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Graded microstructure and mechanical properties of additive manufactured Ti–6Al–4V via electron beam melting

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ABSTRACT

Electron beam melting (EBM®)-built Ti–6Al–4V has increasingly shown great potential for orthopedic implant and aerospace applications in recent years. The microstructure and mechanical properties of EBM-built Ti–6Al–4V have been systematically investigated in this work. Its microstructure consists of columnar prior β grains delineated by wavy grain boundary α and transformed α/β structures with both cellular colony and basket-weave morphology as well as numerous singular α bulges within the prior β grains. The β phase is found to form as discrete flat rods embedded in continuous α phase and its volume fraction is determined to be ~3.6%. Moreover, α' martensite was not observed as it has decomposed into α and β phases. In particular, the α/β interface was studied in detail combined transmission electron microscopy with atom probe tomography. Of note is that graded Ti–6Al–4V microstructure i.e. both prior β grain width and β phase interspacing continuously increase with the build height, was observed, which mainly arises from the decreasing cooling rate. Furthermore, an increasingly pronounced strain hardening effect was also observed as the previously built layers undergo a longer annealing compared to the subsequent layers. As a result, graded mechanical properties of Ti–6Al–4V with degraded microhardness and tensile properties were found. A good agreement with the Hall–Petch relation indicates that the graded property takes place mainly due to the graded microstructure. In addition, this graded microstructure and mechanical properties were discussed based on a quantitative characterization.

1. Introduction

Additive manufacturing (AM), commonly known as 3D printing, is a process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies [1]. In recent years, AM processes have been recognized as attractive techniques for producing prototypes or finalized parts rapidly and cost-effectively, correspondingly they were termed “rapid prototyping” or “rapid manufacturing” [2]. Electron beam melting (EBM®) is an AM technique which was made commercially available quite recently, using a high-energy electron beam to selectively melt a conductive metal powder bed directed by a computer aided design (CAD) model under a high vacuum. EBM is capable of producing fully dense, near-net-shape complex parts with high mechanical properties [3].

Ti–6Al–4V is the most prevalent titanium alloy and one of the most important engineering materials. Due to its high strength-to-weight ratio, good biocompatibility and outstanding corrosion resistance, Ti–6Al–4V has been widely used in aerospace, biomedical implants, marine and offshore, etc. [4]. Ti–6Al–4V is a typical α (hcp: hexagonal close-packed) + β (bcc: body-centered cubic) dual-phase alloy, where α phase normally precipitates in β matrix with the classic Burgers relationship: $(0001)_\alpha//(110)_\beta$, $[11\bar{2}0]_\alpha//[111]_\beta$. Aluminum is added to increase the strength of the alloy by solid solution hardening and α stabilization. Vanadium addition stabilizes β which significantly improves the room temperature ductility, by achieving balanced mechanical properties from duplex α/β microstructure. The duplex microstructure usually consists of numerous lamellar colonies composed of alternating layers of acicular α and thin layers of retained β, which gives rise to a combination of attractive mechanical properties such as high strength and good ductility [5]. Of particular interest is the so-called “interface phase” or “interfacial layer” which occur under certain conditions at the α/β interfaces in the two-phase titanium alloys [6,7]. Previous work indicates that the interface phase width in Ti–6Al–4V will increase with decreasing cooling rate and can be varied from 50 nm to 450 nm. The interface phase

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was previously identified as an intermediate transition phase with a face-centered cubic (fcc) or hcp structure formed during the β to α transformation, probably as a result of sluggish diffusion of β stabilizer such as vanadium [8]. However, it was argued that it is an artifact and consists of a titanium hydride formed during thin foil preparation for TEM examination [9]. The α/β interface phase has been extensively studied by using transmission electron microscope (TEM) over the past decades. Nevertheless, it is still a controversy on the nature of this interface phase or interface layer [10]. More recently, a grain boundary α–β–α layered structure was observed under TEM mode in a sintered Ti–6Al–4V, and its formation was explained as a result of a high O content of 0.49 wt.% [11]. Its morphology appears to be very similar with the interfacial layer that often found in α/β duplex titanium alloys. The α/β interface phase has been shown to be a significant factor in the tensile or fatigue fracture of two-phase Ti–6Al–4V alloy, because it may provide an easy crack path or crack initiation sites [8]. Therefore, it is very necessary to investigate this particular interface phase in order to obtain a better understanding of the α ↔ β phase transformation. Atom probe tomography (APT) is the unique and powerful technique, which is able to locate the alloying elements and quantify the composition at the atomic scale and in the three dimensions. In particular, APT is specialized in analysis of interphase interfaces in a wide variety of materials [12].

EBM is a preferred AM technique for fabricating Ti–6Al–4V parts because of titanium's high affinity for oxygen. Due to its broad application prospect in orthopedic implant and aerospace industries, Ti–6Al–4V parts built by EBM has been most investigated till date. The microstructure of Ti–6Al–4V fabricated by EBM consists of columnar prior β grains delineated by grain boundary α and a transformed α + β structure with both colony and basket-weave (or Widmanstätten) morphology within the prior β grains [13,14]. The columnar β grains formed due to the extreme solidification conditions (e.g. high thermal gradient and rapid solidification rate in the small melt pool) and the strong partitioning behavior of the main alloying elements in Ti–6Al–4V. They will grow epitaxially along the <001> build direction as a pseudo-uniaxial heat transfer direction occurs [15]. The retained β phase was found to be rod-like and its volume fraction was calculated to be only ~2.7% [16]. Moreover, numerous spherical or even irregular pores can be observed in EBM-built samples either as a result of the entrapped argon during the production of gas-atomized Ti–6Al–4V powder [16] or an insufficient melting around the interface between in-fill hatch and contour [14,17]. In addition to the typical α/β duplex microstructure that stated above, the presence of α′ martensite was also reported either in short builds, thin-wall structures or net structures of EBM-fabricated Ti–6Al–4V [13,18,19]. As a thermal cycling of rapid solidification, high cooling in solid state and long-term annealing at a build temperature of ~600–650 °C is involved layer by layer during the entire EBM process, an out-of-equilibrium, very fine, and build space- and geometry-dependent microstructure will be obtained in the as-built materials. It is thus easy to imagine that graded microstructure as well as the resulting graded properties would appear in EBM-built Ti–6Al–4V parts. Murr et al. [20] reported earlier that there existed significant differences on α platelet thickness (e.g. 1.6 μm and 3.2 μm at the bottom and the top, respectively) in microstructure and the corresponding hardness (42 HV and 37 HV) within a dimensional range of ~40 mm build height in EBM-built cylindrical Ti–6Al–4V samples. However, Hrabe et al. [21] pointed out that there was no difference on microstructure and mechanical properties with varying build height in EBM-built parts with a total build height of 27 mm. Ladani et al. [22] studied the mechanical anisotropy of EBM-built parts. It suggests that the flat-built (X-orientated) net-shaped specimens have superior tensile strength and microhardness compared to the other two build directions. It is imperative for any AM parts to have consistent microstructure and properties throughout, particularly for critical load bearing components. Overall, the published works on the microstructural consistency of EBM-built Ti–6Al–4V parts are very limited.

It has been reported that the tensile properties of EBM-built Ti–6Al–4V are comparable to wrought form and better than that of cast form [3]. However, it is still not extensively studied regarding the high strength and good ductility that are achieved in as-built EBM parts. It is worth noting that the α/β duplex microstructure of EBM-built Ti–6Al–4V has not yet been characterized in detail, particularly on the structure of α/β interface and the elemental segregation behavior at α/β interface. In order to elucidate the microstructure–property relationship from the viewpoint of atomic scale, APT was employed for α/β interface analysis in this work. Furthermore, the paper provides a quantitative composition and crystallography analysis of the microstructure of EBM-built Ti–6Al–4V, and discusses its microstructural evolution under such a complex process in detail.

2. Experimental

All test samples were fabricated by an Arcam A2XX system (as schematically shown in Fig. 1a) using the standard processing themes provided by Arcam AB. Pre-alloyed Ti–6Al–4V ELI (Grade 23) powder supplied by Arcam AB was used for the evaluation of graded microstructure and mechanical properties. The perfectly spherical morphology and clean surface of each powder are revealed in Fig. 1b–d, indicating good flowability and no oxidation. The powder size ranges from 45 to 105 μm. The nominal composition of as-supplied powders is 6Al–4V–0.03C–0.1Fe–0.15O–0.01N–0.003H and Ti Bal. (wt.%). Two build themes were employed in this work, i.e. Ti6Al4V-PreHeat-50 μm and Ti6Al4V-melt-50 μm. A 10 mm-thick stainless steel start plate is heated by the electron beam when a pressure of ~5.0e−4 mBar within the build chamber is achieved. Once a bottom temperature of 730 °C is reached, parts are built directly onto the preheated start plate by selectively melting layers of 50 μm under a controlled vacuum in the temperature range of 600–650 °C. The entire building process was kept under a vacuum of ~2.0e−3 mBar, controlled by using high-purity helium as regulating gas in order to prevent powder charging. Recycling of non-melted and/or sintered powder was achieved via the powder recovery system (PRS) and a vibrating sieve (mesh size ⩽150 μm). In order to investigate the consistency of microstructure and mechanical properties of EBM-built parts, two horizontal blocks (100 mm × 10 mm × 30 mm) were fabricated. Four tensile testpieces were wire-cut one-by-one from a horizontal block. They are termed 10 mm-1, 10 mm-2, 10 mm-3 and 10 mm-4 from bottom to top (as illustrated in Fig. 2).

Optical microscopy (OM; ZEISS Axioskop 2 MAT), scanning electron microscopy (SEM; JEOL JMS-6700F; 10 kV), X-ray diffraction (XRD; PANalytical Empyrean; Cu Kα; step size of 0.01°) and transmission electron microscopy (TEM; JEOL-2010; 200 kV) were used to examine the microstructure of as-built Ti–6Al–4V. OM and SEM samples were etched in Kroll's reagent (1–3% HF, 2–6% HNO_3, and 91–97% H_2O) for 10 s. Quantitative image analysis was carried out by using Image J software. TEM samples were prepared using ion milling with a beam voltage of 3.5 kV and a milling angle of 4–8°. Phase identification was carried out by means of XRD and selected area electron diffraction (SAED). APT specimens were prepared by focused ion beam (FIB) on a FEI Helios dual-beam via the lift-out technique [23]. The micro-tips were prepared using the annular milling method [24] to obtain an end radius of ~50 nm. APT specimens were analyzed by laser-pulsed local-electrode atom probe LEAP® 3000X HR at 40 K and a gauge pressure