

COST STSM REPORT

Investigation of anisotropic properties of Rapid Prototyped metallic implants

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STSM Applicant: Mr Bartlomiej Wysocki, Warsaw university of Technology, Faculty of Materials Science and Engineering, Warsaw (PL), wysocki.bartlomiej@gmail.com

STSM Topic: Investigation of anisotropic properties of Rapid Prototyped metallic implants

Host: Joseph Buhagiar, University of Malta, Msida (MT), joseph.p.buhagiar@um.edu.mt

AIM OF THE COST STSM

The aim of the COST STSM entitled “Investigation of anisotropic properties of Rapid Prototyped metallic implants” was two fielded. First area of the work was investigation of crystallographic orientation (CO) of titanium implants and the second one was an attempt to produce by EBM scaffolds with dimensions and porosity similar to SLM ones fabricated in home institution. Because of the scanning electron microscope breakdown at the host institution the main area of work was moved to implant modelling, fabrication by electron beam melting and characterization of fabricated samples. The STSM applicant decided also to make by EBM additional solid samples in which anisotropy of mechanical properties was checked at the applicant home institution. The additional crystallographic orientation (CO) test are planned as soon as the maintenance of the microscope at host institution will be finished.

1. INTRODUCTION

The major advantage of using RP methods in medical applications is their ability to manufacture complex 3D patient-specific implants by using computer tomography data. Till now, publications on RP of metal implants have focused mainly on knee and hip replacements or bone scaffolds for tissue engineering. The direct fabrication of metallic implants can be achieved by various methods including Direct Metal Laser Sintering (DMLS), Selective Laser Melting (SLM) and Electron Beam Melting (EBM). Research on the properties of titanium and its alloys made by selective laser or electron beam melting is continuously conducted to optimize the

manufacturing parameters. An interesting aspect of the research concerns particularly the impact of process parameters on the microstructure, mechanical properties and dimensional reproduction. Especially the anisotropy of mechanical properties is a major technological problem as evidenced by numerous research articles concerning this topic and heat treatment as a remedy. Anisotropy of mechanical properties, often conducted with anisotropy of microstructure, is observed in the printed elements whether they are fabricated from polymers or metals. Elements produced using 3D printers also have an undesired characteristic layered structure with developed surface visible to the naked eye. It has been shown that the anisotropy of mechanical properties is dependent on both the location of the item on the platform, as well as production parameters such as energy source power used for the consolidation.

2. MATERIALS AND METHODS

The titanium scaffolds ($R = 10\text{ mm}$, $h = 10\text{ mm}$) were modeled in Magics software (Materialise NV, Belgium) by filling solid cylinders with diamond elemental structures of various sizes (0.87, 1.07 and 1.27 mm). The scaffolds were modeled with constant pore size 500, 600, 750 μm (Fig. 1a) and with bimodal pore sizes – core 500 μm and shell 750 μm (Fig. 1b). The bimodal scaffold was made with Boolean operations using previously created models with one size of pores. The modelled porosity of created structures was 70%. Used diamond structure is shown in Fig. 2. Solid cubic samples (14 mm edge) were modelled in Magics software and converted similar like scaffolds to .STL files for EBM fabrication.

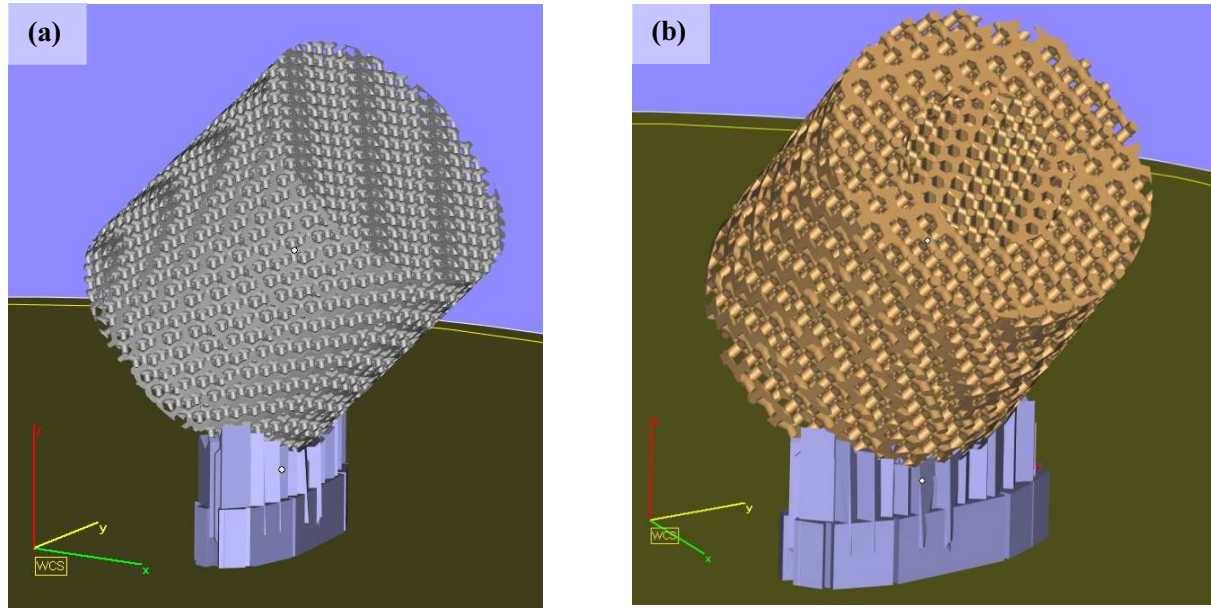


Fig. 1. Models of scaffolds created in Magics Materialise software; 500 μm pore size (a); biomodal 500 μm core + 750 μm core pore size (b).

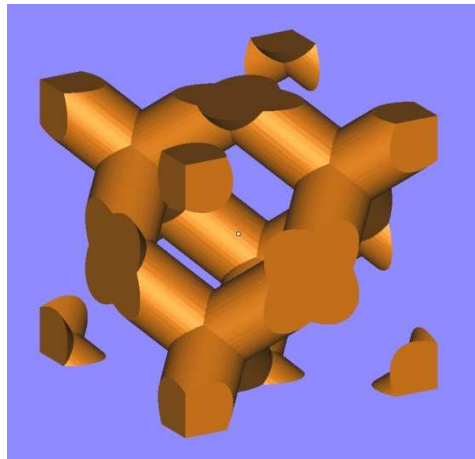


Fig. 2. Diamond structure used for porous titanium scaffolds fabrication (Magics Materialise software).

Both scaffolds and solid samples were fabricated from Ti-6Al-4V alloy powder with mean diameter 70 μm by EBM S12 (ARCAM AB) machine for manufacturing metal components (Fig. 3a). The layer thickness in all processes was 50 μm . The machine maintenance performed before scaffold fabrication is shown in Fig. 3b. The process parameters are summarized in Table 1 (scaffolds series 1 and solid samples) and Table 2 (scaffolds series 2).



Fig. 3. Arcam EBM S12; a) Machine overview; b) Electron beam maintenance.

Table 1. Process parameters for scaffold series 1 and solid samples

Layer thickness	50 μm
Beam scanning speed	4530 mm/s
Beam current	15 – 21 mA
Chamber pressure	2.0 e-3 mBar
Bottom temperature	589 °C

Table 2. Process parameters for scaffold series 2

Layer thickness	50 μm
Beam scanning speed	500 mm/s
Beam current	1,7– 3,0 mA
Chamber pressure	2.0 e-3 mBar
Bottom temperature	595 °C

3. RESULTS

3.1. FABRICATED SAMPLES

During STSM there were fabricated several scaffold samples by EBM machine. The models were created with various porosity from 500 μm to 750 μm to find the best accuracy at EBM S12 machine. The first series of samples had supports which were connected to scaffolds to strong (Fig. 4) and were melted similarly to solid samples. The scaffolds from series 1 had no open porosity independently the structure size used for fabrication. Another series of the scaffolds, fabricated with different support and scaffold parameters (Fig. 5a), had open porosity but overall accuracy of the scaffolds was much lower than those fabricated in home institution by SLM method (Fig. 5b). There was successfully fabricated by EBM bimodal scaffold with pore sizes 500+750 μm .



Fig. 4. Scaffold series 1. Diamond structure size (from the left to right): 0.87, 1.07 and 1.27 mm; side view (a); top view (b).



Fig. 5. Comparison of scaffolds fabricated by EBM and SLM method; Scaffold series 2. Diamond structure size (from the left to right): 0.87, 1.07, 1.27, 0.87+1.27 mm (a); Scaffolds fabricated by SLM. Diamond structure size (from the left to right): 0.47, 0.87, 0.47+0.87 mm (b).

The solid samples fabricated by EBM at host institution were very successful (Fig. 6). They had good accuracy, there were no visible defects at layers or under metallographic observations (Fig. 7). There were cut from them micro samples for tensile tests (Fig. 6b) and micro-tomography which will be performed at home institution.

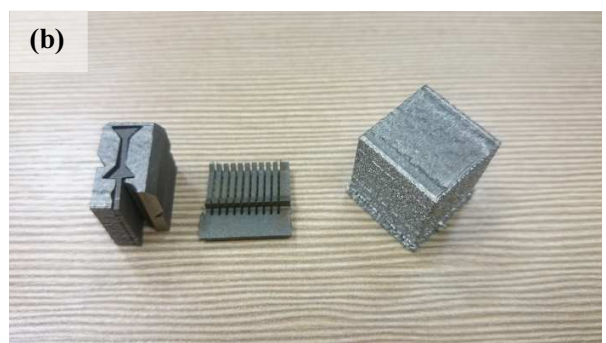


Fig. 6. Solid samples from Ti-6Al-4V alloy fabricated by EBM method; cube (a); microsamples for tensile tests cut from cube (b).

3.2. MICROSTRUCTURE

Cross-sections perpendicular (XY) and parallel (XZ) to the build direction (Z), for microstructure observations on a light microscope, were cut from solid samples. For the metallographic

observations cut surfaces were prepared by new procedure developed at host institution. The procedure involved grinding on a resin bonded diamond disc (Struers MD-Piano 1200) and fine polishing with colloidal silica suspension (Struers OP-S) on porous neoprene disc (Struers MD-Chem). The solution of HNO₃:HF:H₂O (4:1:1 vol.) was used for the samples etching.

The microstructure of Ti-6Al-4V alloy fabricated by EBM (host institution) and SLM (home institution) are shown in Fig. 7 and Fig. 8 respectively. Pores and unmelted regions were apparent in the samples produced by each method. Both processes provided epitaxial solidification of the new layers through heterogeneous nucleation. Epitaxial grains fabricated during the EBM process were no longer than a few hundred micrometres and were around 60 μm wide (Fig. 7b). However, epitaxially grown grains 100 – 200 μm wide and even a few millimetres long were observed in the laser melted samples (Fig. 8b). Optical microscopic observations revealed a microstructure with many parallel and perpendicular etched plates which are described as an acicular α' martensite type of microstructure in the SLM fabricated [1, 2] (Fig. 8a, 8b). The EBM process produced a very fine needle-like $\alpha+\beta$ Widmanstätten microstructure where the α -Ti acicular plate grains were a light phase surrounded by dark β -Ti grain boundaries (Fig 7a, 7b). Furthermore an equilibrium α -phase participation at the boundaries of epitaxially grown grains (Fig. 7b), which were not apparent after the SLM process, were observed. The epitaxial grains observed under optical microscopy can suggest microstructural anisotropy which will be further checked at host institution.

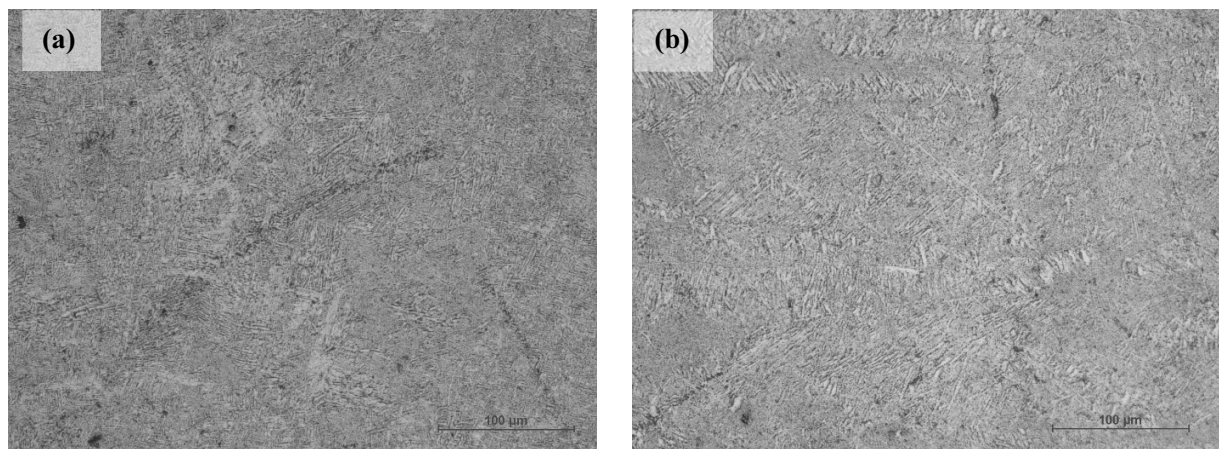


Fig. 7. Microstructure of Ti-6Al-4V alloy processed by EBM; a) XY plane; b) XZ plane.

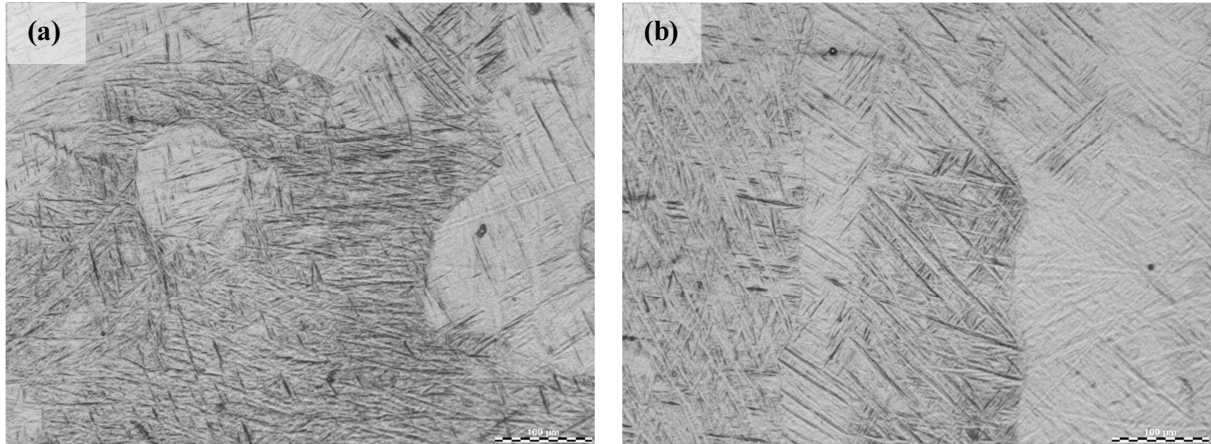


Fig. 8. Microstructure of Ti-6Al-4V alloy processed by SLM; a) XY plane; b) XZ plane.

3.3.MECHANICAL TESTS

Elongation and Tensile tests were performed by using MTS QTest/10 machine equipped with attachments for non-standard sample tests. Mechanical tests were performed on 2 sets of microsamples [3, 4] cut by WEDM saw perpendicularly and parallel to the building direction in XY and XZ planes respectively. The samples had a measurement length of 5 mm and 0.8 mm thickness. The average ultimate tensile strength was 960 MPa and 970 MPa for samples cut in XY (Fig. 9) and XZ (Fig. 10) planes respectively. Highest differences were observed at elongation at break which was 12 - 16 % and 14 – 16 % for samples cut in XY (Fig. 9) and XZ (Fig. 10) planes respectively. The values of tensile strength and elongation of EBM fabricated Ti-6Al-4V alloys meet ASTM requirements for implants for surgical devices.

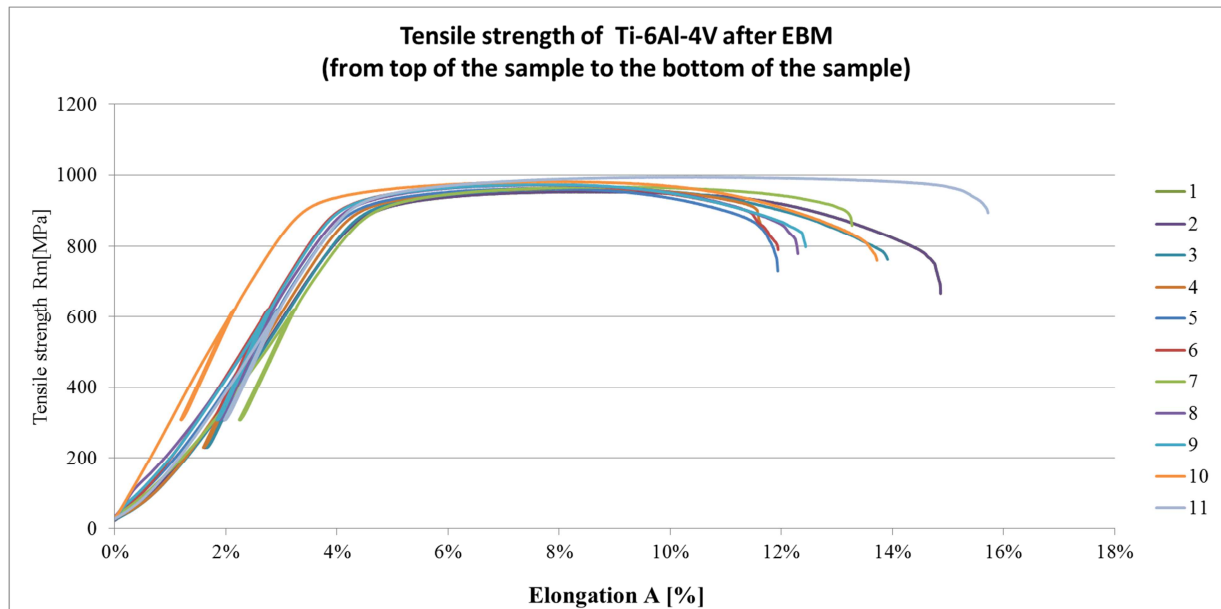


Fig.9. Stress-Strain curves for Ti-6Al-4V alloy microsamples cut in XY plane from top to the bottom of the sample.

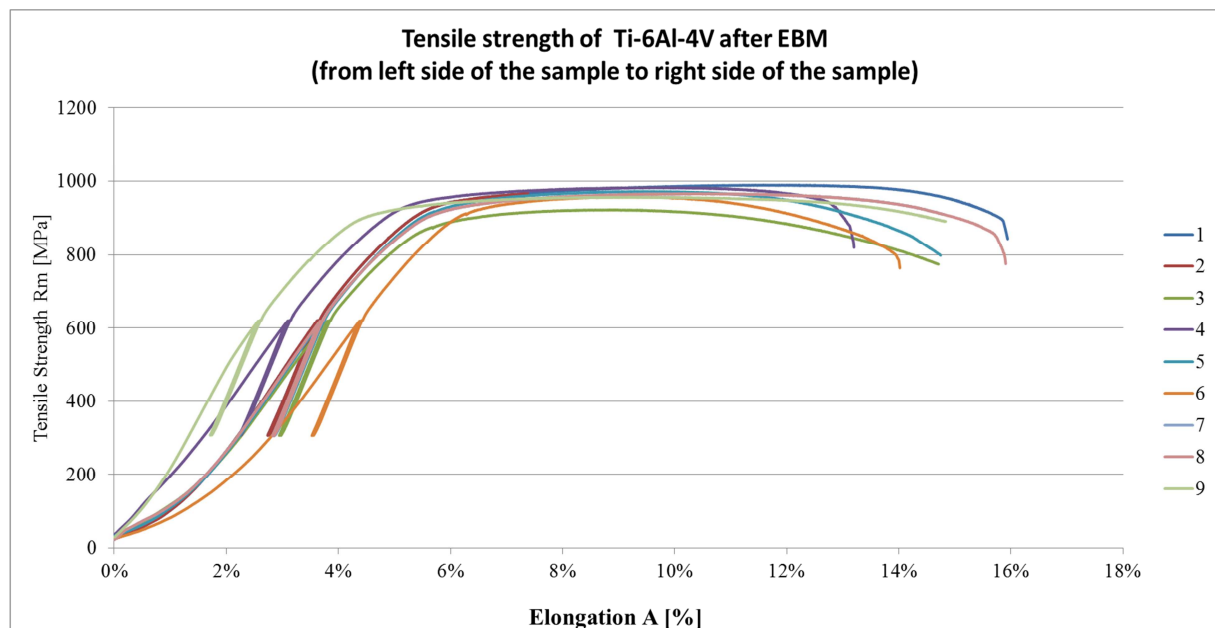


Fig. 10. Stress-Strain curves for Ti-6Al-4V alloy microsamples cut in XZ plane between edges of the sample.

CONCLUSIONS

The microstructure and mechanical properties of EBM fabricated implants meet ASTM requirements for surgical devices. There are no mechanical properties differences within the EBM fabricated samples which could exclude Ti-6Al-4V alloy from usage for medical implants. The epitaxially grown grains after EBM process which are visible under optical microscope had not generated mechanical properties anisotropy. There is no possibility for fabrication by EBM S12 titanium scaffolds for bone regeneration with pore size below 500 μm and with kept open porosity. The EBM process because of its accuracy is more suitable for solid implants fabrication than for porous scaffold fabrication. We suggest SLM process for titanium scaffolds fabrication because of higher accuracy and possibility of controlling more process parameters.

FUTURE COLABORATION

- To ensure the obtained results crystallographic orientation investigation by EBSD will be performed for both solid and porous samples fabricated by both methods.
- Similar mechanical test will be performed on solid samples fabricated from Ti-6Al-4V alloy by SLM at home institution.
- Obtained results are very promising and will be send for publication in scientific journal.

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