Electron Beam Freeform Fabrication of Titanium Alloy Gradient Structures

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July 2014
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Abstract

Historically, the structural optimization of aerospace components has been done through geometric methods. A monolithic material is chosen based on the best compromise between the competing design limiting criteria. Then the structure is geometrically optimized to give the best overall performance using the single material chosen. Functionally graded materials offer the potential to further improve structural efficiency by allowing the material composition and/or microstructural features to spatially vary within a single structure. Thus, local properties could be tailored to the local design limiting criteria. Additive manufacturing techniques enable the fabrication of such graded materials and structures. This paper presents the results of a graded material study using two titanium alloys processed using electron beam freeform fabrication, an additive manufacturing process. The results show that the two alloys uniformly mix at various ratios and the resultant static tensile properties of the mixed alloys behave according to rule-of-mixtures. Additionally, the crack growth behavior across an abrupt change from one alloy to the other shows no discontinuity and the crack smoothly transitions from one crack growth regime into another.

Introduction

Electron beam freeform fabrication (EBF\textsuperscript{3}) is an emerging additive manufacturing process that utilizes an electron beam heat source with alloy wire feedstock to create a fully dense, three-dimensional metal structure directly from a computer model [1]. A schematic of the process is shown in Figure 1. The computer model of the part to be fabricated is first sliced into discrete layers and rows and a tool path is generated that can recreate the part from these individual layers and rows. The electron beam is then used to form a molten pool on a substrate plate into which a feedstock wire is fed, thus creating a bead of deposited material. By following the tool path of the deconstructed 3-D object, the EBF\textsuperscript{3} machine can deposit sequential beads and layers in order to create the part in a freeform manner.

A particular advantage of the EBF\textsuperscript{3} process is the ability to create functional gradient structures through the use of multiple feedstock wires. Thus, within a single structure the chemical composition can be locally changed. This could be advantageous for optimizing properties within a given structure based on the local design limiting criteria [2]. For example, areas of high toughness could be smoothly transitioned into areas of high strength with the transition determined by the loading conditions within the part. This could greatly increase the structural efficiency of complex loaded aerospace components.

Two titanium alloys were chosen for demonstration of this gradient approach: commercially-pure (CP) grade 2 titanium and titanium with 8% aluminum and 1% erbium (by weight; wt%). Grade 2 titanium is a commonly-used CP variant characterized by low yield strength (275-410 MPa), but with excellent weldability and corrosion resistance. For titanium alloys, aluminum is used as a solid-solution strengthener up to about 8 wt%. For aluminum content greater than 8 wt% strength begins to suffer as an intermetallic compound is formed. The addition of erbium forms an dispersion of oxide nano-particles that also acts as a strengthener. Erbium is not typically alloyed with titanium because the formation of strengthening nano-dispersoids (Er\textsubscript{2}O\textsubscript{3})
requires some level of rapid solidification, which standard ingot production does not offer. Alloying titanium with erbium is well suited for EBF\textsuperscript{3} processing as it involves high-cooling rates and rapid solidification.

These materials (CP titanium and Ti-8Al-1Er) were chosen for a number of reasons. First, they are chemically compatible in that there are no brittle intermetallic phases expected over the full range of possible mixtures and processing temperatures. Second, they have vastly different static mechanical properties. Finally, the mixtures are easy to quantify by measuring aluminum concentration (in solid solution) and also by measuring particle density of Er\textsubscript{2}O\textsubscript{3} dispersoids. This will give an indication of the diffusion-based mixing effects (aluminum in solid solution) and convection-based mixing effects (Er\textsubscript{2}O\textsubscript{3} in liquid titanium).

**Experimental Results and Discussion**

**Sample Preparation**

Electron beam freeform fabrication structures were produced using titanium and titanium alloy wire feedstock deposited on grade 2 commercially-pure (CP) titanium plate substrates. The deposited material was created using grade 2 CP-Ti wire, Ti-8Al-1Er wire, or a combination of the two wires. Constant ratio mixtures were created using fixed feed rates for each of the two wires. A graded composition deposit was created by changing feedstock wire halfway through each layer (i.e., five rows of CP-Ti followed by five rows of Ti-8Al-1Er). As there is about 33% overlap between successive rows, there is some degree of mixing between the two alloys as the wire feedstock is changed. This creates a gradient in composition near the interface between the deposited materials. Mechanical testing was conducted on EBF\textsuperscript{3} titanium monolithic and graded materials, including tensile testing, micro-hardness testing, and fatigue crack growth testing.

**Tensile Testing**

A total of 10 single wall deposits were made for tensile testing. Each deposited wall was approximately 250 mm long and 14 mm high. A 973 K, 2 hour vacuum anneal heat treatment was performed on all the deposited walls prior to machining. From each wall, two flat dog-bone tensile test specimens were extracted using wire electro-discharge machining (EDM). The specimens had gage section dimensions of 32 mm long by 6.4 mm wide by 3.2 mm thick. Five different compositions were tested: 100% of each starting wire composition and mixed volumetric ratios of 25:75, 50:50, and 75:25. Four specimens were fabricated with each composition. Tensile testing was performed according to the ASTM Standard E8 [3].

The mechanical properties from tensile testing of the EBF\textsuperscript{3} products are shown in Figures 2 and 3 as a function of composition. The results shown in Figure 2 indicate a slight increase in elastic modulus (12%) as the composition varies from CP-Ti to Ti-8Al-1Er. This composition change also results in a reduction of elongation at failure (ductility) from approximately 50% to 10%. The data in Figure 3 show an increase in yield stress from approximately 240 MPa to 740 MPa due to the addition of the alloying elements (Al and Er). The strength increase can mainly be attributed to solid solution effects for aluminum and dispersoid effects for erbium. The trend is linear with respect to composition implying a rule-of-mixtures effect. The variation in ultimate
Tensile strength is similar in that it increases nearly linearly with increasing alloying elements, and increased from approximately 340 MPa (CP-Ti) to 810 MPa (Ti-8Al-1Er).

**Microhardness Testing**

A single layer deposited sample containing five rows of 100% CP-Ti with a step change to five rows of 100% Ti-8Al-1Er was used to evaluate the interface transition. A cross-section cut was made in this deposit and micro-hardness measurements (Vickers indenter with 300g force) were made at constant intervals across the entire deposit. The micro-hardness data are plotted in Figure 4. The hardness values in the CP-Ti deposit are approximately 140 VHN_{300gf}. A sudden increase in micro-hardness occurs at the interface, increasing from approximately 140 VHN_{300gf} to 240 VHN_{300gf} over a distance of about 1 mm. In the Ti-8Al-1Er deposit, the hardness increased slightly with distance from the interface (increasing from about 240 VHN_{300gf} to 290 VHN_{300gf} over a distance of about 8mm). This is attributable to the diminishing effects of dilution that occurs as successive rows are deposited further from the interface.

**Fatigue Crack Growth Testing**

Deposited structures of approximately 150 mm by 63 mm dimensions, as shown in Figure 5, were fabricated for fatigue crack growth testing. Within each structure, the deposited rows were approximately 6 mm wide and were oriented parallel to the loading axis of the crack growth specimens (see Figure 6, right side). The structures contained a total of 20 rows in each layer with five total layers per sample. A 973 K, 2 hour vacuum anneal heat treatment was performed on all samples prior to machining. Eccentrically-loaded single-edge notch tension (ESE(T)) fatigue crack growth specimens were machined from the EBF^3 product forms. A schematic of the ESE(T) specimen configuration is shown in Figure 6 (left side) [4]. As seen in the photograph in Figure 6 (right side), the loading direction is parallel to the direction EBF^3 material was deposited and the crack is expected to grow across (perpendicular to) the EBF^3 deposited rows.

Fatigue crack growth testing was performed in accordance with the ASTM Standard E647 [5]. During testing, crack length was monitored using a crack-mouth-opening displacement (CMOD) gauge and applied loads were continuously changed such that pre-determined values of the crack-tip stress intensity range (ΔK) were achieved [6]. Testing was conducted in room temperature laboratory air at a cyclic loading rate of 5 Hz and an R value of 0.1. Constant-ΔK testing was performed in this study to look for subtle changes in crack growth behavior as the crack propagated through the EBF^3 deposited material [7, 8]. Maintaining constant ΔK is useful should the crack growth resistance vary as crack propagation occurs across boundaries between the EBF^3 deposited rows.

Constant-ΔK test data is best plotted as crack length (a) versus cycle count (N). This data is presented in Figure 7 for a monolithic CP titanium specimen and a monolithic Ti-8Al-1Er specimen. Note that the slope of these data is the fatigue crack growth rate (da/dN). A fitted line through these data is used to determine the crack growth rate for ΔK = 7.9 MPa\(\sqrt{m}\) (R = 0.1), for CP-Ti (blue triangular symbols) and for Ti-8Al-1Er (red square symbols). Testing was conducted at room temperature in laboratory air at a cyclic loading rate of 5 Hz. From these
data, it can be seen that for a cyclic crack-tip stress intensity factor range of $\Delta K = 7.9 \text{ MPa}\sqrt{\text{m}}$ that the CP-Ti material exhibits crack growth rates approximately twice that of the Ti-8Al-1Er alloy ($\frac{da}{dN} = 3.55 \times 10^{-9} \text{ m/cycle}$ compared with $\frac{da}{dN} = 1.75 \times 10^{-9} \text{ m/cycle}$, respectively). Further, the crack growth path did not exhibit any deviation that would have been expected if the boundaries between EBF3 deposition passes were relatively weak in fatigue crack growth resistance. This observation suggests that the material produced is rather homogenous in terms of fatigue crack growth performance.

The fatigue crack growth data for the graded specimen are plotted in Figure 8. This specimen was tested at a constant $\Delta K$ value of 9.9 MPa$\sqrt{\text{m}}$. Steady state crack growth data in the Ti-8Al-1Er material ($\frac{da}{dN} = 4.15 \times 10^{-7} \text{ m/cycle}$), indicated as red square symbols, was shown to occur between crack lengths of approximately 6 mm to 12 mm. Around a crack length of 12 mm, the crack began to propagate into the transition region between the two materials; these transient data are indicated as open circular symbols. Upon propagating into the CP-Ti region, steady state crack growth was re-established (blue triangular symbols; $\frac{da}{dN} = 8.65 \times 10^{-7} \text{ m/cycle}$) over a range in crack length between approximately 14 mm to 20 mm. Note that the earlier observation that the crack growth rates in CP-Ti were nearly a factor of 2 greater than for the Ti-8Al-1Er alloy (recall Figure 6; $\Delta K = 7.9 \text{ MPa}\sqrt{\text{m}}$) also is true at higher $\Delta K (= 9.9 \text{ MPa}\sqrt{\text{m}})$.

Photographs of the crack profiles are shown in Figure 9. Fatigue cracks can be seen originating at the crack starter notches on the left side of each photograph. The crack profile of the CP-Ti (Figure 9a) exhibited a few “jogs” where the crack briefly deviated from the direction perpendicular to the applied load. Two such jogs are shown in the red circle of Figure 9a. This seemed to occur along the boundaries between e-beam deposition rows, however, this did not produce any anomalous fatigue crack growth behavior. In comparison, the crack path of the Ti-8Al-1Er alloy, shown in Figure 9b, seems to be somewhat less tortuous and did not exhibit any jogs in the crack path for the specimen tested. A photograph of the crack path in a specimen with a gradient from Ti-8Al-1Er to CP-Ti is shown in Figure 9c; note that the Ti-8Al-1Er region was better polished than the CP-Ti region despite being subjected to the same polishing procedure. Presumably this is due to the differences in the mechanical properties (e.g., hardness) between the materials in the two regions. The crack path in this specimen was straight and did not exhibit any of the jogs that were seen in the monolithic CP-titanium specimen.

The crack growth testing presented in this document was done at a load ratio (ratio of minimum to maximum load in a fatigue load cycle) of $R = 0.1$. At such low values of $R$ crack closure is expected to significantly affect fatigue crack growth rates, potentially more so for the relatively ductile CP-Ti. Further work will explore this area.

**Implications**

As with other additive manufacturing methods, EBF3 allows components to be fabricated near net shape, which significantly improves cost and lead time while reducing waste from machining operations. Further, by changing the wire product or processing parameters, EBF3 allows for development of materials with gradients in composition, microstructure, and
mechanical properties. While creating such gradients is not a new idea, further work is needed to develop a physics-based understanding of the interrelations of (1) the composition and processing parameters, with the resulting microstructure and (2) the microstructure with the resulting mechanical performance. The work presented in this document is a first step towards establishing this knowledge. Ultimately, such efforts will permit the design of advanced materials with specific mechanical performance (e.g., crack growth resistance) based on alloy composition and processing parameters. This would reduce the dependence on empirical experimental results, which would decrease the time and costs associated with qualification of new materials for use in aerospace structures.

Summary and Future Work

Electron beam freeform fabrication (EBF$^3$) Ti products were produced to show the feasibility of fabricating metallic materials with gradients in composition and mechanical properties. Materials were made with a gradient between CP-Ti and Ti-8Al-1Er. Experimental data presented in this document show that chemical gradients can be created in EBF$^3$ deposited material that result in mechanical gradients in yield stress, ultimate tensile stress, elastic modulus, elongation at failure (ductility), hardness, and fatigue crack growth rates.

There remains a good deal of experimental testing that should be conducted on the 1-D material produced for this study. To ensure that the fatigue crack growth trends observed in Figure 8 hold in the absence of crack closure, additional fatigue crack growth testing of gradient materials should be done at a higher value of R where crack closure is expected to have a negligible role (R = 0.5).

The EBF$^3$ method deposits material in rows of a finite width; in this study, the rows were approximately 6 mm wide. It is possible that periodic variations in some material properties may occur with respect to the direction normal to the plane of deposition. Although tensile testing has been done on these materials, it was done using extensometers that might fail to capture such periodic variation in strain fields. Therefore, additional testing is planned to perform tensile testing using digital image correlation method, which will provide full-field strain data rather than a single scalar value from an extensometer.

Finally, in order to use a computational approach for science-based design of structurally graded materials, an analytical model will be generated of the mechanical test response of these materials. Although the materials produced for this study had a gradient in only one direction (1-D), ultimately, the models will be generated for materials with gradients in multiple directions (2-D and fully 3-D gradients).

References


Figures

Figure 1: Schematic of the EBF$^3$ additive manufacturing process.
Figure 2: Tensile properties (elastic modulus and elongation) as a function of alloy content for EBF$_3$ deposited samples.
Figure 3: Tensile properties (ultimate and yield stress) as a function of alloy content for EBF$^3$ deposited samples.
Figure 4: Micro-hardness test data across the interface of CP-Ti to Ti-8Al-1Er alloy of EBF³ deposited sample.
Figure 5: Photograph of EBF$^3$ titanium-alloy deposit on a CP-Ti substrate.
Figure 6: Fatigue crack growth specimen details; ESE(T) specimen configuration on the left and specimen orientation with respect to EBF₃ deposit on the right.
Figure 7: Fatigue crack growth results from CP-Ti and Ti-8Al-1Er monolithic ESE(T) specimens fabricated by EBF$^3$
Figure 8: Fatigue crack growth results for gradient CP-Ti/Ti-8Al-1Er ESE(T) specimen fabricated by EBF3.
Figure 9: Photographs of crack profiles for monolithic (a) and (b), and gradient (c) ESE(T) specimens fabricated by EBF3.
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