ELEVATED TEMPERATURE CHARACTERIZATION OF DISPERSION STRENGTHENED, DIRECT LASER DEPOSITED Ti-8Al-1Er

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Abstract

Direct laser deposited Ti-8Al-1Er combines the technology of the Laser Engineered Net Shaping (LENS™) process with in-situ dispersion alloying and rapid solidification of an α titanium alloy. The result is a low cost, precipitation strengthened titanium alloy that has potential for excellent high temperature properties.

The room temperature tensile, elevated temperature tensile, and creep properties of direct laser deposited Ti-8Al-1Er were measured and compared to Ti-6Al-4V, the most common titanium alloy used in aircraft, and Ti-6242, the most common commercial titanium alloy developed for high temperature use in compressor disks. Elevated temperature tensile properties were measured between 93^o to 538^o C. Creep tests were performed between 445^o to 478^o C. It was found that the room temperature strengths of Ti-8Al-1Er are comparable to those of Ti-6Al-4V and Ti-6242. The elevated temperature strengths of Ti-8Al-1Er are superior to those of Ti-6Al-4V and comparable to Ti-6242 in both absolute strength and in percentage of room temperature strength retained at temperature. The creep resistance of Ti-8Al-1Er is superior to that of Ti-6Al-4V and comparable to that of Ti-6242. The primary concern with the new product is its low ductility, less than 1% at room temperature.

This work indicates that direct laser deposited in-situ alloyed titanium products may hold promise for elevated temperature applications. However, further work is needed to develop commercially feasible products.

Introduction

The Laser Engineered Net Shaping (LENS™) process, also known as Direct Laser Deposition, is a process under development for fabrication of near-net shaped parts. [1-4] The LENS™ process uses a CAD file of the part being made to control a laser and metal powder stream being rastered back and forth to build up a 3-dimensional part. As metal powder is being expelled from a nozzle at the proper location, a laser is focused at this spot to locally melt the powder as it hits the target and fuses the powder to material already deposited. The end product is a near-net shaped part with mechanical properties similar or equivalent to a part machined from wrought product. [5] If successfully utilized, this process could decrease manufacturing costs by reduction of part count, elimination of machining steps, and elimination of purchasing extra material that ends up as chips on the shop floor.

In addition, the LENS™ process may enable materials scientists to produce unique alloys via in-situ alloying and rapid solidification. A class of materials in this category is rapidly solidified, dispersion strengthened titanium alloys. A number of these materials have been studied to evaluate the possibility of increasing the operating temperature range of titanium alloys. These studies found that elevated temperature tensile properties and creep performance of the dispersion strengthened materials was superior to those of the base materials. [6, 7]

One such titanium alloy in this class of materials is Ti-8Al-1Er, which has a nominal composition of 8 wt% Al and 1 wt% Er. During fabrication by the LENS™ process, the erbium chemically combines with oxygen interstitial atoms to form Er_2O_3 precipitates. The resulting product is a rapidly solidified alloy in which the aluminum is a solid solution strengthener, the rapid solidification produces a fine acicular α' martensitic microstructure, and the erbium getters the available oxygen to form fine submicron size precipitates for strengthening and creep resistance purposes while reducing the interstitial O_2 content to minimize the chance of embrittlement by the α_2 phase. The very low solubility and diffusivity of Er_2O_3 particles in α titanium result in a stable distribution of strengthening particles that resist coarsening up to temperatures in excess of 700^o C. [8]

Based on the acicular α' microstructure, for optimum creep resistance, and the stability of the Er_2O_3 precipitates at elevated temperatures, it is believed that direct laser deposited Ti-8Al-1Er could be a good candidate for aircraft parts that operate at elevated temperatures up to 450^o C and above. The purpose of the study described in this paper was to characterize the elevated temperature tensile and creep properties of direct laser deposited Ti-8Al-1Er. These properties are compared to those for Ti-6Al-4V, the most common titanium alloy used in aircraft and specified for use up to 315^o C [9], and Ti-6242, the most common high temperature titanium alloy used in jet engine compressors manufactured in the US and specified for use up to 540^o C. [10]

Background

A practical method of designing for creep involves generating creep test results at relatively short times, high stresses and high temperatures. The test data can then be extrapolated to the time-temperature-stress region of interest using a parametric relationship that describes the creep behavior in terms of the variables of interest. Several parametric relationships for creep have been suggested in the literature. The Larson-Miller parameter is among the most well-known of these parametric relationships used for creep characterization of materials. [11-13]

Use of the Larson-Miller parameter is based on the observation that creep is a thermally activated process and that the creep rate can be described by an Arrhenius type equation of the following form.

$$\frac{1}{t} = Ae^{-Q/RT} \qquad (1)$$

where: t = time to a specified creep strain or to final rupture
Q = the activation energy for the creep process
R = the universal gas constant
T = the absolute temperature
A = an empirical constant

Rearrangement of Eqn (1) results in the following relationship.

$$Q/R = T(C + \log t) \qquad (2)$$

or

$$P_{LM} = T(C + \log t) \qquad (3)$$

where C is a constant related to the constant A in Equation 1. *Q/R* is often written as P_{LM}, as in Equation 3, and is termed the Larson-Miller parameter. Larson and Miller made the assumptions that P_{LM} is a function of stress, but independent of temperature, and that the constant C is independent of stress and temperature. Empirical evidence has shown that these assumptions are generally valid for many commercial alloys if the creep deformation mechanism remains constant and there are no phase transformations between the test temperature and the temperature of interest. It has also been shown that P_{LM} is a function of creep strain. Values of P_{LM} and C may be obtained by plotting log *t* vs. 1/T and fitting the curve by using a linear regression process.

The Larson-Miller parameter can also be used as a figure of merit for the creep resistance of materials with the came value of C. The larger the Larson-Miller parameter, the greater is the creep resistance of the material. Experimentally determined values of C for metals generally range between 17 and 21. Much of the data found in design handbooks and material property references fix the value of C at 20 to facilitate comparisons. Also, once the values of P_{LM} and C are known, they can be used in conjunction with Equation 3 to predict times to attain given creep strains or to rupture at a temperature of interest, or to predict the temperature at which a given creep strain will be attained in a given time.

Experimental Procedure

Material

Direct laser deposited Ti-8Al-1Er material was supplied in an annealed condition, 700^{o} C for 2 hours, by Lockheed Martin in the form of flat dog-bone shaped test specimens. These specimens were fabricated by depositing the titanium alloy to form a "test specimen" approximately 2.5" thick, followed by a wire EDM process that cut this piece into eighteen 0.125" thick slices to be used as the actual coupons for testing. The fillet radii in the as-received coupons were smaller than ASTM test specification recommendations, hence the fillet radii were subsequently machined to a larger radius to meet ASTM specifications.

Microstructural observation of the as-received material showed a predominately acicular α' martensitic structure, separated by thin, parallel regions of α phase about 10-20 µm thick. These thin α regions were parallel to the direction of buildup in the part. Occasional equiaxed α grains were observed interspersed throughout the microstructure. These features are shown in Figure 2. Sparsely distributed angular shaped lack-of-fusion pores were also observed, as illustrated in Figure 3.

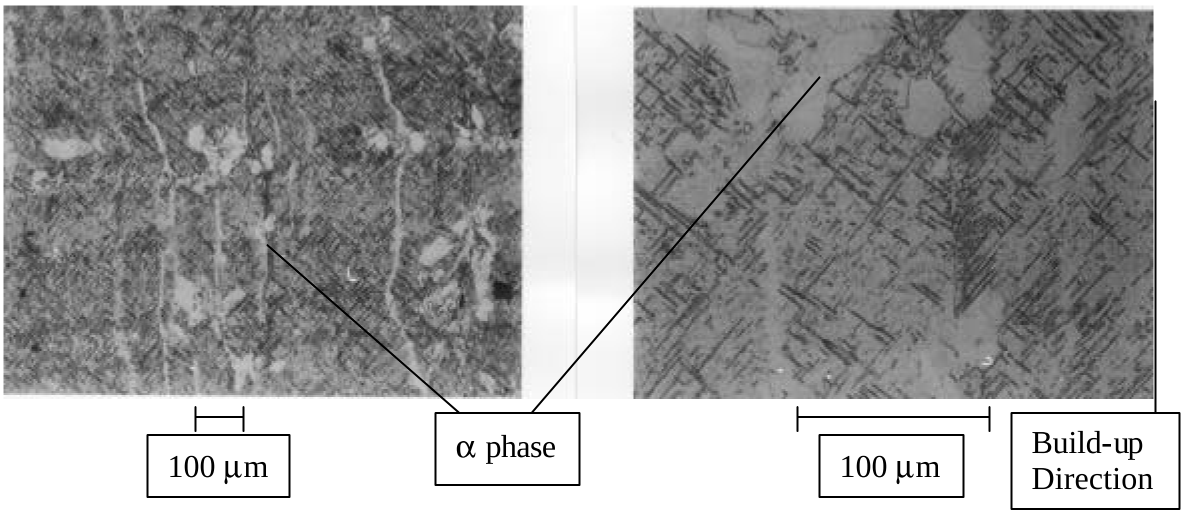

Figure 2: Microstructure of As-received Ti-8Al-1Er

Room Temperature Tensile Tests

Two room temperature tensile tests were performed to establish a baseline for which to compare the elevated temperature tensile test results. The room temperature tests were run in accordance with ASTM test standard E8-99.

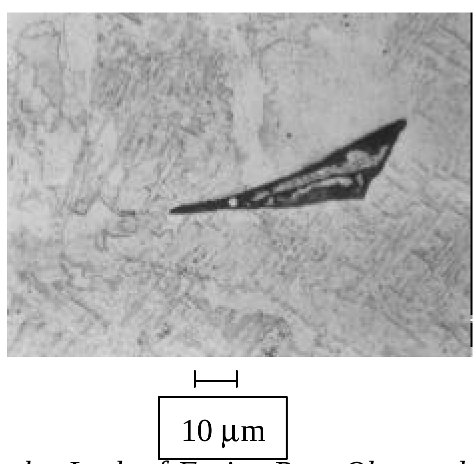

Figure 3: Angular Lack-of-Fusion Pore Observed in As-received Ti-8Al-1Er

Elevated Temperature Tensile Tests

Elevated temperature tensile tests were performed at seven temperatures ranging from 93 to 538^{o} C. Since this study was an exploratory testing survey, only a single specimen was tested at each temperature. Each specimen was held at temperature for 10-15 minutes hour before beginning the test.

Heat was applied to the specimen through the use of two sets of quartz lamp heaters placed approximately 0.5" on either side of the specimen. Each heater contained four lamps individually controlled by its own temperature controller. Four thermocouples were spot welded to each specimen to act as feedback for the controllers. The width of each quartz lamp fixture was approximately 2". Therefore the center 1" of the gage length was the only portion of the specimen held at the specified test temperature. A 0.5" gage length elevated temperature extensometer with ceramic rods was used to measure strain. All tests were run in stroke control at a rate of 0.0005"/second.

ASTM Test Method E21-92 was used as a guideline to perform these tests.

<u>Creep Tests</u>

The creep test matrix was designed in a fashion that enabled the Larson-Miller creep parameter to be determined at two stresses. Assuming that the creep deformation mechanism does not change from that of the tests in this study, this enables the test results to be extrapolated to other test conditions and allows comparison of the performance of Ti-8Al-1Er with that of other titanium alloys found in the literature.

The creep tests were stopped once a creep strain of 0.6% was attained. Some methods of comparing the creep resistance of high temperature materials found in the literature are: the temperature at which a material attains a 0.1% creep strain in 150 hours, and the Larson-Miller parameter at values of 0.1 and 0.2% creep strain. Stopping the tests at 0.6% creep strain made efficient use of test equipment and time, yet enabled sufficient data to be gathered to make a good assessment of the small strain creep resistance of Ti-8Al-1Er as compared to commercially available titanium alloys.

Seven creep tests were performed, three each at stresses of 483 and 552 MPa , and one at a stress of 534 MPa. All tests were performed at temperatures between 445^{o} C and 478^{o} C. The first six tests were used to determine the Larson-Miller parameter as a function of creep strain and stress. The final test was used to verify whether the ensuing model could reasonable predict behavior at conditions other than those tested. Given the exploratory nature of this study, only one specimen per condition was tested. The test matrix can be found in Table I.

Table I: Creep Test Matrix

Stress (MPa)	Temp (^{o}C)
483	445
483	455
483	478
552	445
552	455
552	475
534	457

All tests were run in accordance with ASTM Test Method E139-96.

Results

Room Temperature Tensile Test

The room temperature yield strength was equal to ultimate tensile strength of the Ti-8Al-1Er material at a value of 971 MPa.   The elongation value was on the order of 0.1%.

Fractography

The fracture surface of a room temperature tensile specimen was examined using the scanning electron microscope (SEM). Three different types of features were observed on the surface. All three are indicative of brittle behavior to varying degrees.

The most common feature observed was that of a rough surface covered with shallow dimples typically 1-3 μm across. This feature is illustrated by a series of five fractographs at magnifications ranging from 50x to 14,700x in Figure 4. Notice the roughness observed at 50x, the relative flatness of the local features at 200 and 940x, and the shallow flat dimples at 5100x.

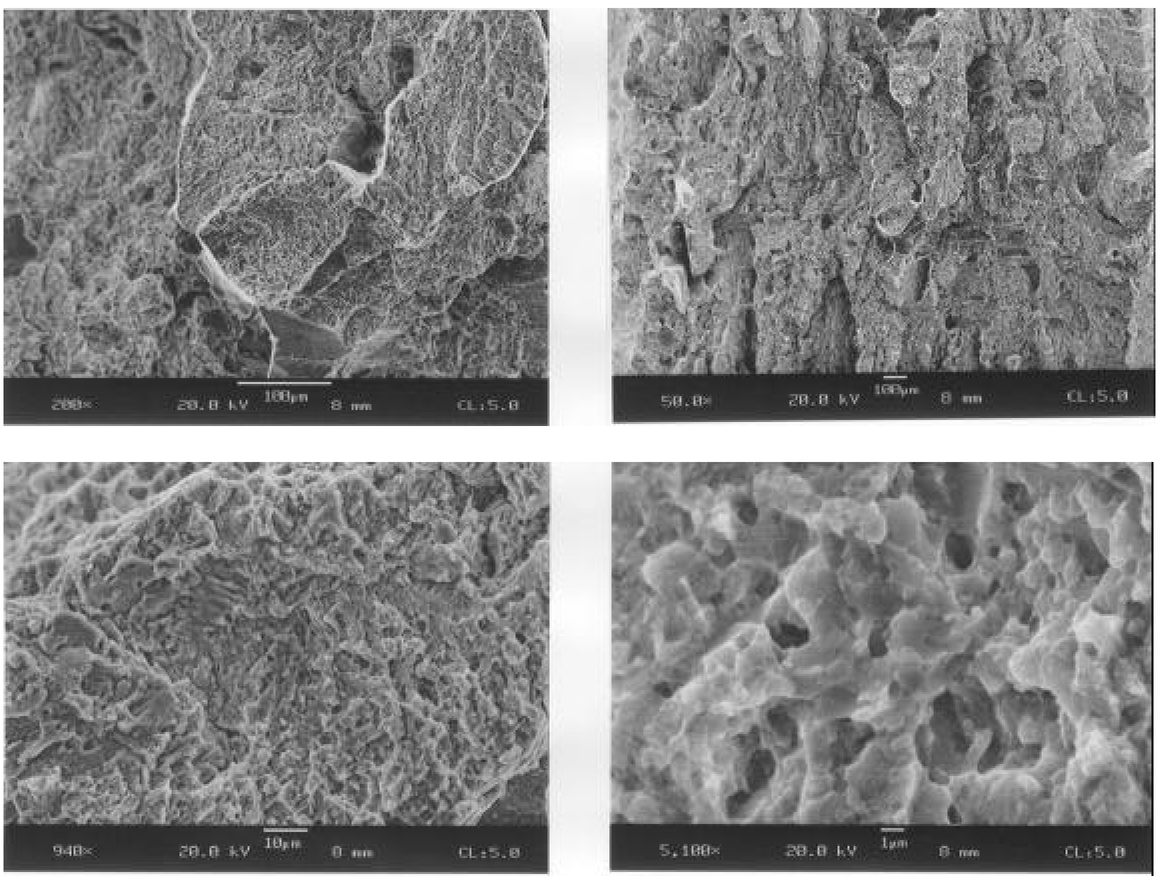

Figure 4: Most Common Feature Observed on Fracture Surface of Room Temperature Tensile Specimen. Shallow, Small Dimples.

The second feature noted was that of flat, cleavage type and intergranular failure. These features are illustrated in a series of photographs in Figure 5.

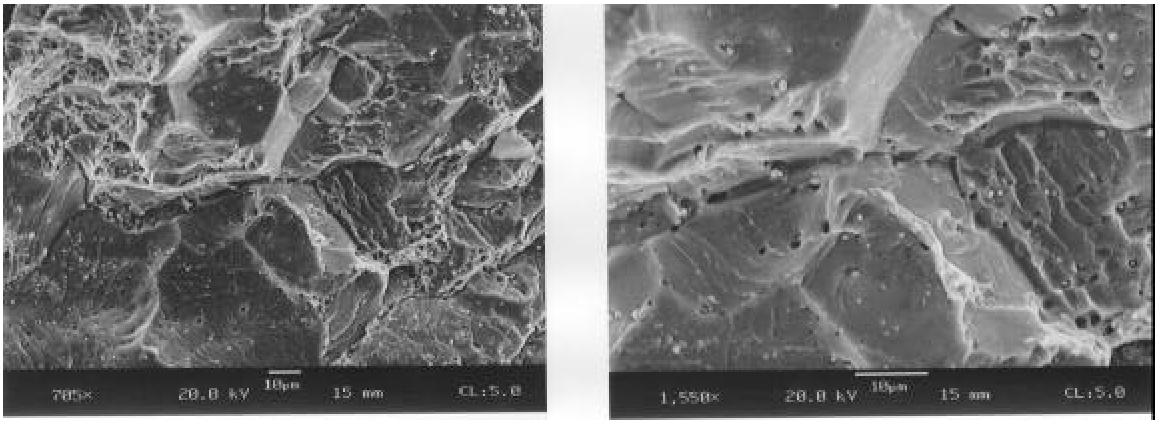

Figure 5: Cleavage Facets Observed on Fracture Surface of Room Temperature Tensile Specimen.

The third observed feature was similar to the first, but on a finer scale. These features are shown in a series of photographs in Figure 6. The roughness of these areas was much less than that shown in Figure 4. The shallow micropores could only be observed at magnifications of about 5000x and greater. These pores were generally smaller than 1 µm in size and contained submicron size particles at the bottom. It is believed that these submicron size particles are the erbium oxide precipitates. These features are sometimes associated with intergranular failure where grain boundaries are embrittled by a tramp chemical species.

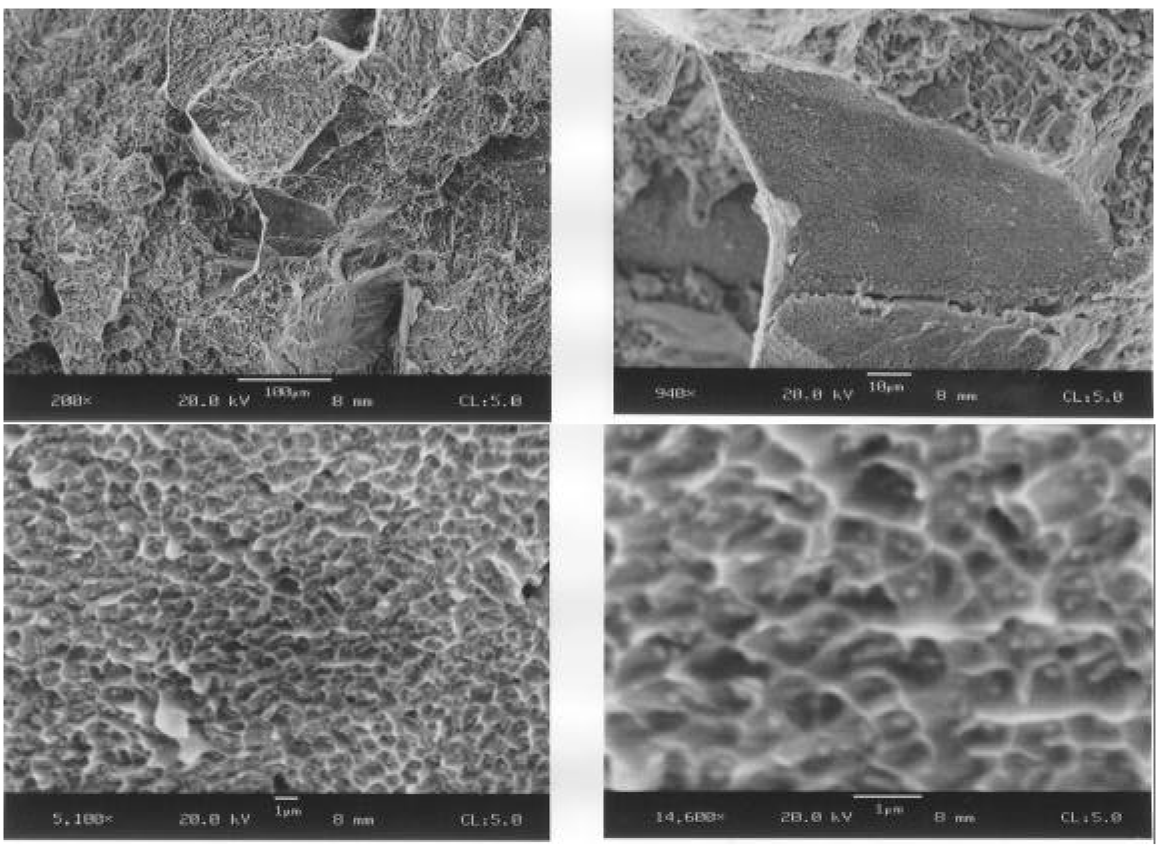

Figure 6: Shallow Dimples on Grain Boundaries. This Feature is Indicative of Brittle Behavior. Note Submicron Size Particles at Bottom of Dimples at 14,600x Magnification.

Elevated Temperature Tensile Tests

Reduction of area rather than elongation was used as a measure of ductility for two reasons. First, the low ductility at room temperature indicated that the stress concentrations at impressions used for measuring elongation may have caused premature failure. Second, only the central 25.4 mm of the gauge length was at the indicated temperature. Therefore use of the standard 50.8 mm gauge length used to measure elongation would have sampled an area where the temperature varied significantly.

The elevated temperature tensile test results are presented in two ways. The first is the absolute values of strength as a function of temperature. The second is the percentage of retained room temperature strength as a function of temperature. This latter method normalizes the results and cancels out any effect of a comparison to other alloys when the room temperature strengths are different.

Absolute values of yield and tensile strengths at elevated temperatures for Ti-8Al-1Er are shown in Figure 7 and tabulated in Table II. Reduction of area values are also shown in Table II. As a means of comparison, reduction of area values reported for Ti-6Al-4V and Ti-6242 at room temperature usually range from 20 to 35%. Also shown in Figure 7 is a comparison of the Ti-8Al-1Er strengths to those reported in the literature for Ti-6Al-4V and Ti-6242. It can be seen from this figure that Ti-8Al-1Er is superior to Ti-6Al-4V in retaining its strength at elevated temperatures. The elevated temperature strength retention capabilities of Ti-8Al-1Er and Ti-6242 are comparable.

Table II: Elevated Temperature Tensile Test Results for Ti-8Al-1Er

Test Temperature (^{o} C)	Yield Strength (MPa)	% of RT Yield Strength	Tensile Strength (MPa)	% of RT Tensile Strength	Reduction of Area (%)
93	931	96	958	99	0
204	793	82	896	92	0.5
260	745	77	848	87	3.5
316	717	74	814	84	5.5
427	669	69	758	78	8.0
482	607	62	724	74	10.5
538	579	60	669	69	13.5

Figure 8 illustrates the percentage of retained room temperature strength as a function of temperature for Ti-8Al-1Er and also compares the performance of the experimental alloy to that of Ti-6Al-4V and Ti-6242. As was the case for the absolute values of strength, the strength retention capabilities of the two alpha alloys, Ti-8Al-1Er and Ti-6242, are comparable and are superior to that of the α–β alloy, Ti-6Al-4V.

Visual inspection of the failed test specimens indicated that the specimens tested up to 260^o C failed at the spot welds affixing the thermocouples to the specimen. At temperatures of 316^o C and above the specimens failed at locations away from the spot welds.

Creep Tests

Creep test results in the form of creep strain vs. time plots are shown in Figure 9. The plot shows that the time to a given creep strain increases with decreasing temperature and stress.

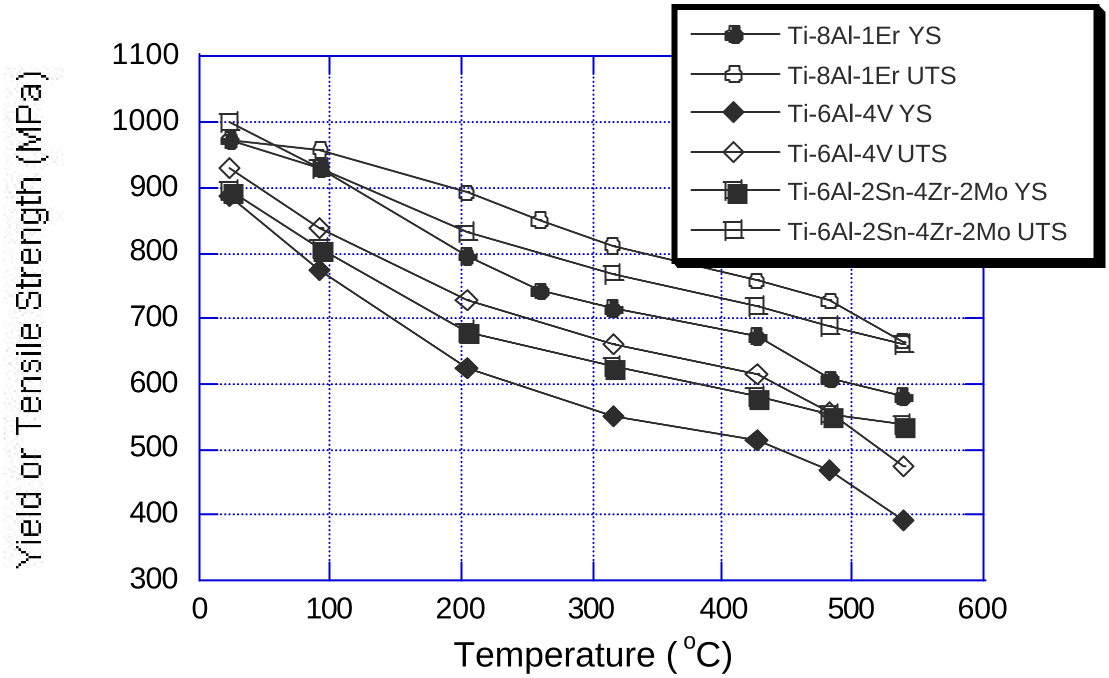

Figure 7: Elevated Temperature Tensile Property Comparison of Ti-6Al-4V, Ti-8Al-1Er, and Ti-6Al-2Sn-4Zr-2Mo

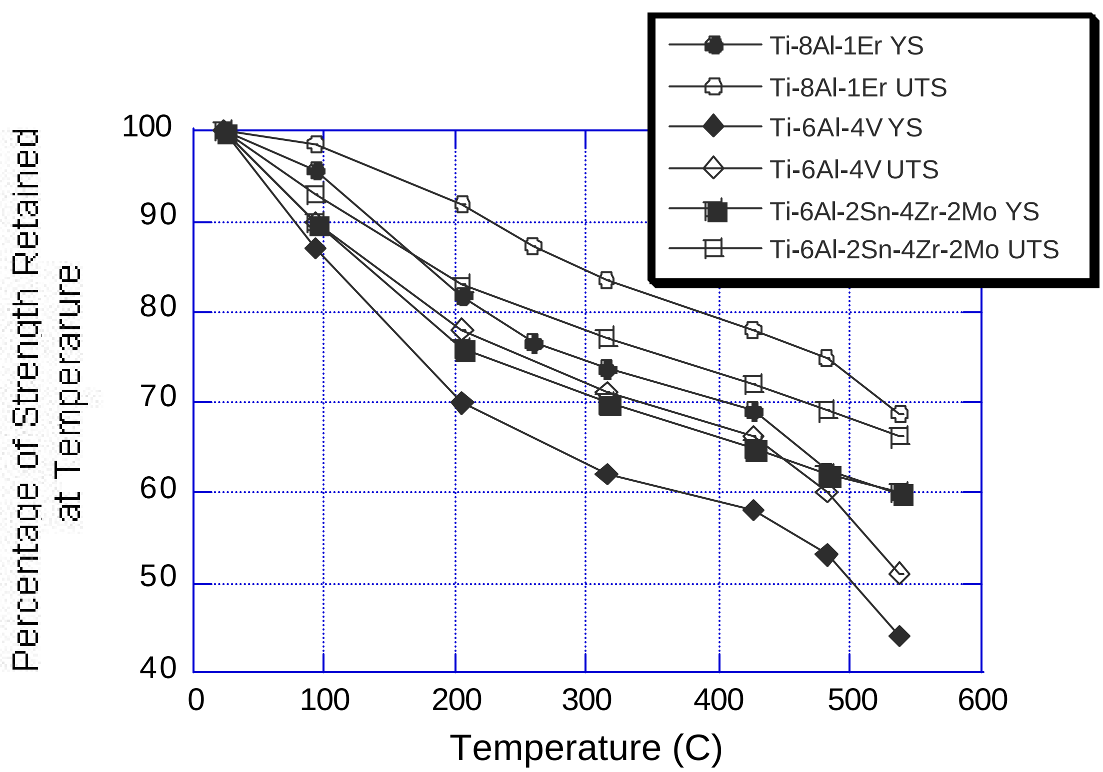

Figure 8: Percentage of Room Temperature Strength retained at Elevated Temperature for Ti-6Al-4V, Ti-8Al-1Er, and Ti-6Al-2Sn-4Zr-2Mo.

A comparison of times to achieve three specific creep strains at a temperature of 455^o C and stresses of 483 and 552 MPa for Ti-6Al-4V and Ti-8Al-1Er can be found in Tables III and IV. The values in these tables indicate that the times for Ti-8Al-1Er to attain a given creep strain are 7 to 75 times greater than that required for Ti-6Al-4V.

Larson–Miller parameters for titanium alloys found in the literature are typically plotted with C values of 20. Therefore, the Larson-Miller parameters calculated in this study were fit by forcing C to be equal to 20, to facilitate comparison to published data for material ranking purposes. A plot of the Larson Miller parameters as a function of creep strain and stress for Ti-8Al-1Er is shown in Figure 10.

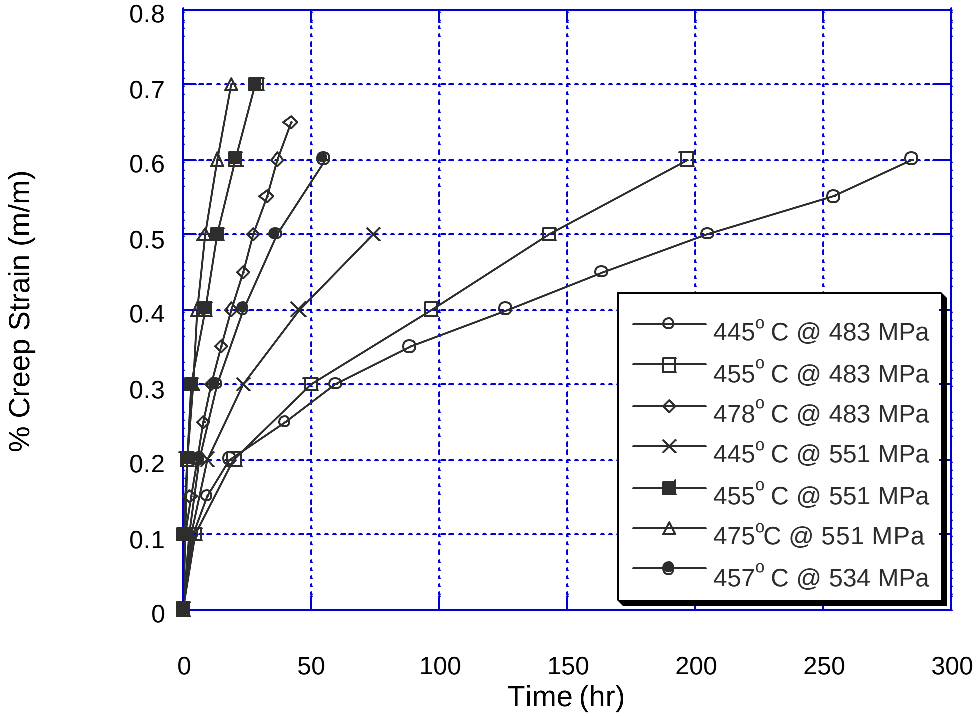

Figure 9: Creep Strain vs. Time as a Function of Test Temperature and Applied Stress. Ti-8Al-1Er

Table III. Comparison of Time to Attain Given Creep Strains for Ti-6Al-4V and Ti-8Al- 1Er @ 552 MPa and 455° C.

% strain	Ti-6Al-4V (hr)	Ti-8Al-1Er (hr)
0.1	0.06	0.4
0.2	0.2	1.5
0.5	0.6	13.6

Table IV. Comparison of Time to Attain Given Creep Strains for Ti-6Al-4V and Ti-8Al- 1Er @ 483 MPa and 455° C.

% strain	Ti-6Al-4V (hr)	Ti-8Al-1Er (hr)
0.1	.2	4.8
0.2	0.6	20
0.5	1.9	143

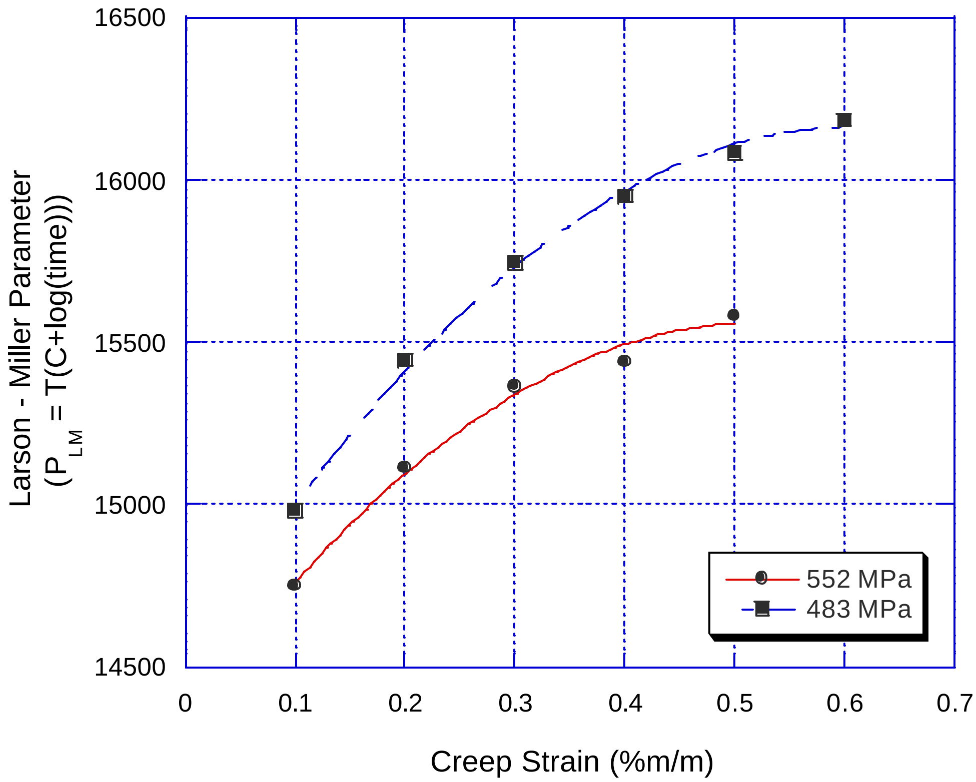

Figure 10: Larson-Miller Parameter as a Function of Stress and Creep Strain
Ti-8Al-1Er

At a creep strain of 0.2% and stress of 483 MPa, P_{LM} of β processed Ti-6Al-4V is 14500 or less [14-16], and that for Ti-6242 ranges from approximately 15200 to 15800 [15, 17]. At 0.1% creep strain and 483 MPa stress the P_{LM} for Ti-6242 ranges from approximately 14300 to15300 [14, 17]. Comparing these values to those in Figure 10 for Ti-8Al-1Er and plugging these numbers into Equation. 3 indicates that at 483 MPa the time to attain creep strains of 0.1 or 0.2% for Ti-6242 is approximately equivalent to that for Ti-8Al-1Er, and the time required for Ti-8Al-1Er would be 23 to 41 times greater than for Ti-6Al-4V.

Equation 3 in conjunction with the Larson Miller parameter can also be used to compute the temperature at which a given creep strain will be attained in a given time. This was done for 0.1% creep strain in 150 hr and 0.2% creep strain in 100 hr in order to compare the results for Ti-8Al-1Er to that of other Ti alloys. This comparison is plotted in Figure 11. As was the case in comparing creep resistance by use of the Larson Miller parameter, this figure indicates that the creep resistance of Ti-8Al-1Er is substantially superior to Ti-6Al-4V, and on the same order of magnitude as Ti-6242.

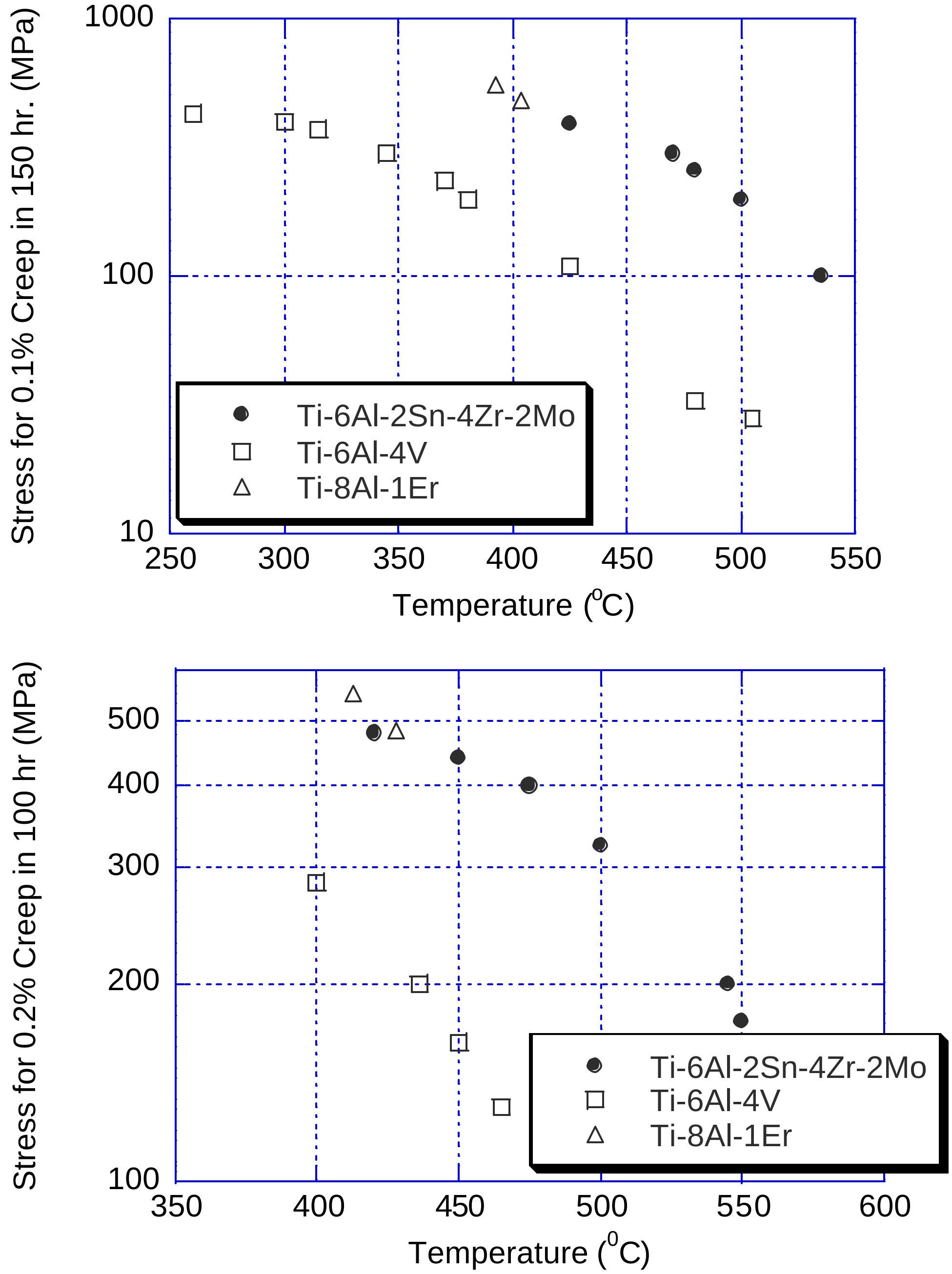

Figure 11: Temperatures at Which Creep Strains of 0.1% and 0.2% are Attained in 150 hr and 100 hr Respectively. Comparison of Ti-8Al-1Er Results to that of Other Ti Alloys. (Data for Ti-6Al-4V and Ti-6Al-2Sn-4Zr-2Mo obtained from References 14-17)

The ability of the derived Larson-Miller parameters coupled with the models of Equations 1 through 3 to predict creep behavior for this study is illustrated in Figure 12. In this figure the actual measured behavior of the coupon tested at 534 MPa and a temperature of 457^{o} C is compared to the predicted behavior. For this prediction, it was assumed that the P_{LM} values changed linearly with stress between 483 and 552 MPa. The predicted times are all within 20% of the actual measured times.

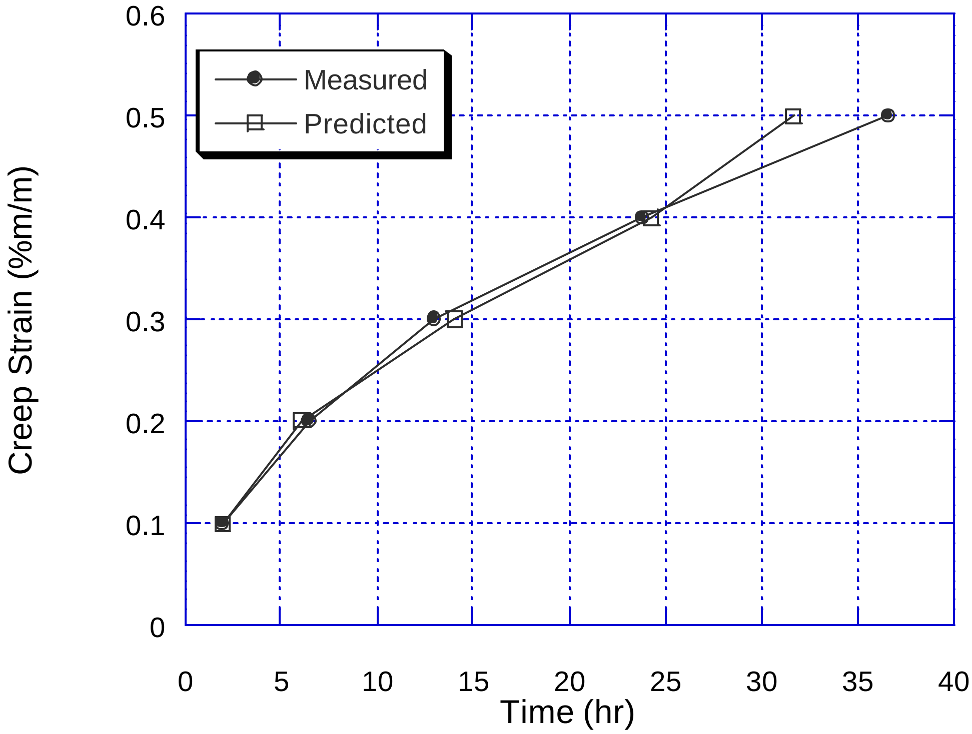

Figure 12: Comparison of Measured Creep Test Results with Predicted Results Using Larson-Miller Model
Ti-8Al-1Er; Stress – 534 MPa, Temperature - 457° C

Discussion

Based on data presented in the Results section of this report, it is apparent that the elevated temperature strength properties and creep properties of direct laser deposited Ti-8Al-1Er are superior to that of Ti-6Al-4V. The elevated temperature strength and creep resistance of this experimental alloy are similar to those of Ti-6242, the most commonly used high temperature "superalpha" alloy used in jet engine compresser sections. However, the ductility of the new material, as is, is too low to be seriously considered for engineering applications.

These results are consistent with microstructure-property relationships observed in titanium alloys. Titanium alloys with acicular α microstructures are typically observed to exhibit the best long term elevated temperature properties. The addition of the Er_2O_3 precipitates should also serve to increase elevated temperature tensile properties and provide additional resistance to dislocation power law creep. This is consistent with methods used to improve the creep resistance of nickel-based superalloys, where it is found that increasing the volume percentage of γ' results in improved creep resistance.

The cause for the low ductility of this product is not clear. However, the features observed on the fracture surfaces suggest that there might be some grain boundary contamination that is embrittling the grain boundaries. Other possibilities might be an excess of gaseous interstitials, formation of ordered α_2 phase (Ti_3Al) precipitates (a phenomenon known to be associated with embrittlement of some Ti alloys with Al contents greater than 6 wt% and in the presence of elevated O_2 levels), [18] or strain localization due to a heterogeneous distribution of precipitates.

Further studies of this material would be warranted to see if the ductility can be improved. It should also be noted that this study was exploratory in nature and that the comparisons of this material to other commercially available alloys is based on sparse data with little statistical validity. By performing further tests it would be possible to more accurately define the behavior of this material and investigate processing routes that could result in improved properties.

Conclusions

Direct laser deposited Ti-8Al-1Er exhibits:

1) Equivalent room temperature strength values as compared to Ti-6Al-4V and Ti-6242.

2) Inferior room temperature ductility of less than 1% elongation.

3) Superior elevated temperature strengths and creep resistance when compared to Ti-6Al-4V

4) Comparable elevated temperature strength values and creep resistance as compared to Ti-6242.

5) Further work would be justified to improve the ductility of this product through process improvements in order to take advantage of lower processing costs associated with direct laser deposition compared to typical fabrication routes.

Acknowledgements

"The views expressed are those of the author and do not reflect the official policy or position of the US Air Force, Department of Defense or the US Government."

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