

# Microstructure Evolution in Additive Manufactured Alloys

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**Abstract**—Martensitic texture-phase transition in Selective Laser Melting (SLM) Ti-6Al-4V (ELI) alloys was found. Electron Backscatter Diffraction (EBSD) analysis showed the initial cubic beta <100> (001) BCC texture. Such kind of texture is observed in BCC metals with flat rolling texture when axis is in the direction of rolling and the texture plane coincides with the plane of rolling. It was found that the texture of the parent BCC beta-phase determined the texture of low-temperature HCP alpha-phase limited the choice of its orientation variants. The {10-12} <-1011> twinning system in titanium alloys after SLM was determined. Analysis of the oxygen contamination in SLM alloys was done. Comparison of the obtained results with the conventional titanium alloys is also provided.

**Keywords**—Additive technology, texture, Ti-6Al-4V, twins, oxygen content.

## I. INTRODUCTION

THE SLM is an additive manufacturing technology implying complete remelting of the powder, rapid direct solidification of the melt and in situ thermal cycling of the material due to repeatable scanning of a surface by laser. Because of these complicated conditions of microstructure formation after manufacturing, the material has metastable, often nonequilibrium nanoscale structure. Materials manufactured by SLM often demonstrated unexpected set of structural and defect states, high level of tensions, and therefore, should be better understood for optimization of the SLM process parameters and producing materials with high exploitation properties [1].

Texture and twinning play an important role in the deformation and manufacturing of the alloys. Twinning process depends on the orientation of the sample with respect to the applied stress and deformation speed. Twinning increases with the increase of the degree of deformation, purity of metal, grain size at low temperatures when the dislocation slip processes are complicated. Twinning plays an important role in the deformation and manufacturing of the titanium alloys. Twinning process depends on the orientation

of the sample with respect to the applied stress and deformation speed. Orientation of the sample affects the value of resolved shear stress of the twinning dislocations.

During solidification under SLM, the BCC  $\beta$  phase preferentially grows in the <100> direction, giving rise to the long, columnar prior  $\beta$  grains. The  $\beta$  martensitic phase is organized within prior  $\beta$  grain boundaries according to the orientation relationship between  $\beta$ - and  $\alpha$ - phases:  $(110)_{\beta} \parallel (0001)_{\alpha}$  and  $\langle 1-11 \rangle_{\beta} \parallel \langle 11-20 \rangle_{\alpha}$ . The texture of the parental  $\beta$  phase is transmitted to a texture of the  $\alpha$ - phase with variant selection [2].

Main purpose of this work is to study the texture and twinning processes in SLM Ti-6Al-4V.

## II. EXPERIMENTAL

TiAl6V (ELI) has the low density, low elastic modulus, and high strength. Biocompatibility of this material allows one to adapt it to human implant production and successfully use in the manufacture of surgical implants. Spherical argonatomized Ti6Al4V (ELI) (45  $\mu\text{m}$ ) powder from TLS Technik was used for study. The chemical composition complies with the ASTM F-136 standard for surgical implant applications. Two machines from two scientific centers (Russia and South Africa) were used for the manufacturing of the alloys. Horizontal samples were produced by the EOSINT M280 machines (EOS GmbH) equipped with an Ytterbium fiber laser operating at 1075 nm wavelength (IPG Photonics Corp.).

The laser beam had a TEM00 Gaussian profile and 80  $\mu\text{m}$  spot diameter. In accordance with the standard process parameters for a Ti6Al4V alloy, a powder layer thickness of 30  $\mu\text{m}$  and a back-and-forth scanning by strips with the hatch distance of 100  $\mu\text{m}$  were applied. The substrate and powder material was similar in chemical composition. Argon was used as the protective atmosphere; the oxygen level in the chamber was 0.07–0.12%.

Method of nuclear microanalysis was used to determine the oxygen concentration. Van de Graaff accelerator with 2 Mega Volts was used. The beam diameter was 1 mm.

## III. RESULTS AND DISCUSSION

X-ray diffraction patterns of the SLM samples are shown in Fig. 1. There are no beta phase lines in the diffraction patterns of both samples. Such kind of diffraction patterns is usual for SLM Ti6Al4V samples and may be explained by the high rate of cooling in the SLM methods which provided the martensitic transformation [3].

Oxygen contamination may also increase the rate of martensitic transformation in titanium alloys [4]. Harder outer alpha case layer in the cast Ti6Al4V alloys is a serious metallurgical problem. In the cast Ti6Al4V alloys the depth of the alpha case layer may be achieved up to 250  $\mu\text{m}$  [5].

Fig. 2 shows the results of the nuclear microanalysis of the surface studying of the samples. Increasing of the oxygen concentration in the surface layer (0.5  $\mu\text{m}$ ) is connected with the preparation of the samples (polishing). One can see that oxygen concentration in both samples is about 2 wt.% and decreases with

the increase in the depth. These results may testify the absence of the oxygen contamination during of the SLM manufacturing process.

Fig. 1 shows the microstructure of the samples manufacturing with the different machines. One can see the long grains with the martensitic plates. The orientation of the long grains is determined as crystallographic direction  $\langle 100 \rangle$  BCC.

TEM studies show the fully martensitic twinning structure without any  $\square$  precipitations (Fig. 4). All observed twins were of one type and were identified as tensile  $\{101\bar{2}\}$  twins. SAED patterns taken from the twin regions reveal a high density of fringes and streaks indicating the possible presence of multiple stacking faults. The width of twins was in range of 10-15 nm. Two variants of twinning system  $\{10-12\}$   $\langle 10-11 \rangle$  may be simultaneously present in the dark-field pictures depending on the

orientation of the plane of the foil (Fig. 4 (a)). Angle between the directions of the output of the twinning plane is about of  $64^\circ 47'$ , which corresponds to the angle between the output directions of  $\langle -1011 \rangle$  and  $\langle 10-11 \rangle$  of the  $(10-12)$  and  $(-1012)$  twinning planes, respectively (Fig. 4 (b)).

The four most commonly activated twinning systems were found in bulk Ti-6Al-4V such as compression twinning  $\{1011\}$  and  $\{11-22\}$ , tension twinning  $\{10-12\}$  and  $\{11-21\}$  [2]. Two variants of the  $\{10-12\}$  deformation twins were found after a simple shear deformation applied on two phase  $(\square+\square)$  Ti6Al4V plate material. The applied shear strains are 10%, 30%, and 50% [6].  $\{10-11\}$  twins were found in Ti-6Al-4V alloys after high strain rate (up to 5000/s) compression of

hcp

hcp

(a)

$\alpha'$  structure with high dislocation density and stacking faults was typical in the as-built samples. No  $\beta$  phase was detected.

- Tensile  $\{10-12\}$  twinning was identified by TEM and electron diffraction. This type of twinning system is correlated with the maximum Schmid factor for  $\square 1-100$  loading direction. Twin presence suggests the high internal stresses in the SLM material.
- Orientation texture - phase transition  $\square \square \square$  was found in SLM Ti-6Al-4V. Parental  $\square$ - phase showed columnar growth with highly pronounced texture  $\langle 100 \rangle$ . The texture of  $\alpha'$  was quite weak.

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