Variant selection during \( \beta \) to \( \alpha \) transformation for Ti-6Al-4V alloy produced by Selective Laser Melting printing

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**Abstract:** During 3D printing by Selective Laser Melting (SLM) of the alloy Ti-6Al-4V, the phase transformation of BCC to HCP is associated with selection of particular crystallographic variants. In the present paper, the distribution of misorientation angle along grain boundaries was investigated by EBSD. It is revealed that the main misorientation pairs were located in the interval between 56° and 66°, which contains three theoretical misorientation angles, i.e. 60.00°, 60.83° and 63.26°. The observed misorientation axis/angle pairs were represented in the Rodrigues–Frank space with a certain tolerance angle, which illustrated clearly the distribution of the observed misorientations around the 5 theoretical values. The main peak of the misorientation pairs was observed for \([10 \bar{5} 5 \bar{3}]\)/ 63.26° with 43.8% frequency, followed by \([1 1 \bar{2} 0]\)/ 60° with 21.8% population, whereas a distribution in the absence of variant selection would have produced values of 18.2% and 36.4%, respectively.

1. Introduction

Since in 1934 Burgers [1] identified the specific orientation relationship (OR) between the parent body-centered cubic crystal structure and the hexagonal closed packed crystal structure during phase transformation, many researchers have performed detailed studies on the mechanism of variant selection in phase transformation, which is important for controlling the microstructure and the related mechanical properties.

Because of the symmetry of cubic and hexagonal structures in Ti-6Al-4V alloy, a single \( \beta \)-BCC crystal would transform 12 different \( \alpha \)-HCP variants. Theoretically, it is equally statistically possible for all 12 product orientations to nucleate and grow during phase transformation, which would imply that the phase transformation occurs without variant selection, giving rise to a weak or nearly random \( \alpha \) texture. However, in reality, due to different factors, the phenomenon of variant selection is often observed. The possible causes of variant selection include: the difference of lattice parameters between parent and product phase [2], externally applied loads and/or strain [3, 4], the specific nature of the pre-existing \( \alpha \) grains [5], the reduced transformed strain energy with variant clusters and special grain...
boundary in β grains [6-9]. Consequently, for the SLM printed Ti-6Al-4V alloy under consideration, the phenomenon of variant selection is intricately linked to phase transformation, which results in a specific crystallographic texture determining the final mechanical properties of the alloy.

In the present work, the texture of SLM Ti-6Al-4V alloy is studied by EBSD, the misorientation profile is examined along grain boundaries and compared with the theoretical distribution to assess the occurrence of variant selection. The Rodrigues – Frank Space (R – F Space) is employed to demonstrate clearly the misorientation pairs in 3D space.

2. Experiment

2.1. Materials
Ti-6Al-4V rod shaped samples were produced by selective laser melting without any post-processing. With the EDS element analysis, the normalized weight percentage of elements were: Al: 4.4%, V: 3.1% and Ti: 92.5%.

2.2. Microstructural characterization
With the defined sample coordinate system (x_s//scanning direction, x_f//building direction), the EBSD scans were acquired with a SEM of type Quanta 450F with field emission gun equipped with a fully automated EBSD device, operated under the following conditions: 20 kV accelerating voltage, 16.0 mm working distance with hexagonal scanning grid with step size of 4 μm. The standard preparation procedures were used for EBSD observation. Necessary cleanup procedures were conducted before post-processing with TSL ® Analysis software, version 7.8.

2.3. Misorientation in Rodrigues – Frank (R–F) Space
In R – F space, misorientation between two grains with rotation matrix Δg can be illustrated by rotation axis/angle pair, which is represented by a single point [10]. Based on the Burgers OR, the theoretical misorientation between product α can be depicted as reference points in the fundamental zone. In the present work, the observed misorientations will be presented as number density within a tolerance angle α. This number density is obtained by dividing the experimentally observed number fraction dr by the hypothetical number fraction dn, which assumes that there is no variant selection and all variants would obey the precise Burgers correspondence. The ratio of dr/dn representing the number density D(Δg) can be evaluated for the considered sample and interpreted as the degree of variant selection to the extent it deviated from unity. Matlab and MTEX toolbox [11] were employed for the calculation and plot.

3. Results and discussion

3.1. Microstructure and texture
Fig. 1 displays the microstructure and pole figures of the scanned product α phase and reconstructed parent β phase by ARPGE [12]. Because of the limited statistics of the scanned dataset, there are ~15% non-identified points in the reconstructed database. On the pole figures, the strongest peak is observed in the center position of the (1 1 2 0) PF, which implies that the building direction is predominantly parallel to the (1 1 2 0) pole, the corresponding β phase demonstrates a peak in the center of (0 0 1) and (1 1 1) pole. The maximum intensity of the (0 0 1) α PF is 1.868 mrd and 3.118 mrd for the (0 0 1) β PF.

For the SLM printing procedure, the β grains are formed during solidification and the β to α phase transformation occurs during cooling at fast rate. Based on the theory of solidification [13], the preferred growth direction is (0 0 1) for BCC crystallographic structure. In the meanwhile, the maximum thermal gradient is along the building direction. Hence, the β grains grow in the (0 0 1) direction along the building direction, forming the strong (0 0 1) texture, as observed in Fig. 1(b).
3.2. Misorientation distribution between product grains

Theoretically, the phase transformation from β phase to α phase follows the Burgers correspondence, i.e.: \(\{1 1 0\}_β \parallel \{0 0 0 1\}_α\); \(< 1 1 1 >_β \parallel < 1 1 2 0 >_α\). With the operation of the cubic and hexagonal crystal structural symmetry, there are 12 product variants from a single β parent grain, as listed in Table 1. Assuming absence of variant selection, Wang [14] figured out that only 5 different types of misorientation pairs between product were induced, cf. Table 2. It is seen from Fig. 2 (a) that there are 3 obvious peaks in the misorientation angle distribution in the following ranges: 8° – 12°; 56° – 66° and 88° – 94°, which corresponds to the 5 theoretical misorientation angles according to the Burgers orientation relation, cf Table 1. Fig. 2 (b) depicts the difference between experimental results and theoretical values for 5 different misorientation peaks, and the data are also shown in Table 2. It is clear that the misorientation pair \([10 5 5 \bar{3}] / 63.26°\) is the dominant type with percentage of 43.8%, followed by \([1 1 2 0] / 60°\) with 21.8%. From Fig. 2 and Table 2, it is concluded that the phenomenon of variant selection occurs during phase transformation from β phase to α phase. However, because of the experimental uncertainty, it is virtually impossible to distinguish the angular distances of the misorientations \([1 1 2 0] / 60°\), \([1 3 7 7 7 2.377 0.359] / 60.83°\) and \([1 0 5 5 3] / 63.26°\). Therefore, it is necessary to also consider the misorientation axis, which implies a representation in 3D Rodrigues-Frank space.

Fig. 2 (a) The distribution of misorientation angle along α/α grain boundaries; (b) the misorientation angle fractions compared to the 5 Burgers reference misorientations.
Table 1  12 possible α variants following the Burgers OR during phase transformation applying, the cubic symmetry operation and ensuing misorientation pairs between product variants

| Variants | Orientation relationship | Symmetry operation | Misorientation pairs from Variant A |
|----------|--------------------------|--------------------|-----------------------------------|
| A        | 〈i 0 1〉[0 0 0 1]       | 1. -1              |                                  |
| B        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 1 2 0] / 60°                  |
| C        | 〈0 1 1〉[0 0 0 1]       | 1. -1              | [1 1 2 0] / 60°                  |
| D        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 3 8 1 3 8] / 90°              |
| E        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 1 5 5 3] / 63.26°            |
| F        | 〈0 1 1〉[0 0 0 1]       | 1. -1              | [1 3 7 7 1 2 3 7 7 0.359] / 60.83° |
| G        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 3 7 7 1 2 3 7 7 0.380] / 90° |
| H        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 3 7 7 1 2 3 7 7 0.359] / 60.83° |
| I        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 3 7 7 1 2 3 7 7 0.359] / 60.83° |
| J        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 3 7 7 1 2 3 7 7 0.359] / 60.83° |
| K        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 3 7 7 1 2 3 7 7 0.359] / 60.83° |
| L        | 〈i 0 1〉[0 0 0 1]       | 1. -1              | [1 3 7 7 1 2 3 7 7 0.359] / 60.83° |

Table 2 Comparison of the number fractions of misorientation pairs according to the Burgers OR and for the current experiment

| Type Reduced axis/angle pairs | Burgers Correspondence | Experiment |
|------------------------------|------------------------|------------|
| I [0 0 0 1] / 10.53°        | 9.1%                   | 1.2%       |
| [1 1 2 0] / 60°            | 18.2%                  | 21.8%      |
| [1 3 7 7 1 2 3 7 7 0.359] / 60.83° | 36.4%                  | 20.0%      |
| [1 3 7 7 1 2 3 7 7 0.359] / 60.83° | 18.2%                  | 43.8%      |
| [1 3 7 7 1 2 3 7 7 0.359] / 90° | 18.2%                  | 13.2%      |

3.3. Rodrigues - Frank Space

The misorientation Δg of two orientations g<sub>a</sub> and g<sub>b</sub> is given by Δg = g<sub>b</sub>g<sub>a</sub><sup>-1</sup>. The Rodrigues vector R corresponds to the misorientation axis/angle pair (r, θ), as the vector is defined by: R = tan(θ/2)v, whereby v is a unit vector parallel to the axis r. Fig. 3 (a) and (e) show the distribution of all misorientation pairs in correlated condition as observed in the scanned dataset, and non-correlated condition as calculated from the scanned dataset while assuming that in the non-correlated condition, all possible variants would appear near the checked variants. It is clear that all measured misorientation pairs are located around the theoretical values, and the three important misorientation pairs with rotation angle between 56° ~ 66° can be identified readily.
Fig. 3 (a) and (e): The experimental correlated and non-correlated misorientation pairs distribution in R - F space; (b)~(d): the distribution of misorientation pairs between variants with different tolerance angle in correlated condition, respectively; (f)~(h): the distribution of misorientation pairs between variants with different tolerance angle in non-correlated condition, respectively.

With the number fraction of misorientation pairs $dr$ and $dn$ in correlated and non-correlated conditions, respectively, the number density of different theoretical types can be expressed as $D(Δg) = dr/dn$. If there is no variant selection during $β$ to $α$ transformation, the value of $D(Δg)$ is equal to unit. By allowing different tolerance angle, the actual values of $D(Δg)$ may change, cf Table 3. Consistent with the misorientation profile along grain boundaries, the number density results conclude that the phenomenon of variant selection occurs during phase transformation following SLM printing.

During $β$ to $α$ phase transformation, the lattice cells of BCC crystal structure with lattice parameters of 3.196 Å have to transform to the lattice cells of HCP crystal structure with parameters of $a = 2.943 Å$, $c = 4.680 Å$ [15]. The difference of lattice parameters between $β$ and $α$ phase induces transformation strain. In order to minimize the transformed strain, different variants may cluster together during growth, this phenomenon of variants clustering is known as self-accommodation and leads to the triangular morphology in microstructure [16]. Another reason for variant selection is the occurrence of residual stresses and defects during printing, such as lack of fusion and micro-voids. Such defects and residual stresses would change the stresses field within samples, resulting in the preferred nucleation and growth of some variants.

| Type Reduced axis/angle pairs | $ω = 2$ | $ω = 5$ | $ω = 8$ |
|------------------------------|---------|---------|---------|
| 1 [0 0 0 1]/ 10.53°          | 1.4     | 0.5     | 0.4     |
| [1 1 2 0]/ 60°               | 3.8     | 1.9     | 1.6     |
| [1.377 1 2.377 0.359] / 60.83° | 2.0     | 0.9     | 0.7     |
| [10 5 5 3]/ 63.26°           | 4.7     | 2.2     | 1.8     |
| [1 2.38 1.38 0]/ 90°         | 3.1     | 1.4     | 1.3     |

4. Conclusion

Through the study of microstructure and misorientation distribution of a Ti-6Al-4V alloy produced by Selective Laser Melting printing, the phenomenon of variant selection has been verified during $β$ to $α$ phase transformation. The misorientation angle profile demonstrates that the main peak in the interval is located between 56° and 66°, which contains 3 major misorientation pairs. The Rodrigues – Frank space is used to clarify the difference among the misorientation pairs of [1 1 2 0]/ 60°, [1.377 1 2.377 0.359]/ 60.83° and [10 5 5 3]/ 63.26°. The main misorientation pair is [10 5 5 3]/ 63.26° with a number density of 4.7 (with a tolerance angle of 2°), followed by [1 1 2 0]/ 60° with 3.8 number density. It can be concluded that variant selection has occurred during phase transformation following SLM printing.

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