

Texture and Twinning in Selective Laser Melting Ti-6Al-4V 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 β phase preferentially grows in the <100> direction, giving rise to the long, columnar prior β grains. The α' martensitic phase is organized within prior β grain boundaries according to the orientation relationship between α - and β - phases: $(110)_{\beta} \parallel (0001)_{\alpha}$ and $\langle 1-11 \rangle_{\beta} \parallel \langle 11-20 \rangle_{\alpha}$. The texture of the parental β phase is transmitted to a texture of the α - 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 argon-atomized Ti6Al4V (ELI) (45 μm) 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 μm spot diameter. In accordance with the standard process parameters for a Ti6Al4V alloy, a powder layer thickness of 30 μm and a back-and-forth scanning by strips with the hatch distance of 100 μm 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].

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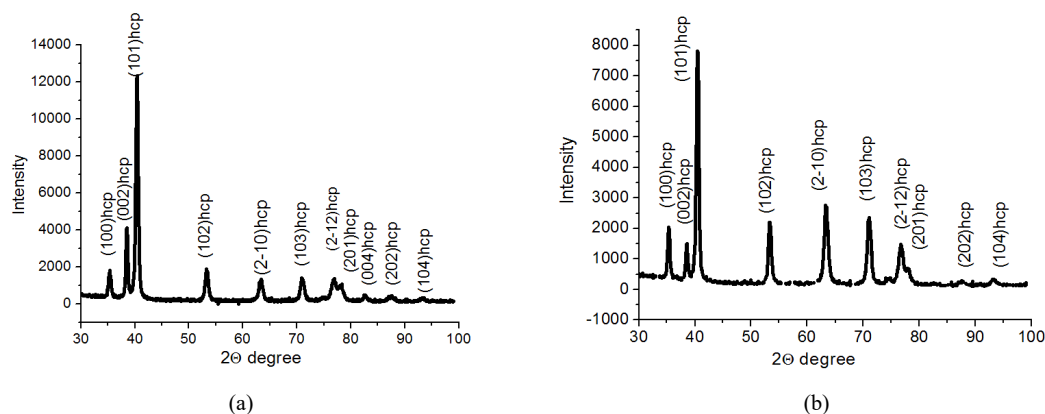


Fig. 1 X-ray diffraction patterns of the SLM samples: (a) sample 1, (b) sample 2

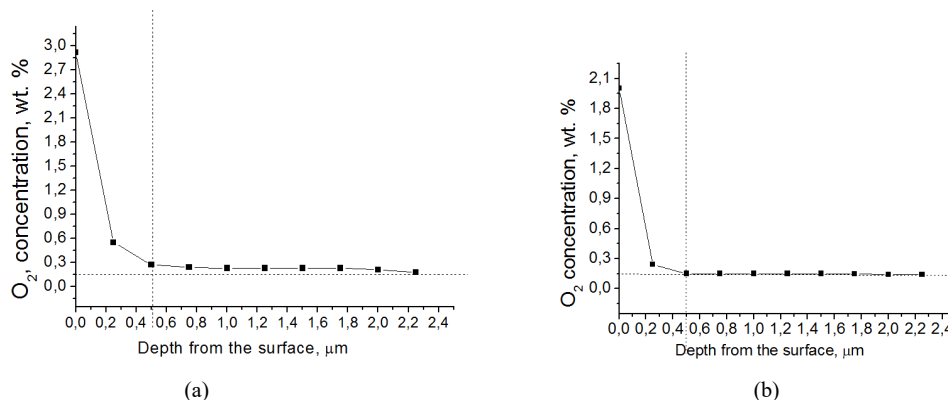


Fig.2 Oxygen concentration in the samples obtained by nuclear microanalysis: (a) sample 1; (b) sample 2

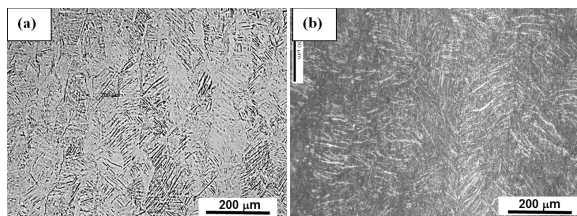


Fig. 3 Initial state (SLM) of the samples, optical microscopy, SEM: (a) sample 1; (b) sample 2

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 μm [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 μm) 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 β 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 $64^\circ 47'$, 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 $\{10-11\}$ 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 ($\alpha+\beta$) 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

cylindrical and hat-shaped specimens in a split Hopkinson pressure bar setup [7]. $\{10\text{-}12\}$ Twinning system in titanium has the lowest critical shear stress for twinning. The observed $\{10\text{-}12\}$ twinning is classified as tensile and it results in 16.7% strain in the $\langle 1000 \rangle$ direction in hexagonal close packed structure [8].

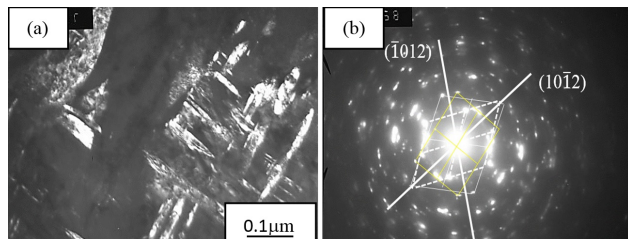
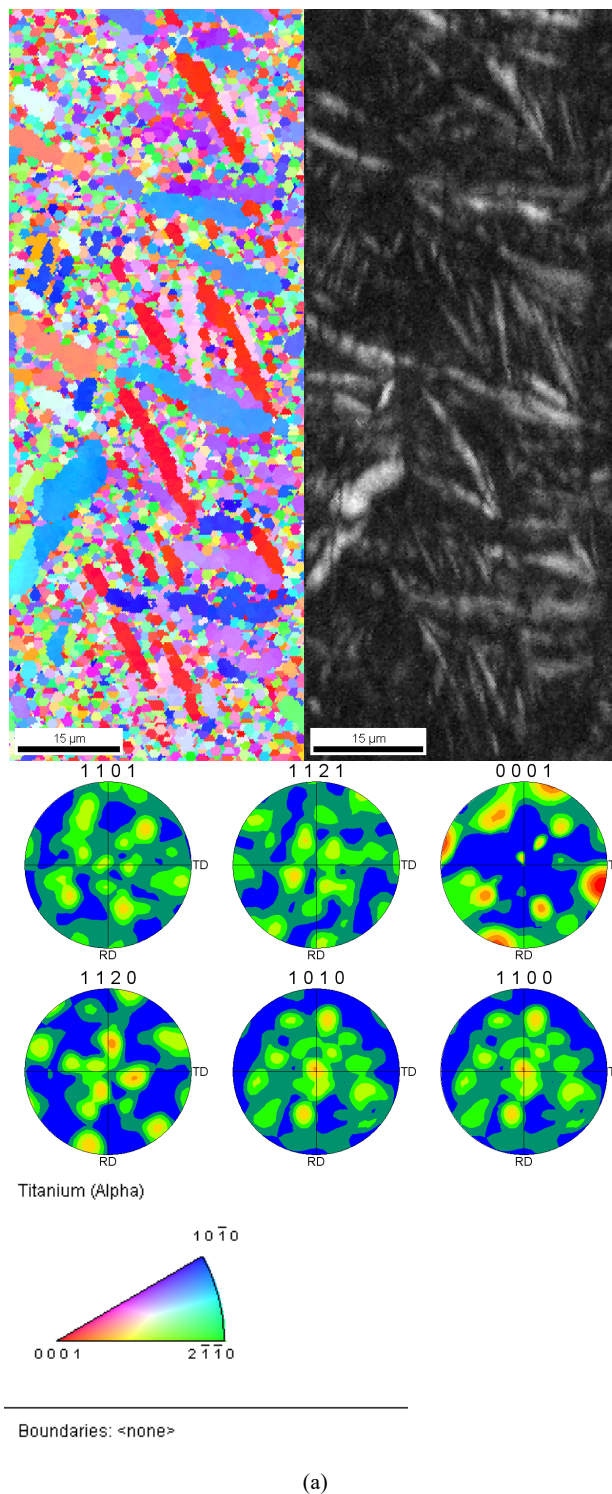


Fig. 4 Microstructure of the SLM Ti-6Al-4V alloy, sample 1, TEM

Variant of twinning plane in titanium alloys depends on the crystal orientation and the loading direction to the crystal c -axis. This fact becomes important in the case of the texture. Criterion for selection the variants of twinning is the Schmid's law. Twin variants having Schmid factors higher than 0.4 is suggested should be activated [9]. Schmid factors for $\{10\text{-}12\}\text{-}\langle 1011 \rangle$ twinning system in Ti-6Al-4V alloy are calculated in our work: under loading along the $\langle 0001 \rangle$ axis, it is 0.25 and it is 0.45 along the $\langle 1\text{-}100 \rangle$ axis.

During solidification of the Ti-6Al-4V alloy in the process of SLM, BCC β -phase mainly grows in the direction of $\langle 100 \rangle$, which leads to the formation of long, columnar grains (Fig. 3). Thus, the parental BCC texture resembles a cubic $\langle 100 \rangle$ (001) BCC texture, which is observed in flat-rolled BCC metals when the texture axis lies along the rolling direction and the plane of rolled texture coincides with the rolled plane [10]. In titanium alloys, α' -martensite phase is formed in accordance with β -grain orientation relationship between α - and β -phases: $(110)_{\beta} \parallel (0001)_{\alpha'}$ and $\langle 1\text{-}11 \rangle_{\beta} \parallel \langle 11\text{-}20 \rangle_{\alpha'}$. In the case of the phase transition $\beta \rightarrow \alpha'$ with orientation of a cubic crystal lattice along the $\langle 100 \rangle$ direction the three variants of the orientation of the hexagonal crystal lattice such as, $\langle 1\text{-}101 \rangle$, $\langle 11\text{-}21 \rangle$, and $\langle 11\text{-}20 \rangle$ should be found. Texture of the parental β -phase determines the texture of α' -phase, limiting the choice of its orientation variants [3]. So, the texture-phase transition with the formation of α' texture such as, $(11\text{-}20) - (\text{ND})$, $\langle 1\text{-}100 \rangle - (\text{RD})$, $\langle 0001 \rangle - (\text{TD})$ occurs because of direct martensitic $\beta_{001} \rightarrow \alpha'$ transformation.

The EBSD analysis of the samples (Fig. 5) shows the absence of $\langle 0001 \rangle_{\beta}$ texture and presence of $\langle 1\text{-}100 \rangle_{\beta}$ texture in both of samples. Two variants of α' -plate directions such as, $\langle 11\text{-}20 \rangle$ and $\langle 11\text{-}21 \rangle$ also may indicate the presence of the texture of α' -phase.



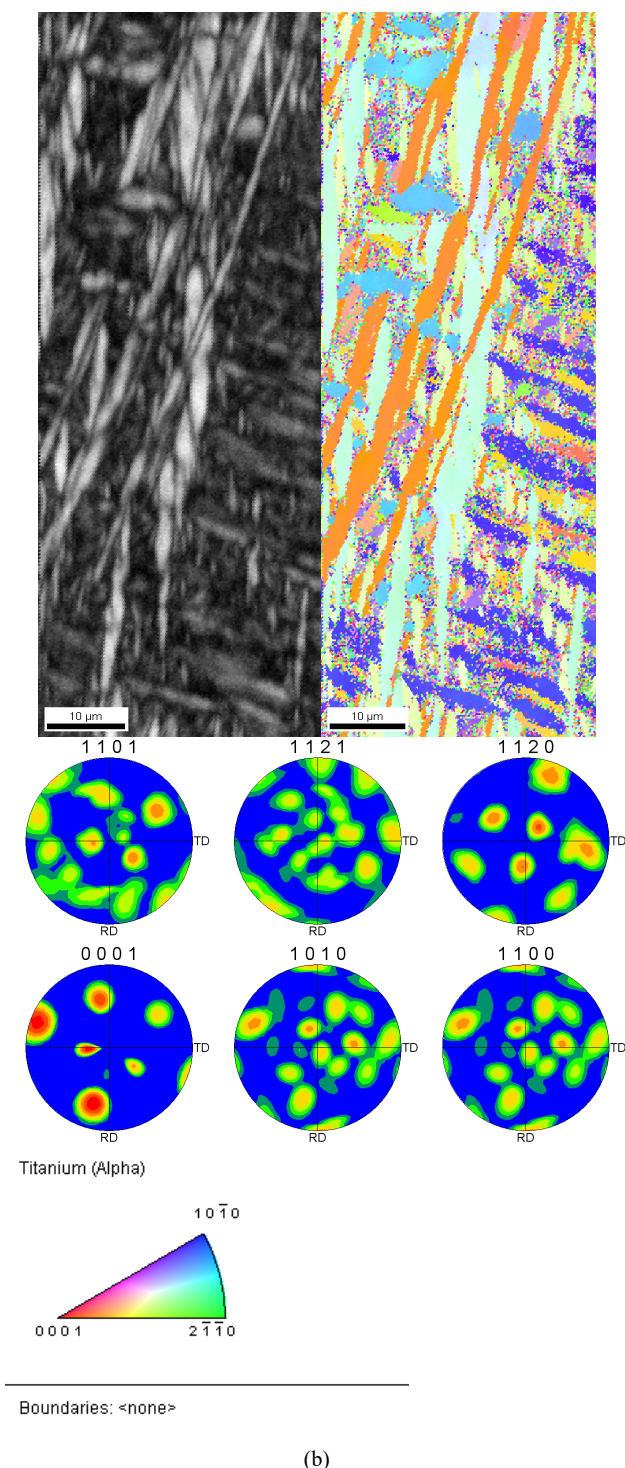


Fig. 5 EBSD analysis of the samples, SEM: a- sample 1; b-sample 2

IV. CONCLUSION

- Microstructure of Ti6Al4V (ELI) material produced by SLM under controlled oxygen content was analysed. Low level of oxygen was found in SLM samples.
- Structural analysis showed that fully martensitic

α' structure with high dislocation density and stacking faults was typical in the as-built samples. No β 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 $\langle 1-100 \rangle_{hcp}$ loading direction. Twin presence suggests the high internal stresses in the SLM material.
- Orientation texture - phase transition $\beta \rightarrow \alpha'$ was found in SLM Ti-6Al-4V. Parental β - phase showed columnar growth with highly pronounced texture $\langle 100 \rangle$. The texture of α' was quite weak.

ACKNOWLEDGMENT

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REFERENCES

- [1] A.M. Beese, B.E. Carroll, "Review of mechanical properties of Ti-6Al-4V made by laser-based additive manufacturing using powder feedstock", *JOM*, vol. 68, pp. 724-734, 2016.
- [2] E. Wielewski, C. R. Siviour and N. Petrinic, "On the correlation between macrozones and twinning in Ti-6Al-4V at very high strain rates," *Scripta Materialia*, vol. 67, pp. 229-232, 2012.
- [3] M. Simonelli, Y. Y. Tse, C. Tuck, "Further understanding of Ti-6Al-4V selective laser melting using texture analysis", *Journal of Physics : Conference Series*, vol.371, pp.1-4, 2012.
- [4] H. Miura, Y. Itoh, T. Ueamsu, K. Sato, "The influence of density and oxygen content on the mechanical properties of injection molded Ti-6Al-4V alloys", *Advances in Powder Metallurgy and Particulate Materials*, vol.1, pp.46-53, 2010.
- [5] A. M. Bauristhene, K. Mutombo, W. E. Stumpf, "Alpha case formation mechanism in Ti-6Al-4V alloy investment castings using YFSZ shell moulds", *Journal of the Southern African Institute of Mining and Metallurgy*, vol.113, pp.357-361, 2013.
- [6] W. Tirry, F. Coghe, L. Rabet and D. Schryvers, "Multi-scale observations of deformation twins in Ti6Al4V" in *EMC*, vol. 2: Materials Science, S. Richter, A. Schwedt, Ed., 2008, pp. 499-500.
- [7] J. Peirsa, W. Tirryb, B. Amin-Ahmadib, F. Coghe, P. Verleysena, L. Rabetc, D. Schryversb, J. Degriecka, "Microstructure of adiabatic shear bands in Ti6Al4V", *Materials characterization*, vol.75, pp.79 -92, 2013.
- [8] P. Krakhmalev, G. Fredricsson, I.Yadroitseva, N. Kazantseva, A. Du Plessis, I.Yadroitsev, "Deformation Behavior and Microstructure of Ti6Al4V Manufactured by SLM", *Physics Procedia*, vol. 83, pp. 778 – 788, 2016.
- [9] L. Bao, C. Schuman, J-S.Lecomte, M-J. Philippe, X. Zhao and C. Esling, "A Study of Twin Variant Selection and Twin Growth in Titanium", *Advanced engineering materials*. vol.13, no. 10, pp.928-932. 2011.
- [10] I.V. Egis, A. A. Barbeko, A. I. Horev, M. M. Martinova, E. B. Samarin, "Basic texture formation in VT18u titanium alloy", *Metal Science and Heat Treatment*, no 6, pp. 1-7, 1992.