PROPERTIES OF ADDITIVELY MANUFACTURED TITANIUM PARTS — A COMPARATIVE STUDY

Quality and performance of EIGA vs. plasma atomized titanium powder in laser-powder bed fusion components
SUMMARY

Properties of titanium parts, printed by laser-powder bed fusion: EIGA vs. plasma atomized powder

Titanium-6 aluminum-4 vanadium ELI (Ti64) is one of the most common alloys in additive manufacturing (AM) and is used in numerous applications across the aerospace and medical industries, where orthopedic applications have taken the lead in end-use applications. Additive manufacturing of orthopedic implants using Ti64 alloys has been successful across spine, hip, knee, and extremity applications due to the alloy’s inherent biocompatibility and good mechanical properties combined with additive’s ability to tailor porous structures enabling osseointegration and mass customization for better patient outcomes. The number of Food and Drug Administration (FDA) approved titanium-based AM surgical implants continues to grow as medical OEMs increasingly adopt AM in their production capabilities. In the aerospace industry, several titanium-based additively manufactured parts approved by the Federal Aviation Administration (FAA) are currently in commercial and military use, with numerous other prototypes making their way toward certification.

The standard atomization techniques to fabricate Ti64 powders for AM are Electrode Induction Inert Gas Atomization (EIGA) and plasma atomization. This work is a continuation of the previously published study on Ti64 powder equivalency, which expanded on these two atomization processes and established the advantages of EIGA powders over those produced with the plasma atomization method.

In this comparative study, test specimen parts printed by laser-powder bed fusion utilizing both EIGA and plasma atomized powders were investigated by comparing their chemical, microstructural, mechanical, and physical properties conducted within the framework of the ASTM F3001 and F3302 standards.

The results from this study show the advantages of EIGA powder over plasma atomized powder as observed from several quantitative measures. Further, at the minimum, the powder types produce statistically equivalent results when formed into consolidated AM parts and that EIGA minimizes the risk of tungsten contamination.
Quantitative measures

Oxygen measure
The oxygen measured in EIGA printed parts were lower than parts printed using plasma atomized powder. Carpenter Technology’s in-house capability to manufacture Ti64 bar feedstock, in addition to the atomization process, allows for strict quality control on chemistry and key interstitial elements in Ti64 alloy powder, enabling better properties in printed parts.

Contamination
The absence of any refractory component in the EIGA process eliminates the risk of high density contaminants, such as tungsten.

Mechanical properties
- The mechanical properties, 0.2% offset yield stress and ultimate tensile stress of parts printed using both EIGA and plasma atomized powder, were comparable and exceed ASTM F3001 requirements.
- Impact toughness of parts printed using EIGA powder were higher than those printed by plasma atomized powder, indicating superior microcrack resistance of parts printed using EIGA powder.
- Preliminary fatigue results indicate statistical equivalency of parts printed using either EIGA or plasma atomized powder.

Porosity/Microstructure
- Mean pore size in parts printed using EIGA powder were lower than those using plasma atomized powder. This was observed in both XY and YZ planes of the samples.
- Grain size, morphology and phase structure were identical in parts printed using either EIGA or plasma atomized powder.
TEST METHODS

Additively manufactured test coupons were printed in vertical and horizontal orientations on an EOS M 290 laser-powder bed fusion system by an independent, third-party service provider. The characteristics of powder used in this study were detailed in the aforementioned powder equivalency study. Printed coupons were first stress-relieved at 1050°F ± 25°F (566°C ± 4°C) for two hours on the build plate followed by an argon quench. Subsequently, half the coupons were subjected to Hot Isostatic Pressing (HIP), with conditions set at 1688°F (920°C) for 14.48 ksi ± 0.25 ksi for two hours then cooled down to a range between a minimum of 122°F (50°C) and room temperature, as per ASTM F3001 and AMS4999A specifications. The other half of the test coupons were not subjected to HIP. All the post-processing steps, including HIP, were carried out following ASTM F3001 and F3302 guidelines. For evaluation, the samples were subjected to uniaxial tensile, compression, Charpy, and fatigue testing, as well as microstructural, chemical, density, and porosity analysis. The results presented are the average of a minimum of five samples.
RESULTS

Chemical analysis

The chemical analysis of the parts printed from EIGA and plasma atomized powders were compared to ASTM F3001-14. The data show that samples produced from both powder batches meet the chemistry requirements for Extra Low Interstitial (ELI), also known as Ti64 Grade 23. The EIGA test coupons’ oxygen levels were lower than the plasma atomized samples, which is consistent with the respective oxygen levels in the original powder chemistry, explained in the powder equivalency study. Bar feedstock and atomization of metal both influence oxygen levels contained in the powdered material. Carpenter Technology’s in-house capability to manufacture bar feedstock in addition to the atomization process allows for oversight of the alloy’s chemistry at each manufacturing step, providing strict quality control on key interstitial elements in Ti64 alloy powder. Interstitial elements have a significant bearing on the mechanical properties of printed parts.

<table>
<thead>
<tr>
<th>ELEMENTS</th>
<th>MIN.</th>
<th>MAX.</th>
<th>EIGA</th>
<th>PLASMA ATOMIZED</th>
<th>TEST METHOD</th>
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<tbody>
<tr>
<td>Aluminum</td>
<td>5.5</td>
<td>6.5</td>
<td>6.1</td>
<td>6.3</td>
<td>ICP</td>
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<td>Vanadium</td>
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<td>4.5</td>
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<td>Iron</td>
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<td>Carbon</td>
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<td>Comb</td>
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<td>0.02</td>
<td>GF</td>
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<tr>
<td>Hydrogen</td>
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<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>ICP</td>
</tr>
<tr>
<td>Titanium</td>
<td>Balance</td>
<td>Balance</td>
<td>Balance</td>
<td>—</td>
<td></td>
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</tbody>
</table>

ICP = Inductively Coupled Plasma Spectrometry
GF = Gas Fusion
Comb = Combustion

OXYGEN LEVEL IN SAMPLES PRINTED WITH EIGA POWDER WERE LOWER THAN IN PARTS PRINTED WITH PLASMA ATOMIZED POWDER. THIS IS CONSISTENT WITH THE TREND IN OXYGEN LEVELS MEASURED IN THE ORIGINAL POWDER CHEMISTRY.
Mechanical testing

Uniaxial tensile testing — HIP and non-HIP machined coupons

Uniaxial tensile tests were conducted on the samples per ASTM E8 requirements. The yield and tensile stress of both the HIPed EIGA samples and plasma atomized samples exceeded ASTM F3001 requirements. The slightly lower average tensile strength values for the EIGA samples can be attributed to the lower oxygen levels in these parts since interstitial oxygen is a key strengthening element in Ti64. However, a lower oxygen level allows for increased powder reusability and the associated cost benefits of extending reuse of the powder. The non-HIP samples showed similar trends in both vertical and horizontal orientations.

The toughness, a measure of plastic deformation, of the EIGA printed samples were correspondingly higher than parts printed using plasma atomized powder. This is observed from the Charpy impact energy tests and is discussed below.

Impact energy — HIP and non-HIP Charpy samples

The impact energy of the samples was studied by Charpy test per ASTM E23. Tested at room temperature (~71°F/22°C), the impact energy of EIGA samples was higher than that of plasma atomized samples. This implies that the EIGA coupons contained fewer microcracks, or critical-sized flaws, in the microstructure introduced during the AM build process. The difference in the impact energy between EIGA and plasma atomized samples was higher in the vertical orientation than in the horizontal build direction. The superior crack resistance of EIGA printed samples correlates with the lower oxygen levels in EIGA powder, which increases ductility and impact absorption at a minor expense of tensile strength.

MECHANICAL PROPERTIES IN VERTICAL BUILD ORIENTATION: COMPARABLE YIELD STRESS AND ULTIMATE TENSILE STRESS OF PARTS PRINTED FROM EIGA AND PLASMA ATOMIZED POWDER. THE TOUGHNESS MEASURED BY IMPACT ENERGY WERE HIGHER IN EIGA PARTS AS OPPOSED TO PLASMA ATOMIZED PARTS.
MECHANICAL PROPERTIES IN HORIZONTAL BUILD ORIENTATION: COMPARABLE YIELD STRESS AND ULTIMATE TENSILE STRESS OF PARTS PRINTED FROM EIGA AND PLASMA ATOMIZED POWDER. THE TOUGHNESS MEASURED BY IMPACT ENERGY WERE HIGHER IN EIGA PARTS AS OPPOSED TO PLASMA ATOMIZED PARTS.

Fractography—non-HIP coupons

Fractography was performed on broken tensile coupons using scanning electron microscopy (SEM). The results typically show voids, dimples, and microvoid coalescence, along with a fibrous morphology indicating primarily ductile fracture mechanisms for both EIGA and plasma atomized coupons. Small, isolated regions showing microcracks and transgranular fracture were also observed in all samples, but predominantly in the plasma atomized samples. The fracture surfaces of both horizontal and vertical build samples displayed similar features, indicating the sample underwent predominantly ductile failure irrespective of build orientation. No inclusions or other anomalies were observed in fractured surfaces.

REPRESENTATIVE SCANNING ELECTRON MICROSCOPY (SEM) IMAGES OF STRESS RELIEVED, NON-HIP COUPONS OF EIGA AND PLASMA ATOMIZED TENSILE FRACTURED SAMPLES.
Compressive stress—HIP and non-HIP coupons

The compressive yield strength of the EIGA samples was slightly lower than the plasma atomized samples. This is due to the lower oxygen levels in the EIGA powder chemistry.

Fatigue testing—HIP and non-HIP samples

Preliminary fatigue results show comparable fatigue thresholds for both EIGA and plasma atomized samples. The fatigue samples were machined from as-printed bars in both HIP and non-HIP conditions. The samples were tested at a frequency of 30 Hz, with R (defined as the ratio of minimum/maximum peak stress) set at 0.1. The run-out was set at 2M cycles. In the non-HIP conditions, the samples failed earlier by around two orders of magnitude at comparable stress levels for both the EIGA and plasma atomized parts. This supports our recommendation to use HIP as a standard post-processing step for fatigue-critical components.
Microstructural analysis — HIP samples

Microstructural analysis of both the EIGA and plasma atomized samples did not reveal any significant difference in texture, grain size, phase morphology, or porosity. This consistency was observed in both the HIP and non-HIP samples. The mean porosity area measured using microscopy analysis from around 800 data points on unetched cross-sections showed the mean porosity area fraction was lower for EIGA printed samples compared to plasma atomized samples. This was observed in both XY and YZ planes. The maximum porosity area fraction of the EIGA printed samples was lower than the plasma atomized printed samples in the YZ plane and slightly higher than plasma atomized printed samples in the XY plane. Further, both the EIGA and plasma atomized printed parts showed outliers in the porosity measured along the XY plane.
CONCLUSIONS

Additive manufacturing of Ti64 for advanced prototyping and mass production applications continues to increase across end-use markets, predominantly in the medical and aerospace sectors. The results demonstrated broad equivalency and consistency between the two feedstock powders after comparing the properties of parts printed from both EIGA and plasma atomized feedstock.

i. Chemistry: The parts printed from EIGA and plasma atomized powders both met ASTM F3001 compositional requirements. EIGA powders tend to have lower oxygen content despite a finer-skewed particle size distribution.

ii. Contamination: The absence of refractory material in EIGA process eliminates the concern regarding high density inclusions such as tungsten.

iii. Mechanical: EIGA and plasma atomized parts showed comparable tensile and compressive properties exceeding ASTM F3001 requirements. Impact toughness testing revealed that parts printed from EIGA powders have superior impact energy compared to plasma atomized parts. This implies the EIGA parts have superior (micro)crack resistance during the AM build process. Further, preliminary cyclic loading/fatigue testing demonstrated statistical equivalency between the EIGA and plasma atomized parts in both the HIP and non-HIP conditions.

iv. Microstructural: The mean porosity of the HIP’ed samples printed using EIGA powder was lower than those printed using plasma atomized powder. No discernible difference in morphology, grain size, or phase structure was witnessed between either of the samples.

In summary, EIGA powders offer several advantages over plasma atomized powder. Manufacturers can use the results from these equivalency studies that establish EIGA powder and parts to be a viable option to reduce costs while maintaining or improving the quality of printed parts, along with an economical and reliable supply chain of high-quality titanium powder.

<table>
<thead>
<tr>
<th>CONDITION</th>
<th>DIRECTION</th>
<th>YIELD STRENGTH ksi</th>
<th>ULTIMATE TENSILE STRENGTH ksi</th>
<th>ELONGATION %</th>
<th>REDUCTION OF AREA %</th>
<th>IMPACT ENERGY ft-lbs</th>
<th>MEAN POROSITY area %</th>
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<tr>
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<td></td>
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<td>—</td>
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<tr>
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<td>0.019</td>
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