The structural and texture analysis of titanium alloy Ti-6Al-4V samples obtained by direct metal deposition

S V Scvortsova, M A German* and I A Grushin

MAI, Volokolamskoe shosse 4, 125993, Moscow, Russia

* skvorcovasv@mati.ru

Abstract. The paper presents the results of structure and texture formation study in Ti-6Al-4V alloy samples, obtained by Direct Metal Deposition technology of additive manufacturing. The effect of annealing on the formation of the structure and texture in 3D-samples was investigated. It is shown that the samples have a two-phase structure of “basket weaving” in the initial state and after annealing. It is established that the samples have significant anisotropy of properties in two mutually perpendicular directions that could be explained by the presence of the crystallographic texture.

1. Introduction

Additive manufacturing is in rapid evolution at the present time [1]. One of the primary goals is to find the optimal powder formation process that could compete with foreign analogues. Issues of structural and texture formation during 3D-fusion, subsequent treatment and its effect on the product properties are fundamentally important.

There are two basic methods of additive manufacturing: Bed Deposition and Direct Deposition [2]. As opposed to Bed Deposition, wherein the layer of building material is formed on the platform surface ("bed"), in Direct Deposition the material is added to the melt pool using a focused powder stream to form a raised portion of material. To create the desired geometry, the substrate is manipulated using a computer-controlled positioning system. Gas and material are fed into the path of a heat source. Material feed angle can be altered to influence the build characteristics [1, 3].

Currently, the effect of 3D-method parameters on structure, mechanical properties and residual stresses has not enough been studied. Complex metallurgical phenomena occur during 3D-printing process and depend on treatment conditions, properties and powder parameters [4, 5].

Some papers [6, 7] shows that 3D-workpieces with subsequent treatment can reach the complex of properties close to ones, obtained by traditional industrial technology.

It is known that besides chemical composition and structure texture strongly influences the mechanical properties of semi-finished products (especially sheets) obtained by traditional technology. This fact should be taken into account. The crystallographic texture formation of additive manufactured samples is of immediate interest as the obtaining of favourable textures in 3D-products could be the efficient way to improve mechanical properties [8, 9].

The purpose of this work is to establish the pattern of structure and crystallographic texture formation and mechanical properties in Ti-6Al-4V alloy workpieces obtained by additive manufacturing with further heat treatment.
2. Materials and methods

Research was carried out using the samples cut out from the titanium alloy workpiece (124×12×70 mm). The workpiece was obtained by Direct Laser Metal Deposition (DLMD) from Ti-6Al-4V alloy powder. The chemical composition is presented in Table 1. Particle size was in the range of 60-80 µm.

| Alloying elements | Impurities |
|-------------------|------------|
| Al, V, Fe, C, O, N, H |           |
| 6,3 | 4,5 | 0,22 | 0,048 | 0,12 | 0,016 | 0,0059 |
| 5,5-6,5 | 3,5-4,5 | ≤0,25 | ≤0,08 | ≤0,13 | ≤0,03 | ≤0,0125 |

The outer appearance of the workpiece is shown in the Figure 1 a. Several kinds of samples were cut out of the workpiece: 20×12×8 mm – for metallographic and X-ray-structural analysis; 12×12×58 mm – for mechanical tests (Fig. 1 b). The following designations of external directions were agreed: GD – growth direction, HD – longitudinal growth direction, TH – transverse direction (Fig. 1 b).

Samples for metallographic analysis were prepared according to a standard technique [10].

Heat treatment of the samples in an air was performed in a SNOL-2.2,5.1,8/10/10-I3 electric furnace.

The microstructure of the samples was examined using an AXIO Observer.Alm (Karl Zeiss Jena, Germany) optical microscope at a magnification of 1000. We used bright-field microscopy in an air atmosphere. Images were analyzed with the NEXSYS ImageExpert Pro3.6 software package designed for quantitative phase analysis with various methods, including the intercept method.

X-ray analysis was carried out on the samples in three orthogonally related directions using the diffractometer DRON-4-07 with copper Kα radiation (Fig. 1 b). To perform quantitative description of crystallographic texture the inverse pole figures method (IPF) was used, which is based on the measurement of α- and β-phases integrated intensity of diffraction maximums [11]. IPF were constructed in the area of standard stereographic triangles. Moreover, at each pole \( N_{hkl} \) the calculated pole density \( P_{hkl} \) was indicated (Fig. 2) that was calculated by the Morris equation (1):

\[
P_{hkl} = \frac{I_{hkl}}{\sum_x A_{hkl} \left( \frac{I_{hkl}}{I_{hkl}^{st}} \right)}
\]

\( P_{hkl} \) – pole density of reflex \((hkl)\); \( A_{hkl} \) – normalization constant; \( I_{hkl} \) – integral intensity (\( I_{hkl}^{st} \) – integral intensity for non-texture standard sample).

The intensities calculation using the equation (1) avoids the necessity for X-ray photography conditions tracking [12].
Figure 2. Standard stereographic triangles of crystals with face-centered (c/α=1,6) (a) and body-centered cubic lattice (b), divided by areas $A_{hk}$ [12].

The hardness of samples was determined by the Rockwell technique using a BUEHLER Macromet 5100T device on an HRC scale at a load of 1.5 kN according to Standard GOST 9013-59.

Short-term mechanical tensile tests were performed in accordance with Standard GOST 1497–84 on a TIRATEST universal machine.

3. Results and discussion
At the first stage, the structure and hardness of samples in the initial state after 3D-printing were examined.

The study showed that the structure of samples obtained by DLMD-technology is two-phase mixture ($\alpha+\beta$) as distinguished from martensite structure, observed in Selective Laser Melting (SLM) samples [7], due to lower cooling rate. The structure consists of lamellar $\alpha$-phase with small amount of $\beta$-phase (Fig. 3 a, b) as confirmed by X-ray structural analysis (Fig. 3 d). In the longitudinal direction the structure of samples slightly differs. The presence of $\alpha$ fringes along the boundaries of the initial $\beta$ grains is observed, inside which the structure is close to “Widmanstätten” (Fig. 3 b).

During the laser temperature influence in 3D-printer the granules are consolidated through the liquid phase. 1,5-2 granules exist in liquid state simultaneously depending on the initial size (40-100 µm). The existence of metal above the melting point lasts a split second. Then the high-speed crystallization occurs ($10^3$-10$^8$°C/s) due to the small size of liquid bath [13].

The cast sample of Ti-6Al-4V alloy was observed to compare the structure and texture. The structure is presented by classic rough lamellar intragrain structure (Fig. 3 c).

Figure 3. Structure of Ti-6Al-4V alloy samples after 3D-printing in the growth direction plane (a) in the longitudinal growth direction plane (b) in the cast state (c) and the section of the diffractogram (d).
Hardness measurement in the initial state after 3D-printing showed the significant difference in values depending on the direction – 37 HRC in the growth direction and 32 HRC in the longitudinal growth direction. That gives us an opportunity to suppose the presence of crystallographic texture and hence the anisotropy of mechanical properties.

At the next stage, the samples in the initial state were subjected to mechanical tests. Obtained results confirm the high anisotropy of properties in different directions: the tensile strength is significantly higher in the longitudinal direction than in the growth direction of the workpiece (Table 2).

Table 2. Mechanical properties of 3D-samples in the initial state.

| Direction of samples cutting                  | σ, MPa | σ₀.2, MPa | δ, % | ψ, % |
|-----------------------------------------------|--------|-----------|------|------|
| Growth direction (GD)                         | 1030   | 980       | 18   | 46   |
| Longitudinal growth direction (LGD)           | 1100   | 1035      | 11   | 29   |

The presence of properties anisotropy is related to crystallographic texture. Consequently, at the next stage the inverse pole figures (IPF) for initial state samples were constructed.

The increased pole density of family of planes \{100\}_β is in the growth direction, and for the \(\alpha\)-phase - \{10T2\} \(\cap\) \{1120\} due to \(\beta\rightarrow\alpha\)-transition during cooling.

It is known that the nucleation of the low-temperature phase in metals and alloys with polymorphic transition is performed by shear displacement [14]. The further growth of formed nuclei can be occurred by shear displacement (quenching) or by diffusive mechanism (annealing). Shear mechanism of nucleation determines the presence of orientation relationship between \(\beta\)- and \(\alpha\)-phases [8].

In the process of obtaining strain semi-finished products from titanium alloys by traditional means texture type and intensity are defined by pattern, degree and temperature of strain. Hence in strain semi-finished products there is strain and dynamic recrystallization texture besides the \(\beta\rightarrow\alpha\)-transition texture formation [15, 16].

In the case of DLMD-method the material has no deformation effect. Hence as a result of \(\beta\rightarrow\alpha\)-transition there is only \(\alpha\)-phase transition texture to form, which is related to initial \(\beta\)-phase by Burgers orientation relationship [17], i.e. planes of \(\alpha\)-phase \{10T2\}, \{1120\} and \{2130\} are formed from the plane families \{100\} and \{111\} of \(\beta\)-phase (Fig. 4 a). In the IPF in the longitudinal growth direction the increase of pole density of planes family \{110\}_β (besides \{100\}_β) which forms the basis planes \{0001\}_α and pyramid \{10T1\}_α is observed.

The low pole density of \(\alpha\)-phase planes \{10T0\} should be noted. Their formation should also occur from \{110\} planes of \(\beta\)-phase (Fig. 4 b). High values of pole density of plane family \{112\}_β are observed and hence the planes \{10T3\}_α \(\cap\) \{10T0\}_α. The absence of characteristic component \{1122\}_α should be noted (Fig. 4 c).

The IPF analysis showed that there is some pole density increase of basis planes in the longitudinal growth direction, and \{10T2\}_α and \{1120\}_α planes in the growth direction. There is a case for anisotropy of strength in two orthogonally related directions.

We studied the cast sample texture cut out from the VT6 ingot obtained by industrial process to figure out the stage whereon the texture formation proceeds. According to research carried out the \(\beta\)-phase texture formed during the melt consolidation process is almost identical to 3D-workpiece \(\beta\)-phase texture.
The further cooling to the room temperature leads to $\alpha$-phase transition texture formation as a result of ($\beta \rightarrow \alpha$)-transition. The main texture components are similar to ones in 3D-workpiece (Fig. 5).
At the next stage the effect of annealing on the structure and texture formation and mechanical properties of DLMD-samples was examined. For heat treatment one of the most frequently used annealing condition for Ti-6Al-4V alloy was chosen: 800°C, 1 hour, air cooling. Annealing does not lead to any fundamental changes. Basket weave structure remains almost unchanged with insignificant increase of α-phase lamellas in the growth direction (Fig. 6). Hardness after heat treatment remains about 37 HRC in all directions.

**Figure 6.** Microstructure of Ti-6Al-4V alloy 3D-samples after annealing in the growth direction plane (a) in the longitudinal growth direction plane (b).

Annealing leads to some derating of strength values in the growth direction and in the longitudinal growth direction and to increase of plastic properties (Table 3). This is due to the fact of tension-relieving effect and some increase of structural constituents’ size. Anisotropy of properties remains in two orthogonally related directions.

| Direction of samples cutting | Heat treatment conditions | σ, MPa | σ_{0.2}, MPa | δ, % | ψ, % |
|-----------------------------|--------------------------|--------|--------------|------|------|
| Growth direction (GD)       | 800°C, 1 hour, air cooling | 990    | 930          | 19   | 52   |
| Longitudinal growth direction (LGD) |                        | 1060   | 1018         | 14   | 34   |

The IPF analysis showed that texture of β- and α-phases after annealing has no essential changes and its main components remain unchanged (Fig. 7). Some pole density increase of \{110\} β-phase is observed in the growth direction and its derating in the longitudinal growth direction that could related to recrystallization process.

**Figure 7.** Texture of Ti-6Al-4V alloy 3D-samples after heat treatment
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