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Abstract — Electron beam melting (EBM) is an innovative additive manufacturing process in which metal powder is completely melted by a concentrated beam of electrons. In this paper, the effect of powder oxidation, internal porosity and crystallographic texture on the charpy impact energy of Ti-6Al-4V specimen fabricated using electron beam melting and hot-isostatic pressing post processing was reviewed. It was observed that the charpy impact energy dramatically decreases in a smooth but rapid fashion due to excessive powder oxidation and has a deleterious effect due to the internal porosity present in EBM Ti-6Al-4V specimen. Crystallographic texture was also found to influence Charpy absorbed energy. However, HIP post-processing significantly increases the impact toughness for specimens in each case.

Keywords — Additive Manufacturing, Electron Beam Melting, Hot-isostatic pressing, internal porosity, powder oxidation, Crystallographic Texture

I. INTRODUCTION

Electron beam melting (EBM) is an innovative additive manufacturing (AM) process in which metal powder or filament is completely melted by a concentrated beam of electrons. Production in a vacuum chamber ensures that oxidation will not deteriorate highly reactive materials like titanium. Vacuum production is also required so electrons do not collide with gas molecules. Among other additive manufacturing (AM) technologies, metallic powder bed fusion electron beam melt (EBM) has recently gained considerable attention in the medical, automotive, and aerospace communities for the fabrication of production components directly from 3D CAD files bringing enhanced design freedom, minimal material waste, and little post-processing (1).

The electron beam technology began with the experiments by physicists Hittorf and Crookes, who first tried to generate cathode rays in gases (1869) and to melt metals (1879). The heat created by electrons colliding had damaging effect and attempts were made to inhibit this by means of cooling. In 1906, physicist Marcello von Pirani first made use of this effect by building a piece of apparatus for melting tantalum powder and other metals using electron beams. In 1948, Dr. H.C. Karl-Heinz Steigerwald built the first electron beam processing machine in 1952. The initial development work was done in collaboration with Chalmers University of Technology in Gothenburg. In 1993, a patent was filed describing the principle of melting electrically conductive powder, layer by layer, with an electric beam, for manufacturing three-dimensional bodies. In 1997 Arcam AB was founded and the company continued the development on its own, with the objective to further develop and commercialize the fundamental idea behind the patent.

The EBM process involves spreading a layer of pre-alloyed metallic powder in the evacuated build space, selectively melting regions in the layer with an electron beam, spreading another layer of powder and repeating the process until a three-dimensional solid metal part is contained within the powder cake (1). A tungsten filament in the electron beam gun is superheated to create a cloud of electrons that accelerate to approximately one-half the speed of light. A magnetic field focuses the beam to the desired diameter. A second magnetic field directs the beam of electrons to the desired spot on the print bed.

Once a component or prototype has been printed, the build envelope is removed and the build platform and attached object are removed from the loose powder. Powder clinging to the object or remaining in internal cavities is blown or blasted away. Post-processing methods, including hot isostatic pressing (HIP), heat treatment in inert gas or vacuum heat treatment may be employed to release residual stresses and improve mechanical properties.

Today, the potential of electron beam melting technology is recognized and is used to print components used in aerospace, automotive, military, petrochemical and medical applications. By improving access to emerging high-growth submarkets, electron beam melting technology offers a competitive edge to progressive enterprises. In many applications, designers enjoy unprecedented design flexibility. It produces parts with properties similar to wrought parts and better than those of cast parts. For many applications, EBM is a cost-effective process that reduces inventory requirements and with build rates of almost four times those of other AM technologies. The electron beam melting process reduces residual stresses in a variety of
ways. It is possible to control residual stress during the preparation of CAD data, during printing and in post-processing. During printing, residual stress is reduced by preheating the print bed and by the heating of the material before it is struck by the electron beam. To a degree, lower preheating temperatures and slower cool-down rates compared to laser-based AM processes.

Electron beam melting requires the use of pure, unadulterated metals. Any changes in powder properties such as powder oxidation, internal porosity and crystallographic texture leads to changes in material property and hence product properties. Many researchers have investigated the effects of EBM processing parameters on material properties, with the aim of identifying the optimal parameters for near-fully-dense parts [11-14]. The EBM process has many parameters that can be altered by the user, but the EBM manufacturer provides recommended settings for Ti-6Al-4V.

II. LITERATURE REVIEW

In this paper, the effect of powder oxidation, internal porosity and crystallographic texture on the charpy impact energy of Ti-6Al-4V specimen fabricated using electron beam melting and hot-isostatic pressing post processing was reviewed. It was observed that the charpy impact energy dramatically decreases in a smooth but rapid fashion due to excessive powder oxidation and has a deleterious effect due to the internal porosity present in EBM Ti-6Al-4V specimen. Crystallographic texture was also found to influence Charpy absorbed energy. However, HIP post-processing significantly increases the impact toughness for specimens in each case.

A. Effect of powder oxidation

Powder quality in additive manufacturing (AM) electron beam melt (EBM) is crucial in determination of material properties. The effect of powder oxidation on the Charpy impact energy of Ti-6Al-4V specimen manufactured using EBM was studied by considering oxygen content significantly higher than 0.2 % mass fraction and precisely controlling the amount of oxygen as a function of time and temperature [3].

Powder samples were prepared by artificially oxidising powder in air at elevated temperature and then mixing it with low oxygen content powder. Using this method, four batches of powders were used (A) virgin powder (B) five-times reused powder (C) marginally oxidized powder, made up of a mixture of five-times reused powder mixed with artificially oxidized powder and (D) highly oxidized powder (Table 1). These powder samples were then used to fabricate Charpy specimen with the EBM process in three distinct orientations. Further, half of the blanks were post-processed using a standard hot-isostatic pressing (HIP) procedure common for Ti-6Al-4V castings and EBM produced components and then machining them into Charpy specimens and testing at room temperature. The comparison of the absorbed energies and fracture appearances was done.

<table>
<thead>
<tr>
<th>Identifier</th>
<th>Powder Mixture Description</th>
<th>Powder Oxygen Content (% wt) (± 2.5 %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>100% Virgin Powder</td>
<td>0.11</td>
</tr>
<tr>
<td>B</td>
<td>1005 5x Reused powder</td>
<td>0.142</td>
</tr>
<tr>
<td>C</td>
<td>Mixture of 96.5 % 5x reused and 3.5 % aged at 650 °C for 4 h</td>
<td>0.340</td>
</tr>
<tr>
<td>D</td>
<td>Mixture of 88.5 % 5x reused and 11.2 % aged at 650 °C for 4 h</td>
<td>0.525</td>
</tr>
</tbody>
</table>

for all test conditions. The HIP process is typically performed at α+β annealing temperatures, and thus results in a coarsened microstructure compared to the as-built condition.

B. Effect of internal porosity and crystallographic texture

The effects of internal porosity and crystallographic texture on Charpy absorbed energy (over temperature range of -196 to 600 °C) were determined by two heat treatment conditions (As-Built and Hot Isostatically Pressed (HIPed)) on Vertical and Horizontal specimen orientations [4]. The specimens were fabricated using an Arcam1 A1 EBM machine at the standard Arcam A1 build theme for Ti-6Al-4V (accelerating voltage 60 kV, layer thickness 50 μm, speed factor 35, software version 3.2.132) and the standard Arcam Ti-6Al-4V gas atomized powder (particle size range approximately 40–100 μm, average approximately 70 μm). Charpy build volumes were built 5mm above the build plate and connected to the build plate using standard thin wafer supports. It is important to note that Chaput, et al. [5] did not use supports and instead built parts directly attached to the build plate.

Total twenty specimen for each orientation (Horizontal, Vertical) were fabricated of which half were randomly assigned for HIPing, and other half remained As-Built leading to four testing conditions (As-Built/Horizontal, As-Built/Vertical, Horizontal/As-Built, Vertical/As-Built).
HIPed/Horizontal, and HIPed/Vertical). The standard Ti-6Al-4V HIP cycle was used (2 h at 900 °C and 100 MPa in argon, with 12 °C/min heating and cooling rates). Charpy tests were performed at temperatures ranging from -196–600 °C (±3°C). Wrought Ti-6Al-4V (mill-annealed condition) was also Charpy tested to provide a non-AM comparison for Charpy properties due to its different microstructure. Chemistry was measured for wrought Ti-6Al-4V, As-Built EBM Ti-6Al-4V, and HIPed EBM Ti-6Al-4V and then compared to ASTM F2924 [6].

Internal porosity was non-destructively evaluated for As-Built and HIPed material using a laboratory x-ray micro-computed tomography (CT) instrument. Texture was measured using electron backscatter diffraction (EBSD) on a scanning electron microscope (SEM) operated at 20 keV. Samples were mounted and polished with diamond slurry to 1 μm and then vibratory polished with 0.05 μm colloidal silica for up to 8 h. Both β and α texture were measured directly at 30 nm step size for 13 fields of view per testing condition.

No pores were observed (1 μm voxel size) in the HIPed condition and the pore size distribution for the as-built condition agreed well with previous work on the same material with pores up to 10 μm diameter in HIPed material (calculated relative density as 99.8% dense) [7]. All observed porosity was approximately spherical, indicating it is of the gas porosity, and not the lack of fusion, variety [8-10].

Representative fracture surfaces for all conditions are shown in Figure 3, displaying two main features of interest, internal pores and ridges. Internal pores (Figure 3f and black arrows) were observed on all As-Built fracture surfaces but not on HIPed fracture surfaces. Ridges (Figure 3e and white arrows) were observed on all Horizontal fracture surfaces but not on Vertical fracture surfaces which ran perpendicular to the layers (X-Y plane) and parallel to the build direction (Z).

Significant coarsening was observed due to HIPing (Figure 4) whilst, α lath thicknesses did not change appreciably as a function of distance from the bottom of the build volume. The As-Built/Vertical testing condition had a majority of 001 β orientation, but it was far from an exclusive 001 α fibre texture. The Burgers relationship was found to hold for α orientation with respect to β. It is apparent from Figure 5 that prior-β grains are elongated in the build (Z) direction. In larger area texture maps, no appreciable differences were perceived between the between HIPed/Horizontal (Figure 5a, Figure 5c) and HIPed/Vertical (Figure 5b, Figure 5d) conditions when compared. However, considerable differences were observed between the two planes (X-Y and ZX) due to the anisotropic morphology of the elongated prior-β grains in the z-direction. It has recently been shown that prior-β grain boundaries impede dislocation motion as long as the two adjacent prior-β grains are of different texture orientation [19].

III. RESULTS

A. Effect of Powder Oxidation

For a given oxygen content, no differences were observed in the overall roughness and appearance between the samples tested in different orientations. For the five-times reused and marginally oxidized samples, the fracture surface features had characteristics of both the virgin and highly oxidized samples, with more brittle-type features in the artificially oxidized samples and more ductile-type features in the five-times reused samples. However, voids were always detected on the fracture surfaces of the non-HIP specimens but were not observed on the HIP surfaces, irrespective of their oxidation levels and orientations. This was found to be true for the X-ray Computed Tomography (CT) scan results of HIP and non-HIP specimens. Figure 6 and figure 7 show the comparison between Charpy absorbed impact energies and Hardness Rockwell C values from the ends of the Charpy specimens (respectively) for the
Fig. 4: EBSD images showing representative direct β texture measurements (bright green) using 30 nm step size for a) As-Built and b) HIPed conditions [2]

Fig. 5: EBSD larger area α texture maps for a) HIPed/Horizontal X-Y plane, b) HIPed/Vertical X-Y plane, c) HIPed/Horizontal X-Z plane, and d) HIPed/Vertical X-Z plane [2]

four oxygen levels including the effects of the three specimen orientations and non-HIP versus HIP post-processing. Figure 8 shows the charpy absorbed impact energy as a function of consolidated material oxygen content, including the effects of the three specimen orientations and non-HIP versus HIP post-processing

It was perceived from the figures 6-8 that the Charpy absorbed impact energies decreased dramatically with increasing oxygen content in case of HIP and that HIP post-processing always
improved the impact energy compared to as-built samples. The orientation effects were found to be much less discernible for the highly oxidized specimens and the Z-X orientation was found to be the toughest. Additionally, the instrumented striker force-time histories indicated relative differences in ductility between the specimens, where the specimens with the lowest oxygen contents, the most brittle behavior by the highly oxidized specimens exhibited the most ductile behavior.

It was observed that HIP post-processing always improved the impact energy (as expected) compared to as-built samples and that reduces the effects of orientation in the most severely oxidized samples due to the dropped impact energies. In addition, HIP post-processing removes the typical micro-voids encountered and improves the ductility of EBM Ti-6Al-4V while maintaining the strength at acceptable levels [15-16].

B. Effect of internal porosity and crystallographic texture

The results suggest that internal porosity has a deleterious effect on Charpy absorbed energy, which has been shown previously for other material systems too [17]. X-ray CT measurements showed internal porosity in the As-Built condition (99.8% dense), but not in the HIPed condition which was supported by the fractography (Figure 3 through evidence of pores on the fracture surfaces of As-Built Charpy specimens. In Charpy results (Figure 10), HIPed conditions have higher absorbed energy compared to As-Built conditions and was attributed to the effect of internal porosity. α lath thicknesses as a function of distance from bottom of build volume are shown in Fig. 9. The observed coarsening of α laths due to HIPing (Figure 4, Figure 9) also contribute to this trend as it is known that coarser Ti-6Al-4V microstructures have higher Charpy absorbed energy [18].

The results also suggested that crystallographic texture has an effect on Charpy absorbed energy. For the HIPed condition, the Vertical orientation exhibited higher absorbed energy compared to the Horizontal orientation but for the As-Built condition, there appeared to be no difference (Figure 10). Even
higher absorbed energy than the Horizontal orientation. However, this apparent trend did not seem to exist for the As-Built condition. All of these observed differences appear to be larger at higher temperatures. HIPed EBM Ti-6Al-4V compares well with Wrought Ti-6Al-4V, despite differences in microstructure (mill annealed) and chemistry (e.g., oxygen content).

IV. CONCLUSION

It was found that excessive powder oxidation (oxygen mass fraction above 0.25 % and up to 0.46 %) dramatically decreases the impact energy, about seven times at the room temperature. As the powder oxygen mass fraction increased from 0.11 % to 0.53 %, Charpy impact energy of Ti-6Al-4V decreased in a smooth but rapid fashion (depending on orientation and post-processing). In addition, HIP post-processing significantly increases the impact toughness, especially for specimens with lower or normal oxygen content. The specimen orientation effect was found to be more significant for low oxidation levels. HIP post-processing increased toughness of the alloy in all directions, especially for low and medium oxygen content. Both the HIP post-processing and orientation effects on toughness largely disappeared at the 0.5 % mass fraction level.

Results suggest that internal porosity has a deleterious effect on the Charpy absorbed energy of EBM Ti-6Al-4V. This was evident as HIPed material displayed higher Charpy absorbed energy over a range of temperatures (-196 to 600 °C) compared to As-Built material, and spherical internal porosity was found (x-ray CT, fractography) in the As-Built condition (99.8% dense) but not after HIPing. Crystallographic texture was also found to influence Charpy absorbed energy along with the anisotropic grain morphology (i.e., prior-β grains elongated in the build (Z) direction). It was observed that for similar texture, crack pathways that cross more prior-β grain boundaries lead to higher Charpy absorbed energy. However, differing textures were found to negate the prior-β grain boundary strengthening effect in some cases, emphasizing the influence of texture and variations in texture on Charpy absorbed energy. For the four testing conditions (As-Built/Vertical, As-Built/Horizontal, HIPed/Vertical, and HIPed/Horizontal), types of texture varied from predominantly 001 β to predominantly 110 β, but none matched the most commonly reported texture i.e. 001 β-fibre.

V. FUTURE SCOPE

Apart from the many advantages of EBM, it has many challenges which are all related to powder used (as powder is the raw material for EBM). So far, researchers have qualitatively reviewed the (limited) parameters related to powder i.e. powder oxidation, internal porosity and crystallographic texture and their effect on the charpy impact energy only. Quantification of the respective contributions of porosity and coarsening haven’t been done till now. In addition, a deeper understanding of the influences of texture processing is necessary and needs to be addressed in future work.
REFERENCES


