

## Process Window for Electron Beam Melting of Ti-6Al-4V

A. Kirchner<sup>1</sup>, B. Klöden<sup>1</sup>, J. Luft<sup>2</sup>, T. Weißgärber<sup>1</sup>, B. Kieback<sup>1,2</sup>

<sup>1</sup> Fraunhofer Institute for Manufacturing Technology and Advanced Materials (IFAM),  
Winterbergstrasse 28, 01277 Dresden, Germany

<sup>2</sup> Institute of Materials Science, Technische Universität Dresden, 01062 Dresden, Germany

### Abstract

Selective electron beam melting (EBM) is a powder-bed based additive manufacturing technique for the fabrication of dense metallic parts. As a free-form deposition process EBM offers a high degree of freedom in design and a highly efficient utilization of material. For a widespread industrial application the achievable build rate is a critical factor. In this paper the process window for Ti-6Al-4V is explored by varying beam current and scan speed in a wide range. The EBM specimen were characterized by their density, porosity size and distribution, chemical composition, microstructure, hardness and tensile strength. Key findings include: (1) the minimum line energy necessary to achieve full density, (2) parameter sets for increased build rates at full density, and (3) formation of  $\alpha'$ -martensite at high scan rates.

### 1. Introduction

Additive manufacturing of metallic components experiences high growth rates presently. Among the advantages is the high degree of freedom of design, enabling the fabrication of light weight components and the integration of additional functionalities. Since additive manufacturing requires no part-specific tools it is economical to manufacture small batches and highly individualized parts. In most cases the parts are built near-net-shape. Thus the efficiency of additive manufacturing in utilization of material and energy is high. Productivity and cost of additive manufacturing processes are strongly influenced by achievable build rates.

In powder bed based processes virtual slices of 3D CAD models are transferred into physical components by selective melting of powder layers. In Selective laser melting this is realized by scanning with a laser beam, while EBM utilizes an electron beam [1]. Evacuating the process chamber to a high vacuum is a necessary condition. Therefore EBM is suitable for reactive metals with a high affinity for oxygen and nitrogen. Another characteristic of EBM is the preheating of the powder bed. This causes a slight sintering of the powder particles and counteracts electrostatic charging. In addition it minimizes residual stress in built parts.

Because of its high specific strength up to elevated temperatures and its superior corrosion resistance titanium and its alloys is used in aerospace, chemical industry, marine equipment and medical implants. Ti-6Al-4V is the most widely used alloy due to its balanced properties. The influence of EBM build parameters on Ti-6Al-4V microstructure has been studied in several papers. A refinement of the  $\alpha$ -lamellae with increasing scan speed has been reported [2,3]. This as well as remaining porosity and variation in chemical composition causes a wide range of tensile strength from 650 to 1200 MPa [2,4]. The mechanical properties of dense EBM specimen were found to be nearly isotropic [5]. In this work the influence of a wide range of build parameters on density, microstructure, chemical composition and mechanical properties is scrutinized.

### 2. Materials and methods

The Ti-6Al-4V powder with a nominal particle size distribution of 45 - 105  $\mu\text{m}$  was supplied by Arcam AB. Selected results of the powder characterization are given in Table 1. Recycling the powder for more than ten build jobs the only apparent change has been a moderately increased oxygen level of 1430 ppm.

**Table 1:** Properties of the used Ti-6Al-4V powder.

Size	Method	Value
D <sub>10</sub>	Horiba LA950	51.4 μm
D <sub>50</sub>	Horiba LA950	73.2 μm
D <sub>90</sub>	Horiba LA950	107.8 μm
Flowability	Hall flowmeter, ø 2.54 mm	21.8±0.14 s
Apparent density	Fill density funnel method	2.59±0.01 g/cm <sup>3</sup>
Al-content	ICP-OES	5.75%
V-content	ICP-OES	3.79%
Fe-content	ICP-OES	0.21%
O-content	LECO TCH 600	1160±100 ppm
N-content	LECO TCH 600	170±20 ppm
H-content	LECO TCH 600	10±10 ppm

EBM was performed on an Arcam A2x machine operating at a vacuum below  $2 \times 10^{-2}$  mbar and an acceleration voltage of 60 kV. The remaining gas consisted predominantly of He, which was bled into the chamber to reduce electrostatic charging. At the beginning of a build process a 150 mm x 150 mm steel start plate was heated to 730°C as measured by thermocouple underneath. The addition of each layer started by raking a powder layer of 50 μm nominal thickness. The fresh powder was preheated by fast scanning with a defocused electron beam. Then the part contours were molten using a focused beam and standard parameters. Subsequently the specimen interiors were solidified by scanning the surface at a constant line offset (lateral distance between parallel lines) of 100 μm with a focused electron beam (focus offset 3 mA). Between layers the principal scan direction was alternated from x-axis to y-axis. Beam currents ranging from 3 to 24 mA and scan speeds ranging from 0.5 to 16 m/s were employed. The line energy  $E_l$  can be calculated by

$$E_l = U_e \cdot I_b / v_{sc} \quad (1)$$

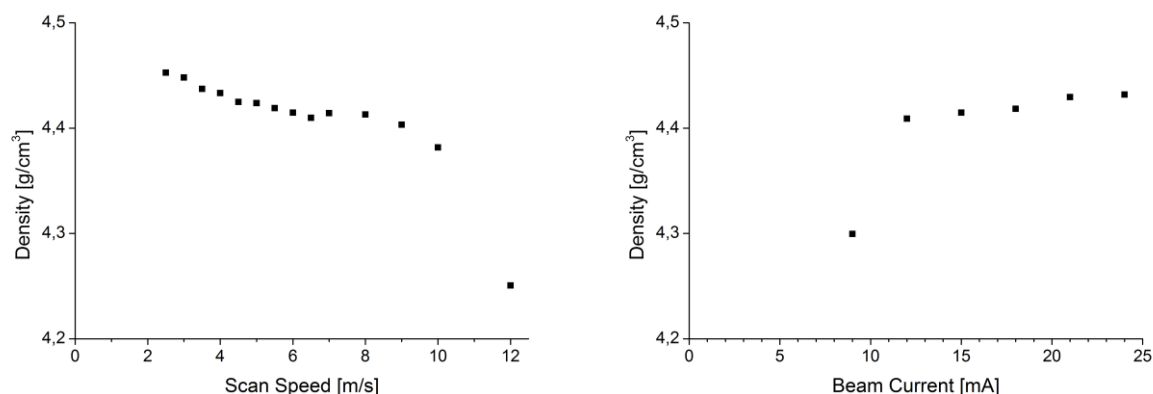
wherein  $U_e$  is the acceleration voltage,  $I_b$  the beam current and  $v_{sc}$  the scan speed.

Ten specimen of 50 mm length, 10 mm width and 14 mm height were built per process. Upon completion excess powder was removed by nitrogen jet blasting. Density of built parts was determined by weighing in air and in water. For metallographic preparation specimen were cut both in and perpendicular to build direction, mounted and polished. To reveal the microstructure polished samples were etched with Kroll's Reagent (100 ml H<sub>2</sub>O, 6 ml HNO<sub>3</sub> and 3 ml HF). Vickers hardness was measured on polished specimen using a load of 98 N. Flat specimen for tensile tests with a cross-sectional area of 4 mm<sup>2</sup> were cut by electrical discharge machining. Tensile tests were performed on a Zwick 1476 machine at room temperature and a deformation rate of 1.8 mm/min. For the chemical composition approximately 20 mg of sample were solved in hydrochloric acid. Elemental composition was analyzed using a Thermo Scientific 6300 DUO ICP-OES. Interstitial impurities were determined using a LECO TCH 600 analyzer.

### 3. Results and discussion

Achieving full density is prerequisite for high mechanical performance in powder metallurgical titanium [6]. The typical influence of the scan parameters speed and beam current on the density of EBM built parts is shown in Figure 1. While keeping the beam current constant a continuous decrease in density is observed with increasing scan speed. Above a scan speed corresponding to a line energy of approximately 100 J/m density drops sharply. A similar relation holds for the beam current. Below a beam current corresponding to the above mentioned 100 J/m no dense parts are achieved.

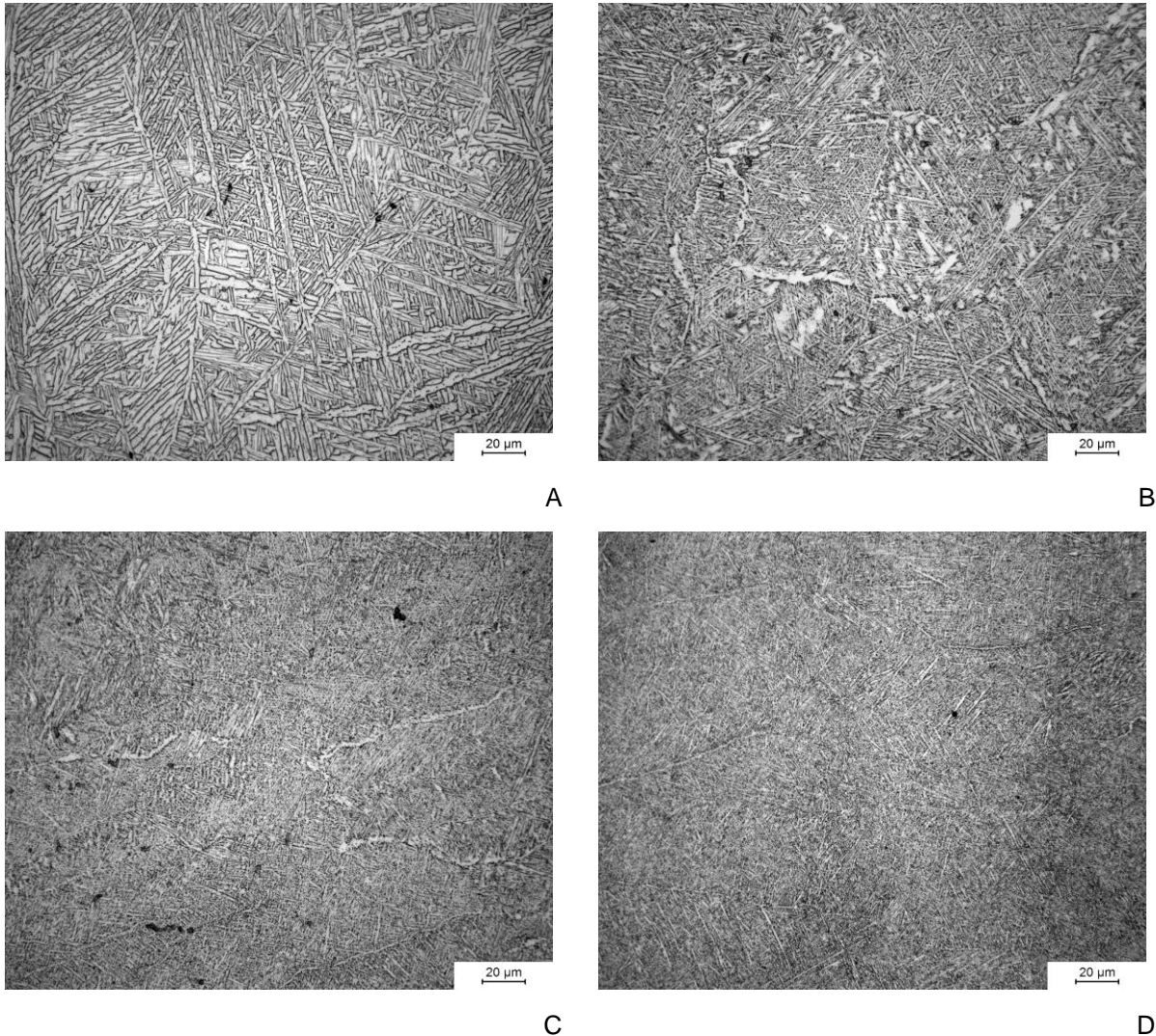
For combinations of high beam currents and low scan speeds the measured densities exceed the value 4.43 g/cm<sup>3</sup> given for Ti-6Al-4V material. This can be understood in terms of chemical composition. Because of its higher vapor pressure aluminum has the tendency to evaporate selectively [7]. A sample built with a line energy of 210 J/m contains only 5.48% Al, which falls below the minimum given by ASTM B265 for titanium grade 5. In addition swelling of the sample top surface is observed at high line energies of 300 J/m and above. This geometric inaccuracy is undesirable and likely induced by overheating causing an excess of powder to sinter to the surface during raking.



**Figure 1:** Density of EBM specimen as a function of scan speed (left, beam current 15 mA) and beam current (right, scan speed 6 m/s).

Any negative deviation of sample density from the theoretical value is caused by the presence of pores which are observed in metallographic cross-sections. Pores can be categorized into two types. All samples contain spherical pores of 20 to 50  $\mu\text{m}$  diameter. Their area fraction is 0.25% independent of the scan parameters employed. A similar value of 0.27% was found by Svensson and Ackelid [5]. These spherical pores can be explained by gas-filled cavities in the powder. In contrast pores caused by incomplete melting of the powder layer are of highly irregular shape and their size exceeds 100  $\mu\text{m}$  typically.

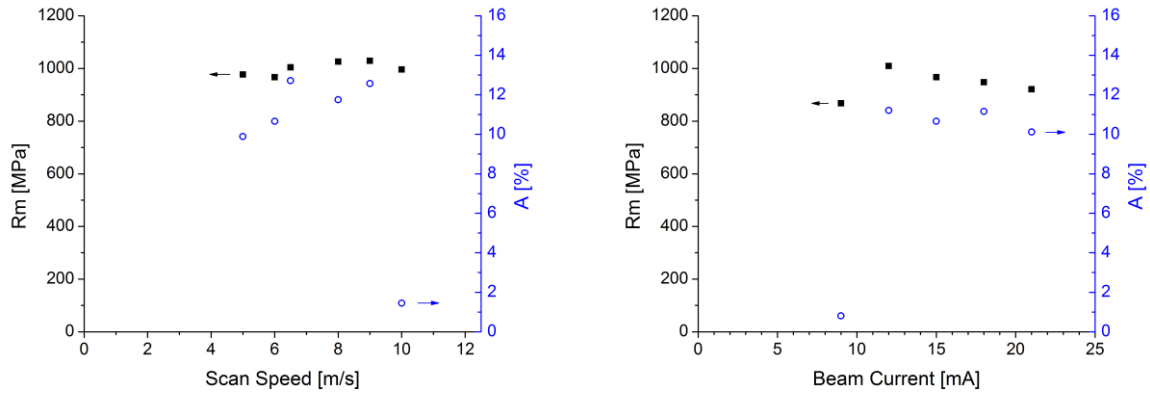
Comparing the microstructure of the specimen cut in and perpendicular to build direction, no significant differences are noticeable. Specimen built at low scan speeds exhibit fully lamellar microstructure (basket-weave) with  $\alpha$ -plate thickness up to 20  $\mu\text{m}$  (Figure 2). With increasing scan speed a significant refinement of the  $\alpha$ -plates is observed. At 4.5 m/s the maximum thickness is 3  $\mu\text{m}$  and reduced to 2  $\mu\text{m}$  at 7 m/s. Additionally the formation of martensitic  $\alpha'$ -phase is apparent. At a scan speed of 12 m/s the fraction of the needle-like  $\alpha'$ -martensite is nearly 100%. According to Ahmed and Rack cooling below 575°C at a rate of more than 400 K/s is necessary for the formation of a fully martensitic microstructure [8]. However depending on chemical composition, starting microstructure and homogeneity martensitic start temperatures as high as 800°C have been reported [9]. Here the specimen is kept at a temperature of approximately 700°C for the duration of the whole build process. Therefore the formation of martensite has to take place above that temperature. In addition the martensite does not decompose significantly during the build process. An incomplete decomposition of  $\alpha'$ -martensite at 700°C has been found previously [10].



**Figure 2:** Microstructure of EBM specimen cut in build direction. Scan speeds were (A) 2.5 m/s, (B) 4.5 m/s, (C) 7 m/s and (D) 12 m/s. The beam current was 15 mA.

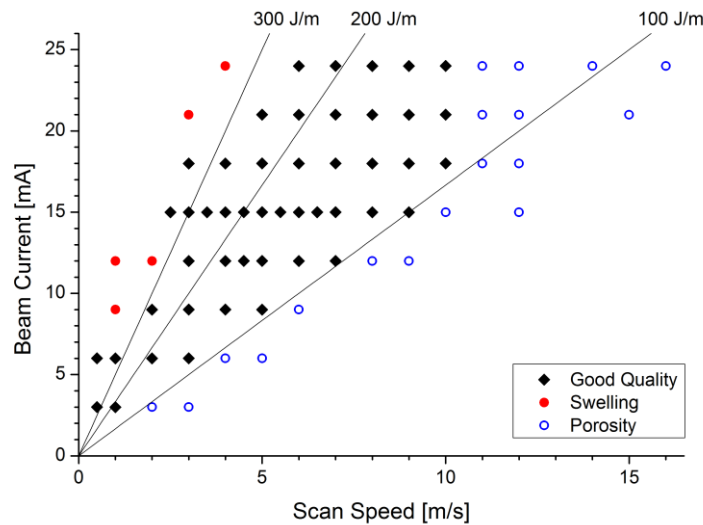
The mechanical properties of the EBM built specimen were characterized without subsequent heat treatment. Vickers hardness was tested on all specimen. For low scan speeds hardness values are approximately 310 HV 10. Moderate increases up to 340 HV 10 are observed for increasing scan speeds at all beam currents. This can be understood by the microstructural refinement and the formation of martensite. At the highest scan speeds hardness values exhibit high variability caused by inhomogeneous spatial distribution of porosity.

Tensile tests were performed on selected samples (Figure 3). Those built at a beam current of 15 mA exhibit tensile strength between 970 and 1030 MPa. With the exception of the sample built at 10 m/s elongations of 10% and more are reached. Because of their finer microstructure specimen built at higher scan speeds are stronger and more ductile. A scan speed of 10 m/s causes a porosity of 1.1%. Together with a fraction of martensite this provokes a sharp decrease of elongation below 2%. The variation of the beam current results in a similar behavior. At high beam currents fairly coarse microstructures are formed, resulting in tensile strength of 930 to 960 MPa. A maximum of tensile strength of 1010 MPa is recorded for 12 mA. At 9 mA the line energy is no longer sufficient for full melting. A porosity of 3% and of irregular shape is the reason for a dramatically low ductility.



**Figure 3:** Ultimate tensile strength  $R_m$  and total elongation at failure  $A$  of EBm specimen as a function of scan speed (left, beam current 15 mA) and beam current (right, scan speed 6 m/s).

From the presented results a process window for the electron beam melting of Ti-6Al-4V is drawn (Figure 4). As the detrimental effect of a porosity exceeding 1% on the mechanical properties is clearly demonstrated, good built quality is defined by a density of 99% or more and accurate geometric dimensions. Combinations of beam current and scan speed achieving these requirements are marked with black diamonds.



**Figure 4:** Process window for EBm of Ti-6Al-4V. Blue circles mark samples with more than 1% porosity, while red circles mark pronounced swelling of the top surface.

#### 4. Conclusion

Utilizing selective electron beam melting for the fabrication of test specimen we have investigated the effect of different parameter sets of beam current and scan speed on density, microstructure and mechanical properties. Using the line energy as the crucial parameter the following is found:

A line energy of more than 100 J/m is necessary for full densification of Ti-6Al-4V. At lower line energies EBm specimen contain more than 1% porosity, yielding lower tensile strength and greatly reduced ductility. Line energies in excess of 200 J/m cause noticeable loss of aluminum, while even higher values result in pronounced swelling of the specimen top surface. Thus it is concluded that good quality specimen can be fabricated employing line energies between 100 and 200 J/m.

Higher scan speeds result in a microstructural refinement and an increasing fraction of  $\alpha'$ -martensite. When combined with full density this yields higher tensile strength without loss of ductility. Tuning the microstructure via scan speed offers the possibility to tailor material properties.

Provided the availability of high beam currents scan speeds might be increased even above 10 m/s, demonstrating the potential for higher build rates. In case hot isostatic pressing (HIP) is employed as a subsequent treatment the requirement for a porosity of less than 1% after EBm might be relaxed.

**Acknowledgements**

The authors like to thank Kerstin Kümme, Marion Eckhardt, Volkmar Schlegel, Ralf Hauser and Tina Richter for their experimental support.

**References**

- [1] LE Murr et al: *Metal fabrication by additive manufacturing using laser and electron beam melting technologies*, J Mater Sci Technol 28 (2012) 1–14.
- [2] K Puebla et al: *Effect of melt scan rate on microstructure and macrostructure for electron beam melting of Ti-6Al-4V*, Materials Sciences and Applications 3 (2012) 259-264.
- [3] N Hrabec and T Quinn: *Effects of processing on microstructure and mechanical properties of a titanium alloy (Ti-6Al-4V) fabricated using electron beam melting (EBM)*, Materials Science & Engineering A 573 (2013) 271-277.
- [4] LE Murr et al: *Microstructures and mechanical properties of electron beam-rapid manufactured Ti-6Al-4V biomedical prototypes compared to wrought Ti-6Al-4V*, Materials Characterization 60 (2009) 96-105.
- [5] M Svensson and U Ackelid: *Titanium alloys manufactured with electron beam melting - Mechanical and chemical properties*, Proceedings of the Materials and Processes for Medical Devices Conference, Minneapolis (2009) 189-194.
- [6] FH Froes: *Titanium Powder Metallurgy: A Review-Part 1*, Advanced Materials and Processes 170 (2012) 16-22.
- [7] A Powell et al: *Analysis of multicomponent evaporation in electron beam melting and refining of titanium alloys*, Metallurgical Transactions B 28 (1997) 1227-1234.
- [8] T Ahmed and HJ Rack: *Phase transformations during cooling in  $\alpha+\beta$  titanium alloys*, Materials Science and Engineering A243 (1998) 206–211.
- [9] SS Al-Bermani et al: *The origin of microstructural diversity, texture, and mechanical properties in electron beam melted Ti-6Al-4V*, Metallurgical and Materials Transactions A 41 (2010) 3422-3434.
- [10] FX Gil Mur et al: *Influence of tempering temperature and time on the  $\alpha'$ -Ti-6Al-4V martensite*, Journal of Alloys and Compounds 234 (1996) 287-289.