the results of the comparisons is graphically with a series of lines representing which data set means are statistically equal. In Figure 9, anytime a line is underneath the treatment, the means connected by that line are statistically equal while any means to the left of the line are statistically greater than the mean and any means to the right are statistically less than the mean. Comparisons between means take into account all lines generated by the pairwise comparisons, so any mean that has any line connecting it to a value to the left or right is statistically equal to those means.

For example, at a 95% confidence level, the 550 K/2000 h elongation mean is statistically less than the 650 K/5000 h mean. It is not statistically less than the 550 K/4000 h, 650 K/1000 h, 650 K/4000 h, and 650 K/3000 h means nor statistically greater than the 650 K/2000 h, As-received WJ, and As-received EDM means. It is statistically greater than the 550 K/5000 h, 650 K/3000 h, and 550 K/1000 h means.

The results of the SNK procedure do show that there are statistically significant differences in the means for all three tensile properties. However, examination of the results did not show any apparent trend in the values with respect to either exposure time or the exposure temperature. In the case of strength most of the exposures had statistically equal strengths. The only result that appears to be consistent and meets expectations

is the highest temperature and longest time did produce the lowest mean strength and one of the highest elongations.

TABLE 5—ONE WAY RM ANOVA OF ROOM TEMPERATURE TENSILE TEST RESULTS

(a) 0.2% Offset yield strength

Source of Variation	DF	SS	MS	F	P
Between Subjects	21	1068.159	50.865		
Between Treatments	11	1374.320	124.938	6.474	<0.001
Residual	43	829.836	19.299		
Total	75	4113.630	54.848		

(b) Ultimate tensile strength

Source of Variation	DF	SS	MS	F	P
Between Subjects	21	188.611	8.981		
Between Treatments	11	784.208	71.292	3.928	<0.001
Residual	43	780.468	18.150		
Total	75	2813.378	37.512		

(c) Elongation

Source of Variation	DF	SS	MS	F	P
Between Subjects	21	160.327	7.635		
Between Treatments	11	650.509	59.137	20.349	<0.001
Residual	43	124.963	2.906		
Total	75	955.578	12.741		

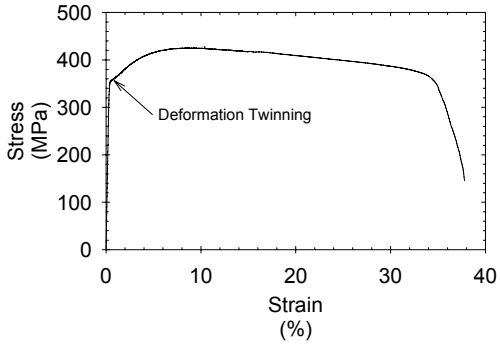


Figure 7.—Typical room temperature engineering stress-strain curve for as-received and thermally exposed CP Ti grade 2 samples

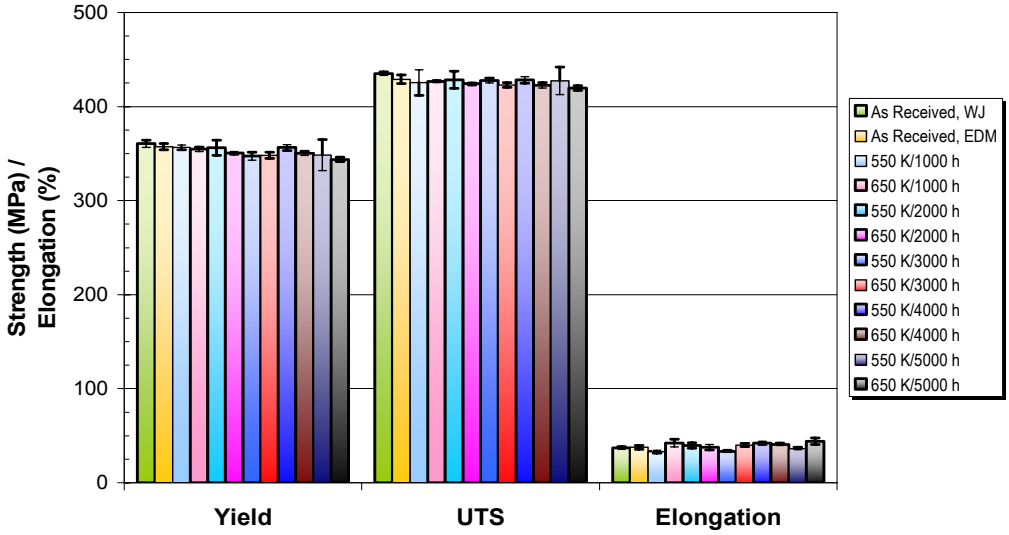


Figure 8.—Tensile test results for as-received and thermally exposed CP Ti grade 2.

0.2% Offset Yield Strength

Rank	1	2	3	4	5	6	7	8	9	10	11	12
Treatment	As-Received WJ	As-Received EDM	550K/1000h	550K/4000h	550K/2000h	650K/1000h	650K/2000h	650K/4000h	550K/5000h	650K/3000h	550K/3000h	650K/5000h
Average	360.5	357.0	356.6	356.4	356.3	354.6	350.8	350.5	348.5	348.2	347.3	343.8

Ultimate Tensile Strength

Rank	1	2	3	4	5	6	7	8	9	10	11	12
Treatment	As-Received WJ	As-Received EDM	550K/2000h	550K/4000h	550K/3000h	550K/5000h	650K/1000h	550K/1000h	650K/2000h	650K/3000h	650K/4000h	650K/5000h
Average	435.1	434.9	428.5	428.4	427.6	427.4	426.7	425.5	424.4	422.9	422.6	419.5

Elongation

Rank	1	2	3	4	5	6	7	8	9	10	11	12
Treatment	650K/5000h	550K/4000h	650K/1000h	650K/4000h	650K/3000h	550K/2000h	650K/2000h	As-Received EDM	As-Received WJ	550K/5000h	650K/3000h	550K/1000h
Average	44.2	42.3	42.1	40.8	40.2	39.6	37.7	37.4	37.1	36.4	33.5	33.1

Interpretation: Values that share a line in any row have statistically equal means
Data sets are referred to by their heat treatment temperature and time

Figure 9.—Results of Student-Neuman-Keuls (SNK) test analysis.

Fractography

Figure 10 shows a macrograph of two typical failed tensile test specimens. Notable in the image is the near 45° angle between the applied axial load (horizontal axis in the image) and the fracture surface. This is typical of almost all of the specimens. The angle indicates that shear stresses were established during the uniaxial tensile testing (ref. 12). This is consistent with a change from a uniaxial to triaxial stress state during the tensile test as necking occurs (ref. 13). The shear stresses are greatest along planes oriented 45° to the applied load and result in the eventual failure of the specimen in the plane of the shear stresses.

The SEM results of the fractography are shown in Figure 11. The surfaces are dimpled but the dimples tend to be elliptical to varying degrees rather than circular. This is consistent with literature fractographs of CP Ti parts that failed in shear (ref. 14). In the images the shear and the plane of the fracture surfaces are tilted ~45° relative to the plane of the paper. The shear direction would project to the horizontal direction in the images.

The edges of the specimens were examined to determine if the machining methods used (water jet and wire EDM) and the small amounts of nitriding (550 K exposures) and oxidation (650 K exposures) had any detectable effect. As shown in Figure 12, the morphology of the fracture surfaces is consistent from the center to the edges. This is also consistent with the results of the tensile testing which show minimal difference in the tensile strength with no apparent correlation to the exposure temperature or machining method.

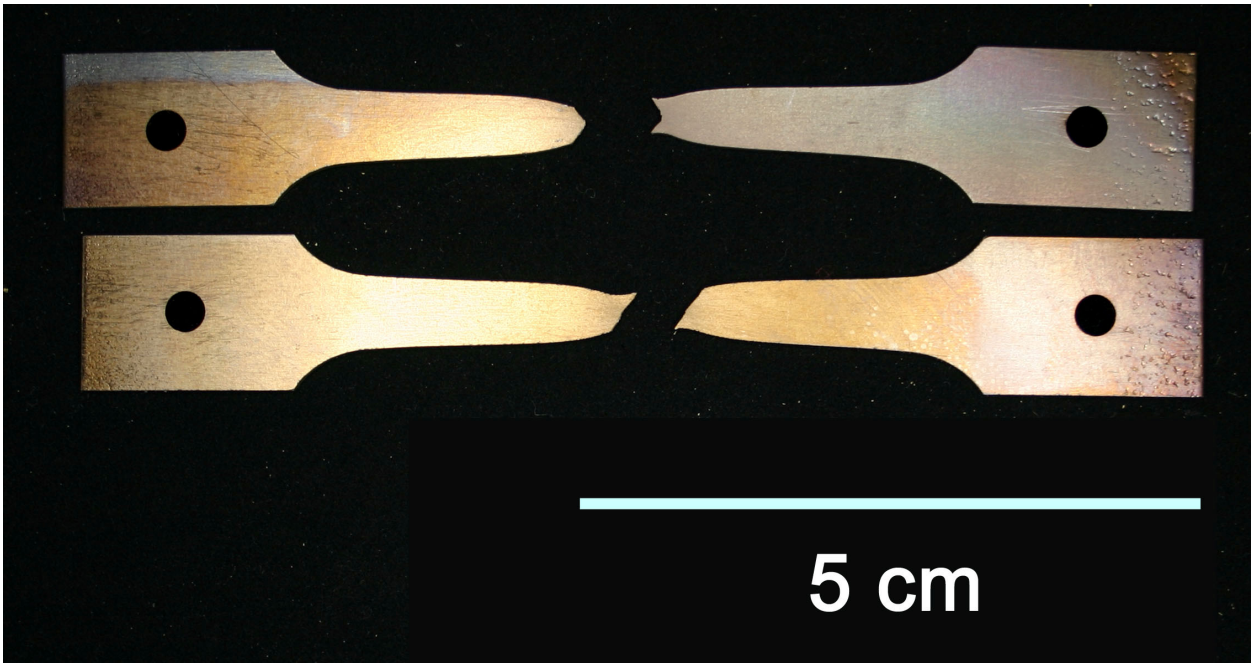
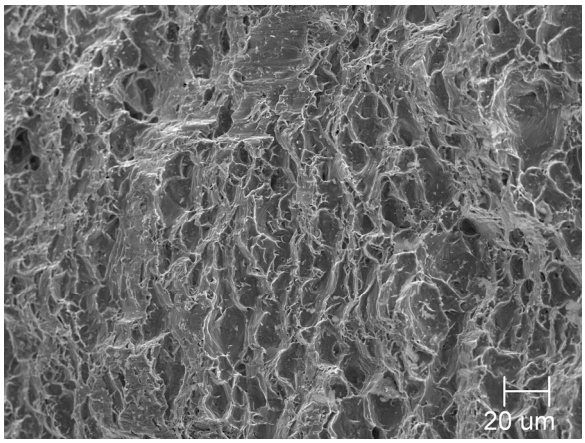
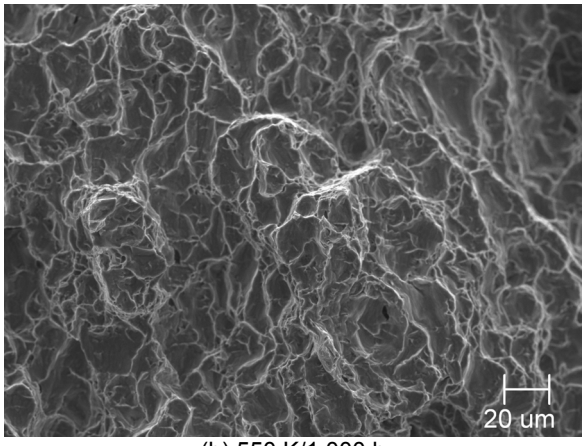


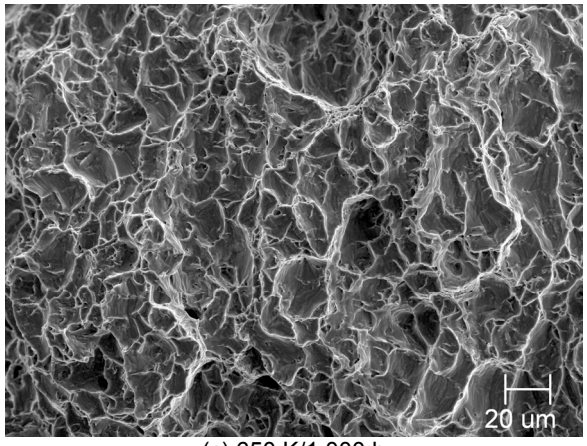
Figure 10.—Room temperature tensile test specimens following testing.
(Top—as received sample RT-2D, bottom—as-received sample RT-2B.)



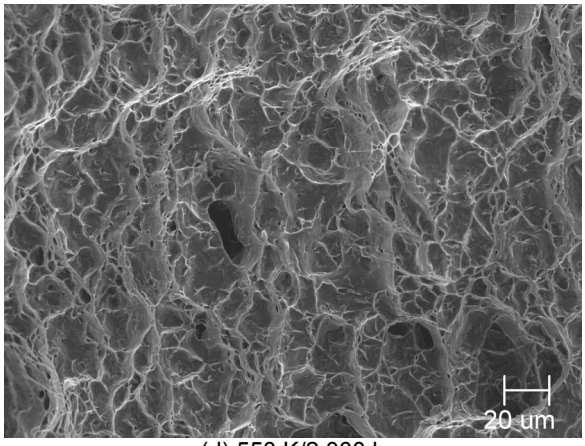
(a) As-received



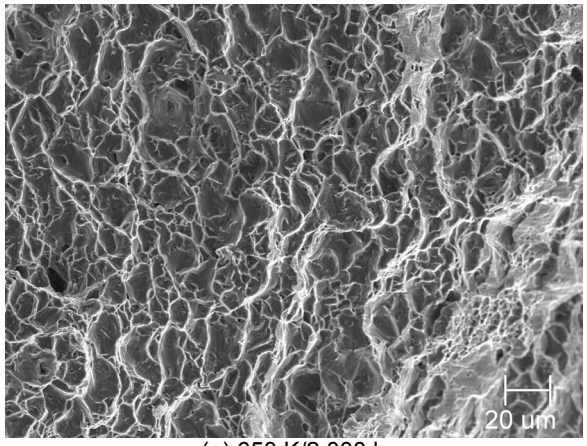
(b) 550 K/1,000 h



(c) 650 K/1,000 h

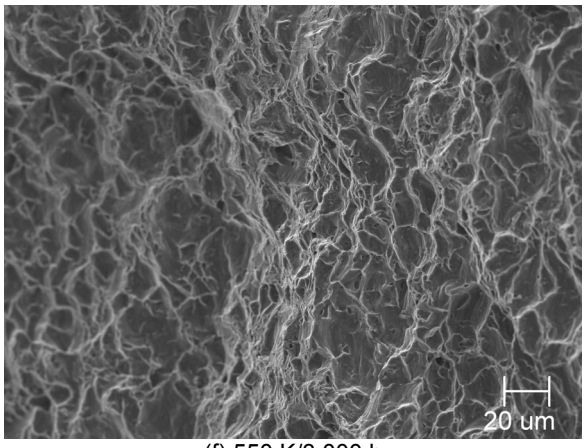


(d) 550 K/2,000 h

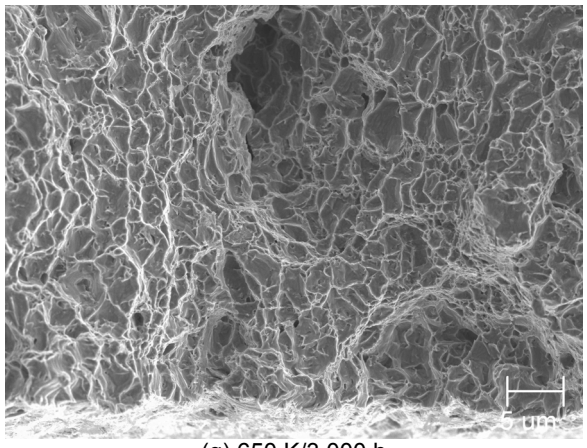


(e) 650 K/2,000 h

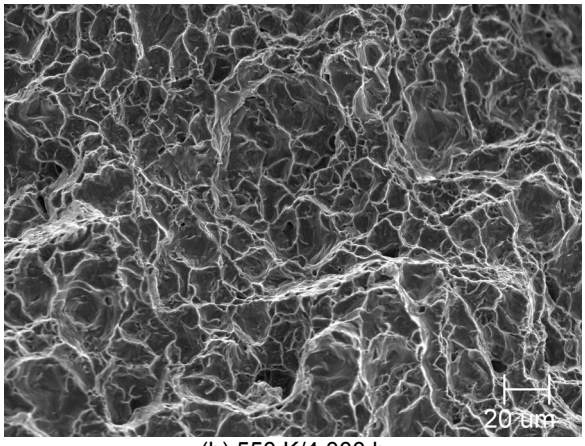
Figure 11.—Room temperature tensile test fracture surfaces.



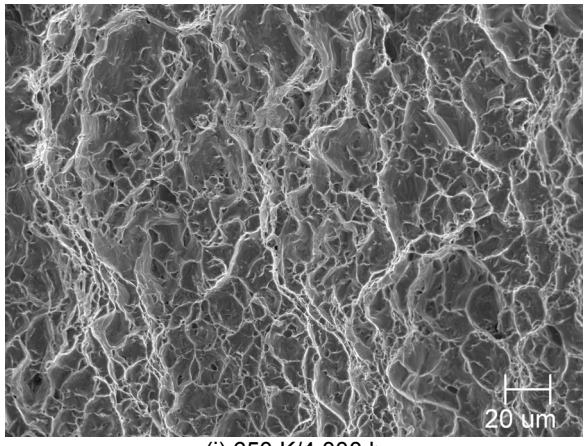
(f) 550 K/3,000 h



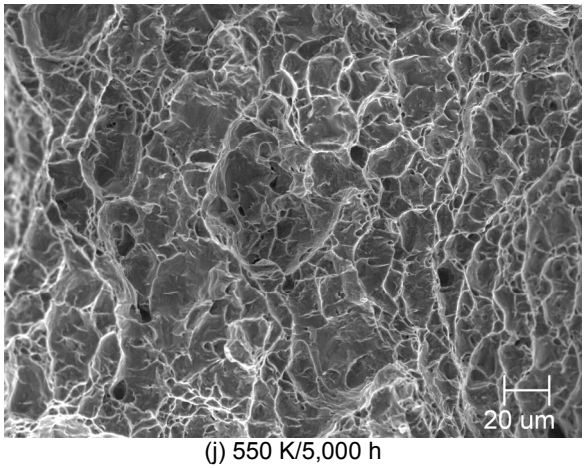
(g) 650 K/3,000 h



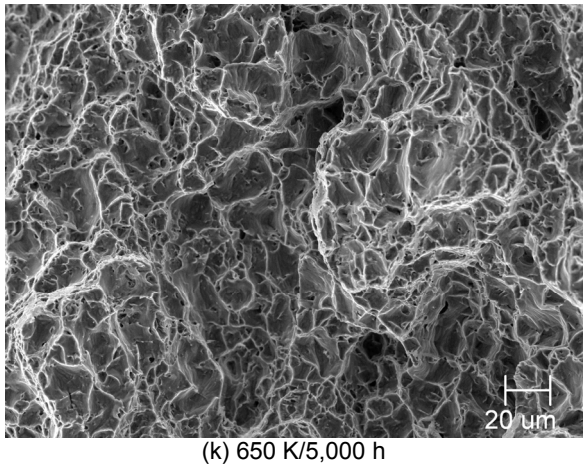
(h) 550 K/4,000 h



(i) 650 K/4,000 h

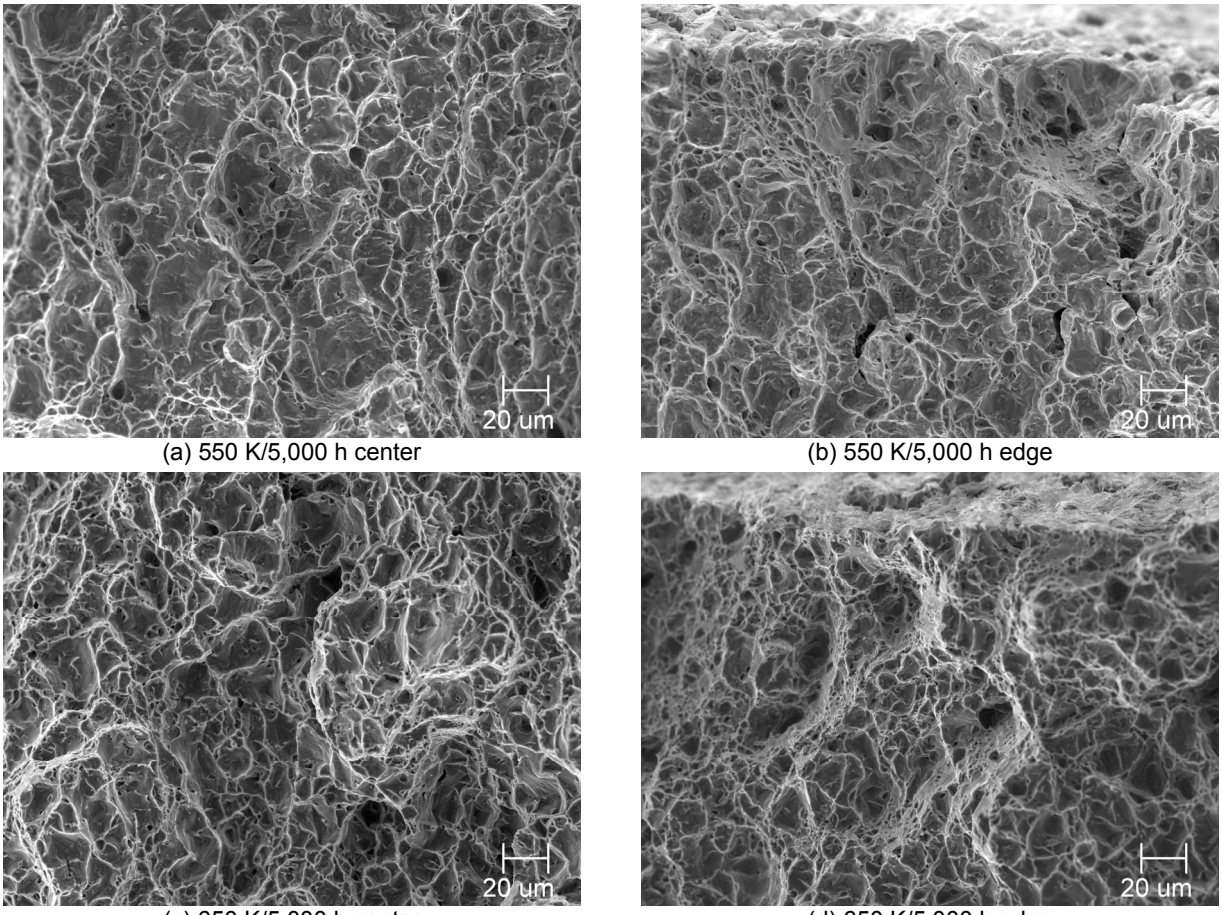


(j) 550 K/5,000 h



(k) 650 K/5,000 h

Figure 11.—Concluded.



(a) 550 K/5,000 h center (b) 550 K/5,000 h edge

(c) 650 K/5,000 h center (d) 650 K/5,000 h edge

Figure 12.—Comparison of edge and center fracture surfaces.

Discussion

Microstructural Changes

While the optical micrographs in Figure 3 give the appearance of possible grain size changes, the semi-quantitative analysis presented in Table 3 indicates otherwise. The apparent discrepancy appears to arise from variability in the etching of the grain boundaries and the inability to easily distinguish the lighter grain boundaries in Figure 3. When observed at higher magnification additional grain boundaries become apparent.

The exposure temperatures were well below the annealing temperature of CP Ti (ref. 5). The material also had been annealed prior to delivery. As a result the grains in the as-received material were equiaxed and had minimal twins. Grain sizes for each sample were not measured rigorously, but as shown in Table 3 they do not vary greatly with thermal exposure and all were similar to the as-received material.

Since there were no changes in the grain size, changes in Hall-Petch and other grain size dependent strengthening mechanisms would not occur. As a result, there was no expectation of changes in the yield or ultimate tensile strengths of the samples following the thermal exposures. As the statistical analysis of the tensile test data sets showed, this was consistent with the tensile testing results which show minimal differences in the means of the twelve data sets.

Fracture of Thermally Exposed CP Ti

No changes were observed in the fracture surfaces of the tensile specimens. Changes in the fracture mode from microvoid coalescence and growth to cleavage or grain boundary decohesion were sought but

not observed even at the surface where some corrosion was known to have occurred. O and/or N could have preferentially diffused along the grain boundaries to embrittle them. Given the long times involved, grain boundary enhanced diffusion could have resulted in deep penetration of the O and N into the Ti. None of this was observed.

The lack of change in fracture was expected but had to be confirmed. In general CP Ti has excellent corrosion resistance which is why it is used in so many chemical applications and aggressive environments (refs. 15 and 16). The major concern was the formation of secondary phases such as oxides or hydrides at grain boundaries or diffusion of the trace elements such as O, N and Fe to the grain boundaries to weaken them. For up to 5,000 h at 650 K, no evidence that any of this had occurred was found in the fractography or optical microscopy. This supports the use of CP Ti for long durations at elevated temperatures.

Differences In Room Temperature Tensile Properties

Some statistically significant differences were observed between the data sets. As can be seen in Figures 8 and 9, there are some subtle differences in the mean strengths and elongations of the twelve data sets.

The SNK analysis shows that the as-received specimens were not affected by the machining method. As such, comparisons in the future can be made between the Ti-NaK samples after exposure in NaK and thermally exposed specimens without concern for the effects of machining on room temperature tensile strength. While it will be confirmed, it is also expected that the elevated temperature tensile properties will not be affected by machining technique.

There were statistically significant differences observed, but in most cases the results did not indicate a pattern based upon the exposure time or temperature. It was observed that, as one would expect, the longest time at the highest temperature produces the lowest strength. The rest of the differences observed appeared to follow a random pattern.

In practical engineering terms, the differences in strengths, while statistically significant, likely do not rise to the level of constituting a difference large enough to require factoring in a loss on properties with time. The entire range of the yield strength (16.6 MPa/2.4 ksi) and UTS (15.6 MPa/2.3 ksi) is small relative to even the smallest mean value (4.8% for yield, 3.7% for UTS). The lowest mean strength values also still greatly exceed the specifications for the minimum room temperature strength for CP Ti Grade 2 listed in Table 1. Since there appears to be no consistent trend for reduction in strength with time or temperature, it is suspected the variations are more consistent with the normal variation of the strength of the Ti sheet. The differences become statistically significant because of the very large number of tests and high number of degrees of freedom which tend to make small differences significant.

The range for the elongation (11.2%) is more of a concern since the span is a third of the smallest mean elongation measured. However there seemed to be no dependency of the mean elongation on time or temperature. The minimum mean value came for the least severe thermal exposure, 550 K for 1,000 h while the highest mean elongation was from the 650 K/5000 h exposure. It appears that these values again reflect the random variation of elongation for CP Ti sheet specimens. Most importantly, the minimum mean elongation still greatly exceeds the minimum room temperature elongation specification.

For all practical purposes, thermal exposures at 550 and 650 K for times up to 5,000 h do not result in changes in the tensile properties of chemically pure titanium. A designer for power conversion systems will need to be aware of the small changes in the room temperature tensile properties with thermal exposure, but the magnitude of the changes are small enough that it should not require changes from a design using the minimum room temperature tensile properties and typical design safety factors.

Summary and Conclusions

Tensile testing revealed small, statistically significant differences in the tensile strengths of CP Ti Grade 2 following thermal exposure. The range in strengths was small and would normally not affect the design of

parts in the thermal management system since the minimum mean of the thermally exposed specimens still exceeded the specification for the minimum room temperature strengths by a large margin. Elongations showed a wider range of means, but the lowest mean again exceeds the minimum room temperature specification and should not affect a design.

Thermal exposure does not appear to precipitate phases or concentrate trace elements at the grain boundaries as evidenced by the optical microscopy and fractography. It also does not greatly affect the grain size of the CP Ti Grade 2 samples. There is therefore no major weakening of the material, loss of ductility or embrittlement observed.

Based upon the room temperature tensile test results, CP Ti Grade 2 is suitable for use in a variety of parts in space power conversion systems at operating temperatures up to at least 650 K (711°F) in service for long durations. Confirmation of data on chemical compatibility in relevant environments is required, but CP Ti should be chemically compatible with the anticipated fluids for Fission Surface Power and other Prometheus Program heat rejection systems. With its low density and now confirmed mechanical stability, CP Ti offers many attractive possibilities for applications throughout the power conversion systems.

Future Work

Additional tensile testing at 550 and 650 K is ongoing for all material conditions. Those results will be reported when the testing is completed. The results will also be compared to the tensile properties of CP Ti Grade 2 samples taken from the same sheet and exposed at 550 and 650 K in a liquid NaK environment when those data are available.

Appendix A—Results of Room Temperature Tensile Tests

As Received, Water Jet	Yield	UTS	Elongation
RT-1A†	329.7	463.2	33.0
RT-1B	363.5	427.4	32.8
RT-1C	362.5	434.0	35.0
RT-1D†	327.8	462.0	33.8
RT-1E	352.8	439.7	35.4
RT-1F	351.7	432.2	35.6
RT-1G	370.7	437.1	41.4
RT-1H	363.1	435.3	34.1
RT-1I	347.9	433.9	40.7
RT-1J	352.6	434.5	44.0
RT-1K	356.6	432.1	39.4
RT-1L	360.9	434.3	34.7
RT-1M	354.3	431.0	37.1
RT-1N	360.3	433.5	34.9
RT-1O	367.0	434.1	36.0
RT-1P	351.1	439.4	37.8
RT-1Q	371.8	440.2	37.1
RT-1R	370.0	438.8	37.4
RT-1S	347.5	438.1	38.8
RT-1T	367.0	437.5	39.7
RT-1U	369.6	435.9	35.8
RT-1V	360.8	430.5	34.2
RT-1W	361.0	432.9	41.0
RT-1X	367.3	439.6	33.0
Average	**360.5**	**435.1**	**37.1**
SD	**7.6**	**3.4**	**3.0**
One-Sided 95% Interval	**3.9**	**1.8**	**1.5**

As Received, EDM	Yield	UTS	Elongation
RT-2A	355.3	425.0	35.7
RT-2B	354.9	427.4	38.2
RT-2C	358.1	432.5	37.7
RT-2D	359.5	429.4	38.9
RT-2E	358.5	430.7	37.6
Average	**357.2**	**429.0**	**37.6**
SD	**2.0**	**2.9**	**1.2**
One-Sided 95% Interval	**3.2**	**4.6**	**1.9**

550K/1000 h	Yield	UTS	Elongation
RT-3A	356.0	428.3	32.5
RT-3B	357.6	429.3	34.0
RT-3C	355.2	415.7	32.8
RT-3D	357.7	428.7	32.9
RT-3E‡			
Average	**356.6**	**425.5**	**33.1**
SD	**1.2**	**6.5**	**0.7**
One-Sided 95% Interval	**2.6**	**13.7**	**1.4**

650K/1000 h	Yield	UTS	Elongation
RT-4A	355.4	427.5	37.8
RT-4B	355.3	425.9	42.0
RT-4C	353.8	426.0	43.1
RT-4D	356.3	426.5	44.7
RT-4E	352.3	427.4	43.0
Average	**354.6**	**426.7**	**42.1**
SD	**1.6**	**0.7**	**2.6**
One-Sided 95% Interval	**2.4**	**1.2**	**4.1**

550K/2000 h	Yield	UTS	Elongation
RT-5A	347.1	418.3	38.4
RT-5B	359.3	432.1	40.7
RT-5C	357.8	429.9	37.4
RT-5D	359.9	432.6	42.3
RT-5E	357.2	429.6	39.4
Average	**356.3**	**428.5**	**39.6**
SD	**5.2**	**5.9**	**1.9**
One-Sided 95% Interval	**8.2**	**9.2**	**3.0**

650K/2000 h	Yield	UTS	Elongation
RT-6A1	351.8	424.9	39.1
RT-6B	351.2	423.5	38.3
RT-6C	350.4	425.6	39.6
RT-6D	349.4	423.7	36.0
RT-6E	351.3	424.1	35.4
Average	**350.8**	**424.4**	**37.7**
SD	**0.9**	**0.9**	**1.9**
One-Sided 95% Interval	**1.5**	**1.4**	**2.9**

550K/3000 h	Yield	UTS	Elongation
RT-7A	346.1	426.1	33.5
RT-7B	350.0	427.4	34.5
RT-7C	344.9	425.9	33.0
RT-7D	345.2	429.6	33.6
RT-7E	350.4	429.1	32.8
Average	**347.3**	**427.6**	**33.5**
SD	**2.7**	**1.7**	**0.7**
One-Sided 95% Interval	**4.2**	**2.6**	**1.0**

650K/3000 h	Yield	UTS	Elongation
RT-8A	345.6	420.0	40.2
RT-8B	348.7	423.1	38.8
RT-8C	348.9	423.6	41.6
RT-8D	346.7	423.7	40.6
RT-8E	351.0	424.0	39.6
Average	**348.2**	**422.9**	**40.2**
SD	**2.1**	**1.7**	**1.1**
One-Sided 95% Interval	**3.3**	**2.6**	**1.6**

550K/4000 h	Yield	UTS	Elongation
RT-9A	359.1	431.3	43.0
RT-9B	354.8	425.4	42.5
RT-9C	355.5	429.4	43.4
RT-9D	357.8	427.4	40.5
RT-9E	354.9	428.6	42.3
Average	**356.4**	**428.4**	**42.3**
SD	**1.9**	**2.2**	**1.1**
One-Sided 95% Interval	**3.0**	**3.5**	**1.7**

650K/4000 h	Yield	UTS	Elongation
RT-10A	351.3	424.1	40.5
RT-10B	349.1	421.6	41.3
RT-10C	351.2	424.6	41.8
RT-10D	348.8	420.0	40.8
RT-10E	352.0	422.7	39.8
Average	**350.5**	**422.6**	**40.8**
SD	**1.4**	**1.9**	**0.8**
One-Sided 95% Interval	**2.2**	**2.9**	**1.2**

550K/5000 h	Yield	UTS	Elongation
RT-11A	353.7	432.8	36.8
RT-11B	355.0	433.6	36.5
RT-11C	351.1	431.2	35.5
RT-11D	329.6	411.1	35.8
RT-11E	353.1	428.1	37.6
Average	**348.5**	**427.4**	**36.4**
SD	**10.7**	**9.3**	**0.8**
One-Sided 95% Interval	**16.7**	**14.6**	**1.3**

650K/5000 h	Yield	UTS	Elongation
RT-12A	343.4	419.0	41.3
RT-12B	342.2	418.1	47.6
RT-12C	343.3	420.3	44.5
RT-12D	346.5	422.0	44.3
RT-12E	343.8	418.3	43.2
Average	**343.8**	**419.5**	**44.2**
SD	**1.6**	**1.6**	**2.3**
One-Sided 95% Interval	**2.5**	**2.6**	**3.6**

† Outlier
‡ Missing Specimen

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14. ABSTRACT
Room temperature tensile testing of Chemically Pure (CP) Titanium Grade 2 was conducted for as-received commercially produced sheet and following thermal exposure at 550 and 650 K for times up to 5,000 h. No significant changes in microstructure or failure mechanism were observed. A statistical analysis of the data was performed. Small statistical differences were found, but all properties were well above minimum values for CP Ti Grade 2 as defined by ASTM standards and likely would fall within normal variation of the material.

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