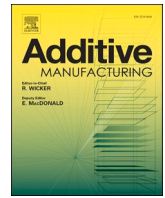


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## Research Paper

# Stability of crystallographic texture in laser powder bed fusion: Understanding the competition of crystal growth using a single crystalline seed

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## ABSTRACT

In metal additive manufacturing, crystallographic orientation control is a promising method for tailoring the functions of metallic parts. However, despite its importance in the fabrication of texture-controlled functional parts, the stability of the crystallographic texture is not widely discussed. Herein, the crystallographic texture stability under laser powder bed fusion was investigated. Two methodologies were employed. One is that a laser scanning strategy was alternately changed for a specific number of layers. The other is a “seeding” experiment in which single-crystalline substrates with controlled crystallographic orientations in the building (*z*-) direction and the *xy*-plane (perpendicular to the building direction) were used as the starting substrate. The transient zone width, where the crystallographic orientation was inherited from the layer beneath, was analyzed to evaluate the texture stability. The crystallographic direction of the seed within the *xy*-plane, rather than the building direction, determined the transient zone width, i.e., the texture stability. In particular, the texture in the newly deposited portion was stable when the laser scanning direction matched the  $\langle 100 \rangle$  orientation in the underneath layer, otherwise the crystal orientation switched rapidly, such that the  $\langle 100 \rangle$  orientation was parallel to the scanning direction. Interestingly, the crystallographic orientation along the building direction in the underneath layer hardly impacted the stability of the texture. Therefore, for the first time, it has been clarified that the  $\langle 100 \rangle$  orientation in the scanning direction, rather than the building direction, was preferentially stabilized, whereas the orientation in the other directions secondary stabilized.

## 1. Introduction

In recent years, metal additive manufacturing (AM) has unleashed new possibilities for the fabrication of high value-added products and has attracted the most significant attention in various metal processing technologies. Generally, since AM is characterized by its flexible shaping ability, it has been utilized for the fabrication of a variety of products, including those with hollow structures, three-dimensional complex porous bodies, and custom-made products [1–4]. Moreover, in some cases, AM has been combined with topology optimization [5,6]. Further, in addition to controlling the shape of products, recent studies have revealed the promising potential of metal AM for controlling the crystallographic texture of metallic materials [7–14]. Crystallographic

texture, which is independent of the shape of products, is an important factor contributing to the functionality of metallic materials, such as the mechanical properties. The most recent study demonstrated that laser powder bed fusion (LPBF), the most common powder bed fusion process in AM, facilitated the fabrication of products with diverse crystallographic textures, ranging from randomly crystallographically oriented polycrystalline to highly textured single-crystalline-like microstructures [7,14]. This was achieved by varying the balance between the thermal gradient at the solid/liquid interface, the migration velocity of the solid/liquid interface, and the melt pool shape [9,10].

Highly textured materials, including single-crystalline materials, are highly desired for specific applications requiring extremely superior mechanical properties because materials with preferential

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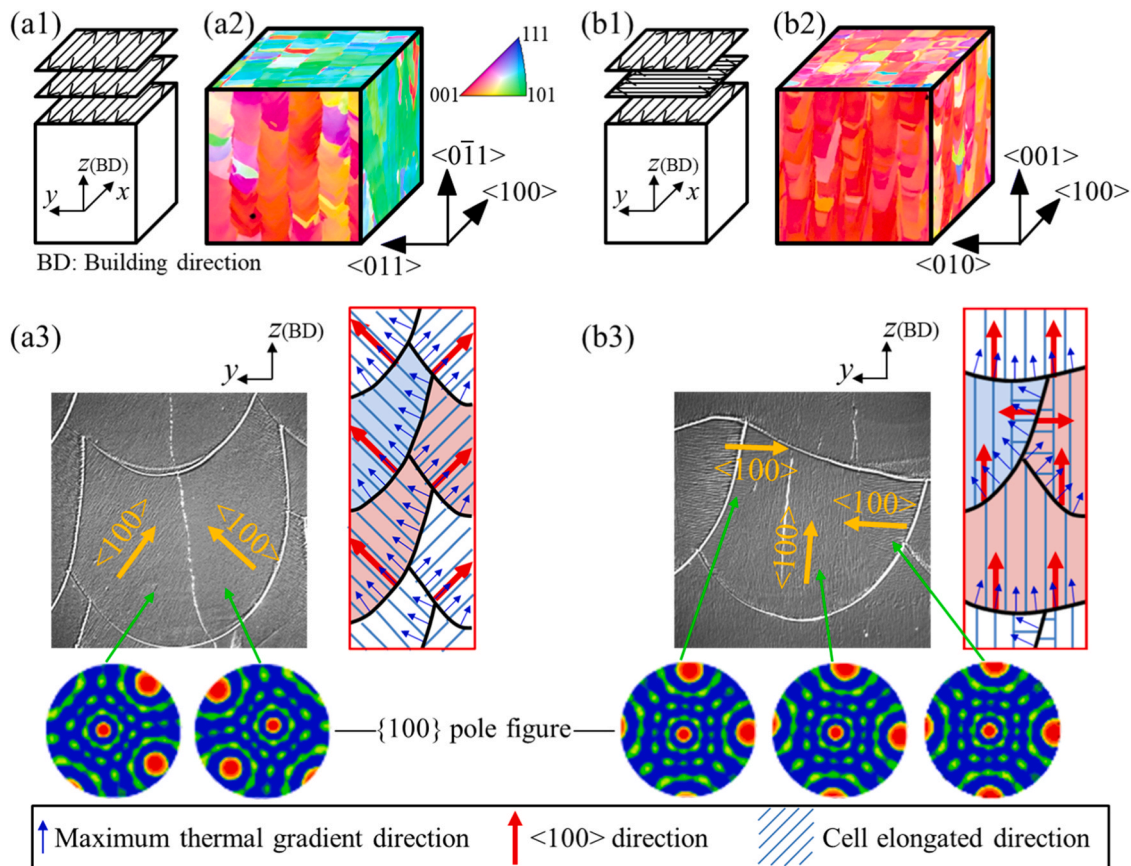
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crystallographic orientations feature mechanical anisotropies, such as Young's modulus [15,16], yield stress and elongation [17], fatigue resistance [18], and creep resistance [19]. Although there are several reports on the formation of a single-crystalline-like texture by LPBF, the formation mechanism has not been elucidated thus far. Our recent investigation revealed that a laser scanning strategy (SS), the combination of laser scanning patterns between layers, impacts the evolution of the crystallographic texture in a beta-type Ti-15Mo-5Zr-3Al alloy with a body-centered cubic (bcc) structure [14]. The SS\_X in which the laser was only scanned bidirectionally along the  $x$ -axis (Fig. 1(a1)) evolves the texture in which  $\langle 011 \rangle$  and  $\langle 100 \rangle$  orient along the building (BD) and scanning directions, respectively (Fig. 1(a2)). However, the SS\_X in which the laser was bidirectionally scanned with a rotation of  $90^\circ$  between the layers (Fig. 1(b1)) produces the texture in which  $\langle 100 \rangle$  orients along the BD and duplicated scanning directions (Fig. 1(b2)). Herein, these two types of crystallographic textures are referred to as  $\{011\}_z\langle 100 \rangle_x$  and  $\{001\}_z\langle 100 \rangle_x$  textures, respectively. A similar texture evolution using LPBF was reported for the X-scan [9, 12,20] and XY-scan [12,21,22] strategies using metallic materials with cubic crystal structures (bcc or face-centered cubic (fcc)).

The scanning strategy-dependent texture development was briefly discussed in a previous paper [14] that the feasibility of an epitaxial growth from the adjacent track and underneath layer is important. The preferential growth direction of the columnar cells for the fcc and bcc metals is parallel to  $\langle 100 \rangle$  [23]. Therefore, the crystallographic texture is formed so that the consistency of  $\langle 100 \rangle$  cell growth between adjacent tracks and between underneath layers is established, as shown in Fig. 1 (a3, b3). In such a way, in the SS\_X, the cell elongation direction in the

cross-section of the melt pool is finally fixed along the  $\pm 45^\circ$  directions from the BD (Fig. 1(a3)). As a result, the identical orientation is achieved in the left and right regions in the melt pool. Conversely, in the SS\_XY, the  $\pm 45^\circ$  cell growth cannot be achieved owing to the rotation of the laser scanning direction by  $90^\circ$  between layers. If the  $\langle 100 \rangle$ -oriented cell growth occur at  $\pm 45^\circ$  direction from the BD in a layer under the SS\_XY,  $\langle 100 \rangle$  cannot grow in a continuous direction in the next layer. Therefore, the  $\langle 100 \rangle$ -oriented cell growth at angles of  $0^\circ$  and  $90^\circ$  from the BD (Fig. 1(b3)) so that the melt pools generated by X-scan and the next Y-scan have a consistent crystallographic orientation, resulting in the evolution of the  $\{001\}_z\langle 100 \rangle_x$  texture. In this way, the importance of the crystal growth with a continuous crystallographic orientation between the tracks and between the layers, i.e., an epitaxial growth, was reported. However, little is understood about the initial stage of developing behavior of crystallographic orientation before the epitaxial growth is achieved.

In this study, we highlight the two above-mentioned types of crystallographic textures and investigate the stability of the crystal orientation by changing the scanning strategy between the layers. In addition, single-crystalline materials with varied and controlled crystallographic orientations are used as seeds to systematically investigate the stability of the crystallographic texture developed by LPBF. The beta-type Ti-15Mo-5Zr-3Al alloy authorized for use as a biomaterial by the International Organization for Standardization (ISO) (ISO 5832-14) is utilized because tailoring the mechanical properties via controlling the textures is a promising strategy for enabling functional improvement in medical metallic implants.



**Fig. 1.** (a1, b1) Schematic representation of SS\_X and SS\_XY in the LPBF process and (a2, b2) the developed distinctive crystallographic texture depending on the scanning strategy, referred to as  $\{011\}_z\langle 100 \rangle_x$  and  $\{001\}_z\langle 100 \rangle_x$  textures, respectively. (a3, b3) Typical cellular microstructure observed in the  $yz$ -cross-section of the products and schematics of the mechanism for the texture evolution based on the microstructural observation. This figure was reproduced under the terms of the Creative Commons Attribution 4.0 International license (CC-BY 4.0). Taken from [14].

## 2. Experimental procedures

### 2.1. Experimental design

An ingot with a nominal composition of Ti–15Mo–5Zr–3Al (wt%) was used as the starting material. The powder for LPBF fabrication was developed utilizing Ar gas atomization (Osaka Titanium Technologies Co. Ltd., Japan). The obtained powder was spherical, with a median particle size of 26  $\mu\text{m}$ . Rectangular specimens with a bottom dimension of 5 mm  $\times$  5 mm were manufactured using an LPBF apparatus (EOS M 290, EOS, Germany) equipped with a 400 W Yb fiber laser. The fabrication was performed in an Ar atmosphere. Two types of laser-beam scan strategies—SS\_X and SS\_XY—were used to fabricate the samples.

The crystallographic texture stability during LPBF fabrication was investigated using two methods. The first involved the fabrication of rectangular samples by alternating the scan strategies at a specific height interval during the fabrication process. The scan process parameters, i. e., the laser power, scanning speed, layering thickness, and scan pitch were set to 360 W, 1200 mm/s, 60  $\mu\text{m}$ , and 100  $\mu\text{m}$ , respectively. Under this condition, two alternating scan strategy schemes were attempted: 1) alternative change in SS\_X and SS\_XY, and 2) alternative change in SS\_X and SS\_Y. Here, SS\_Y denotes that the laser was scanned bidirectionally on the y-axis only. The changes in the scan strategy were conducted every 30 layers. A pure Ti plate was used as the starting plate.

The second method used a Ti–15Mo–5Zr–3Al single-crystal with different crystal orientations from the base plate (seed). The Ti–15Mo–5Zr–3Al single-crystal alloy was prepared using the optical floating zone method (SCI-MDH-20020, Canon Machinery, Japan). The crystal growth rate was set at 2.5 mm/h and the single crystal was grown under an Ar atmosphere. The detailed preparation method of the single crystal was described in our previous paper [24]. The crystal orientation was determined using X-ray back-reflection Laue diffraction, and the plates were cut from the obtained single crystal by electron discharge machining. Eight single-crystalline plates with different crystal geometries were prepared for this study. The details of the crystal geometries will be described in Section 3.2 (Fig. 4). Both SS\_X and SS\_XY strategies were conducted considering the relationship between the crystallographic orientation of the seed and the LPBF-deposited parts.

The microstructures of the prepared samples were examined using optical microscopy (OM; BX60, Olympus, Japan) and field-emission scanning electron microscopy (FE-SEM; JSM-6500, JEOL, Japan). The surface of the sample was mechanically polished using emery papers. Subsequently, final polishing was conducted with colloidal silica. In addition, texture analysis was conducted using an electron backscatter diffraction (EBSD) mounted on the FE-SEM.

### 2.2. Quantitative evaluation of texture development behavior

To quantitatively analyze the crystallographic texture evolved, the degree of crystal orientation was calculated by the Euler angle measured by SEM-EBSD analysis. In the EBSD analysis, the crystal orientation at each measured pixel along the observed direction was characterized by Euler angles, which are determined as follows:

$$h = \sin\Phi \sin\varphi_2, \quad k = \sin\Phi \cos\varphi_2, \quad l = \cos\Phi \quad (1)$$

Under the specimen coordinate axes ( $x_s$ ,  $y_s$ , and  $z_s$ ) which are set to the direction perpendicular to the specimen surface and the [100], [010], and [001] directions in the grain are denoted as  $x_c$ ,  $y_c$ , and  $z_c$  in the EBSD analysis, the Euler angles of  $\varphi_1$ ,  $\Phi$ , and  $\varphi_2$  are defined as the

angles between the line of intersection of the  $x_s y_s$  and  $x_c y_c$  planes denoted as N and  $x_s$ , the angle between  $z_s$  and  $z_c$ , and the angle between N and  $x_c$ , respectively. Using the  $\langle hkl \rangle$  indices measured at each pixel, the degree of crystal orientation of each particular crystal orientation  $\langle uvw \rangle$  relative to the observation direction was quantified. First, the angle between the observation direction and  $\langle uvw \rangle$  was estimated. For example, in the cubic crystal, there were six and twelve equivalent orientations for  $\langle 100 \rangle$  and  $\langle 110 \rangle$ , respectively. Thus, the smallest angle between the observation direction and one of the equivalent orientations of  $\langle uvw \rangle$  was defined as  $\alpha_{\langle uvw \rangle}$ . Under this definition, the value of  $\alpha_{\langle uvw \rangle}$  is evaluated at each measured pixel and the average value of  $\cos^2 \alpha_{\langle uvw \rangle}$  is defined as the degree of crystal orientation for  $\langle uvw \rangle$ , denoted as  $P_{\langle uvw \rangle}$ . Using the aforementioned definition, the maximum value of  $P_{\langle uvw \rangle}$  is 1, and the closer it is to 1, the stronger the crystal orientation alignment of  $\langle uvw \rangle$  along the observation direction. Note, however, that the smaller  $P_{\langle uvw \rangle}$  is not always indicative of a weak texture. For example, the degree of crystal orientation  $P_{\langle 100 \rangle}$  was smallest when  $\langle 111 \rangle$  was oriented along the observation direction at all measurement points, thus signifying a single crystal. At this time,  $P_{\langle 100 \rangle}$  was equal to 1/3. Similarly, the degree of crystal orientation  $P_{\langle 110 \rangle}$  was the smallest when  $\langle 100 \rangle$  was oriented along the observation direction at all measurement points when  $P_{\langle 110 \rangle}$  was 1/2. If the sample is polycrystalline and exhibits a completely random texture, the values of  $P_{\langle 100 \rangle}$  and  $P_{\langle 110 \rangle}$  can be calculated as follows:

$$P_{\langle 100 \rangle \text{ in random polycrystal}} = \frac{\int_S \cos^2 \alpha_{\langle 100 \rangle} dS}{\int_S dS} = \frac{\iint_T z^2 \sqrt{1 + \left(\frac{\partial z}{\partial x}\right)^2 + \left(\frac{\partial z}{\partial y}\right)^2} dx dy}{\iint_T \sqrt{1 + \left(\frac{\partial z}{\partial x}\right)^2 + \left(\frac{\partial z}{\partial y}\right)^2} dx dy} = \frac{\int_0^{\frac{\pi}{2}} d\theta \int_0^{\frac{1}{\sqrt{\cos^2 \theta + 1}}} r \sqrt{1 - r^2} dr}{\int_0^{\frac{\pi}{2}} d\theta \int_0^{\frac{1}{\sqrt{\cos^2 \theta + 1}}} \frac{r}{\sqrt{1 - r^2}} dr} = \frac{1}{3} + \frac{2}{\sqrt{3}\pi} = 0.7008 \quad (2)$$

$$P_{\langle 110 \rangle \text{ in random polycrystal}} = \frac{\int_S \cos^2 \alpha_{\langle 110 \rangle} dS}{\int_S dS} = \frac{\iint_T \frac{(x+z)^2}{2} \sqrt{1 + \left(\frac{\partial z}{\partial x}\right)^2 + \left(\frac{\partial z}{\partial y}\right)^2} dx dy}{\iint_T \sqrt{1 + \left(\frac{\partial z}{\partial x}\right)^2 + \left(\frac{\partial z}{\partial y}\right)^2} dx dy} = \frac{\int_0^{\frac{\pi}{2}} d\theta \int_0^{\frac{1}{\sqrt{\cos^2 \theta + 1}}} \frac{r(\cos\theta + \sqrt{1 - r^2})^2}{2\sqrt{1 - r^2}} dr}{\int_0^{\frac{\pi}{2}} d\theta \int_0^{\frac{1}{\sqrt{\cos^2 \theta + 1}}} \frac{r}{\sqrt{1 - r^2}} dr} = \frac{1}{3} + \frac{\sqrt{3} + 1}{\sqrt{3}\pi} = 0.8354 \quad (3)$$

where  $S$  is a triangle projected on a spherical surface with a radius of 1 with the [001], [101], and [111] orientations as vertices, and  $T$  is the projection of  $S$  on the  $xy$ -plane.  $z$  represents a spherical surface (hemisphere) with radius 1 centered on the origin, and  $z = \sqrt{1 - x^2 - y^2}$ . Details of the definition of  $x$ ,  $y$ ,  $z$  are provided in Fig. S1, Supplementary

$$P_{\langle uvw \rangle \text{ in random polycrystal}} = \frac{1}{3} + \frac{3u^2 + (2\sqrt{3} - 3)(v^2 - w^2) + 4\{uv + (1 + \sqrt{3} - \sqrt{6})vw + (\sqrt{6} - 2)wu\} - 1}{\sqrt{3}\pi} \quad (4)$$

**Information.** Accordingly, a more generalized estimation of  $P_{\langle uvw \rangle}$  in the sample with a completely random texture can be calculated by the following equation:

where,  $u \geq v \geq w \geq 0$ , and the magnitude of the vector  $\langle uvw \rangle$  is normalized to 1, i.e.  $u^2 + v^2 + w^2 = 1$ .

### 3. Results

#### 3.1. Variations in texture when changing the scanning strategy

Fig. 2(a–f) depicts the crystal orientation maps measured in the samples in which the scan strategy was periodically changed. The expected textures formed under the SS\_X, SS\_XY, and SS\_Y used are  $\{011\}_z \langle 100 \rangle_x$ ,  $\{001\}_z \langle 100 \rangle_x$ , and  $\{011\}_z \langle 01\bar{1} \rangle_x$ , respectively. The EBSD analysis was conducted on the yz-section perpendicular to the x-scanning direction. In Fig. 2, the positions at which the scan strategy was changed are indicated by the horizontal gray lines and arrows. It must be emphasized that the transient behavior of the texture differed depending on the changing scheme of the scan strategy. When focusing on the boundary where the texture was altered, the boundary was almost straight in the transition from SS\_X to SS\_XY (red dashed lines and arrows in Fig. 2(a–c)). Conversely, the boundary formed irregular waves in the transition from SS\_XY to SS\_X (blue dashed lines and arrows in Fig. 2(a–c)). In addition, the area fractional where the  $\{011\}_z \langle 100 \rangle_x$  and  $\{001\}_z \langle 100 \rangle_x$  textures were evolved is significantly different, despite, the scan strategy was alternatively changed by the same period (every 30 layers). This can be clearly recognized in Fig. 2(b) and 2(c) in which the area fraction exhibiting the  $\{001\}_z \langle 100 \rangle_x$  texture (red parts) is considerably wider than that of the  $\{011\}_z \langle 100 \rangle_x$  texture (green parts).

In the alternative change between SS\_X and SS\_Y shown in Fig. 2(d–f), the nature of the evolved texture itself was varied by the alternative change in scan strategy. In the SS\_X, the crystal orientation alignment of  $\langle 100 \rangle$ ,  $\langle 011 \rangle$ , and  $\langle 01\bar{1} \rangle$  is expected along the x-, y-, and z-directions, as shown in Fig. 1(a2). Similarly, in the SS\_Y, the evolution of the crystal orientation alignment of  $\langle 110 \rangle$ ,  $\langle 100 \rangle$ , and  $\langle 01\bar{1} \rangle$  is expected along the x-, y-, and z-directions. Thus, only the  $\langle 01\bar{1} \rangle$  orientation along the z-direction (green color in Fig. 2(f)) was expected, however, the unexpected  $\{001\}_z \langle 100 \rangle_x$  texture (red color in Fig. 2(f)) in which  $\langle 100 \rangle$  was aligned along the z-direction, was evolved in the transient zone between SS\_X and SS\_Y with a relatively large area fraction.

To discuss the transient behavior of the texture more quantitatively, the degrees of crystal orientation  $P_{\langle 100 \rangle}$  and  $P_{\langle 110 \rangle}$  were evaluated along the z-direction (BD) in both samples, as shown in Fig. 3. Gray lines and blue/red dashed lines indicate the points at which the scanning strategy was changed and the transient zone was ended, respectively, as similar to Fig. 2. The end point of the transient zone was defined based on the variation in the  $P_{\langle 100 \rangle}$  and  $P_{\langle 110 \rangle}$  values; the point where the P-value for the expected crystallographic direction under a certain scanning strategy exceeded the values for random orientation. For example, at the change in scan strategy from SS\_XY to SS\_X ( $\langle 110 \rangle // z$  is expected), the end point was determined when  $P_{\langle 110 \rangle}$  along the z-direction exceeded the random value (blue dashed lines in Fig. 3(a)); at the change in scan strategy from SS\_X to SS\_XY ( $\langle 100 \rangle // z$  is expected), it was determined when  $P_{\langle 100 \rangle}$  along the z-direction exceeded the random value (red dashed lines in Fig. 3(a)). Interestingly, the transient zone width formed in the alternative change between SS\_XY and SS\_X was not uniform; the width at the SS\_XY to SS\_X transition was  $\sim 790 \mu\text{m}$ , which was much wider than that at the SS\_X to SS\_XY transition of  $\sim 230 \mu\text{m}$ .

Considering the alternative change of SS\_X and SS\_Y shown in Fig. 3(b), a frequent increase and decrease in  $P_{\langle 100 \rangle}$  and  $P_{\langle 110 \rangle}$  were monitored. This is owing to the formation of an unexpected  $\{001\}_z \langle 100 \rangle_x$  texture despite the expected orientation under SS\_X and SS\_Y is  $\langle 110 \rangle // z$ . During the alternative change of SS\_X and SS\_Y, only one

pair of SS\_XY appears locally at the switching point of scanning strategy. This local SS\_XY drastically altered crystallographic orientation into the  $\{001\}_z \langle 100 \rangle_x$  texture, indicating a high stability of the  $\{001\}_z \langle 100 \rangle_x$  texture under the SS\_XY. Then, the texture showed transition to  $\langle 110 \rangle // z$  textures.

The expected texture in each scanning strategy was finally formed, however, the transient zone formation behavior until the expected texture appeared differed largely depending on the combination and order of the scanning strategies that were switched. This indicates that the crystallographic texture stability measured by the transient zone formation behavior is influenced by the crystallographic orientation relationship between the pre-solidified substrate and newly deposited parts (expected texture). To systematically understand the texture stability, the “seeding” experiment was performed using single-crystalline substrates and the behavior of transient zone formation or crystallographic orientation inheritance was analyzed.

#### 3.2. Variations in the transient zone width (texture stability) depending on the combination of crystal orientation in single-crystalline substrate and scanning strategy

We used single-crystalline substrates as a seed for LPBF fabrication. The crystal orientation geometry was systematically altered along x-, y-, and z-directions as schematically described in Fig. 4. Eight different controlled crystal orientations were selected as a seed for fabrication using the SS\_X and SS\_XY. In the fabrication using the SS\_X, in addition to the single-crystalline substrate in which the orientation correlated entirely with that in the  $\{011\}_z \langle 100 \rangle_x$  texture (Fig. 4(a1)), three other single-crystalline substrates in which only one orientation (x-, y-, or z-orientation) corresponded to that in the  $\{011\}_z \langle 100 \rangle_x$  texture (Fig. 4(a2–a4)) were prepared. Hereinafter, they are referred to as xyz-, x-, y-, and z-common SS\_X substrates, respectively. Similarly, the xyz-, x-, y-, and z-common single-crystalline substrates were prepared for SS\_XY with respect to the  $\{001\}_z \langle 100 \rangle_x$  texture (Fig. 4(b1–b3)). In addition, the single-crystalline substrate in which any of the three x-, y-, and z-orientations did not coincide with those in the  $\{001\}_z \langle 100 \rangle_x$  texture (Fig. 4(b4)) was also prepared for the SS\_XY (hereinafter the uncommon SS\_XY substrate).

Figs. 5 and 6 depict the crystal orientation maps showing the variations in texture evolution depending on the crystal orientation in the single-crystalline seed for the SS\_X and SS\_XY products, respectively. In the figures, the corresponding variations in  $P_{\langle 100 \rangle}$ ,  $P_{\langle 110 \rangle}$ ,  $P_{\langle 111 \rangle}$ , and  $P_{\langle 112 \rangle}$  are also displayed below the crystal orientation maps. The graphs on P-value in the upper row indicate the variation in initial (single-crystalline seed) orientation, and those in the lower row indicate the variation in the expected orientation to be formed under each scanning strategy. Here, the time point at which the  $P_{\langle 100 \rangle}$  and  $P_{\langle 110 \rangle}$  satisfied all of the following states was defined as the end point of the transient zone: for SS\_X,  $P_{\langle 100 \rangle}$  along the x-direction and  $P_{\langle 110 \rangle}$  along the y- and z-directions exceeded the values for random orientation; for SS\_XY,  $P_{\langle 100 \rangle}$  along the x-, y-, and z-directions exceeded the values for random orientation.

In the xyz-common SS\_X sample, the evolution of the  $\{011\}_z \langle 100 \rangle_x$  texture was confirmed without the formation of a transient zone, as shown in Fig. 5(a). Similarly, the  $\{001\}_z \langle 100 \rangle_x$  texture was evolved in the xyz-common SS\_XY sample (Fig. 6(a)). In these cases, the P-values in all directions remained higher than those in randomly oriented polycrystalline. The crystal orientations in the single-crystalline substrate were inherited into the newly deposited portion, indicating successful “seeding” under LPBF. However, the formation of the transient zone of the texture was observed in all the other cases. Importantly, the width of the transient zone of the texture was considerably different depending on the crystal geometry of the single-crystalline substrate and the scan strategy, as hypothesized in the previous section. Table 1 summarizes the measured transient zone width.

The seeding experiment using single-crystalline substrates derived

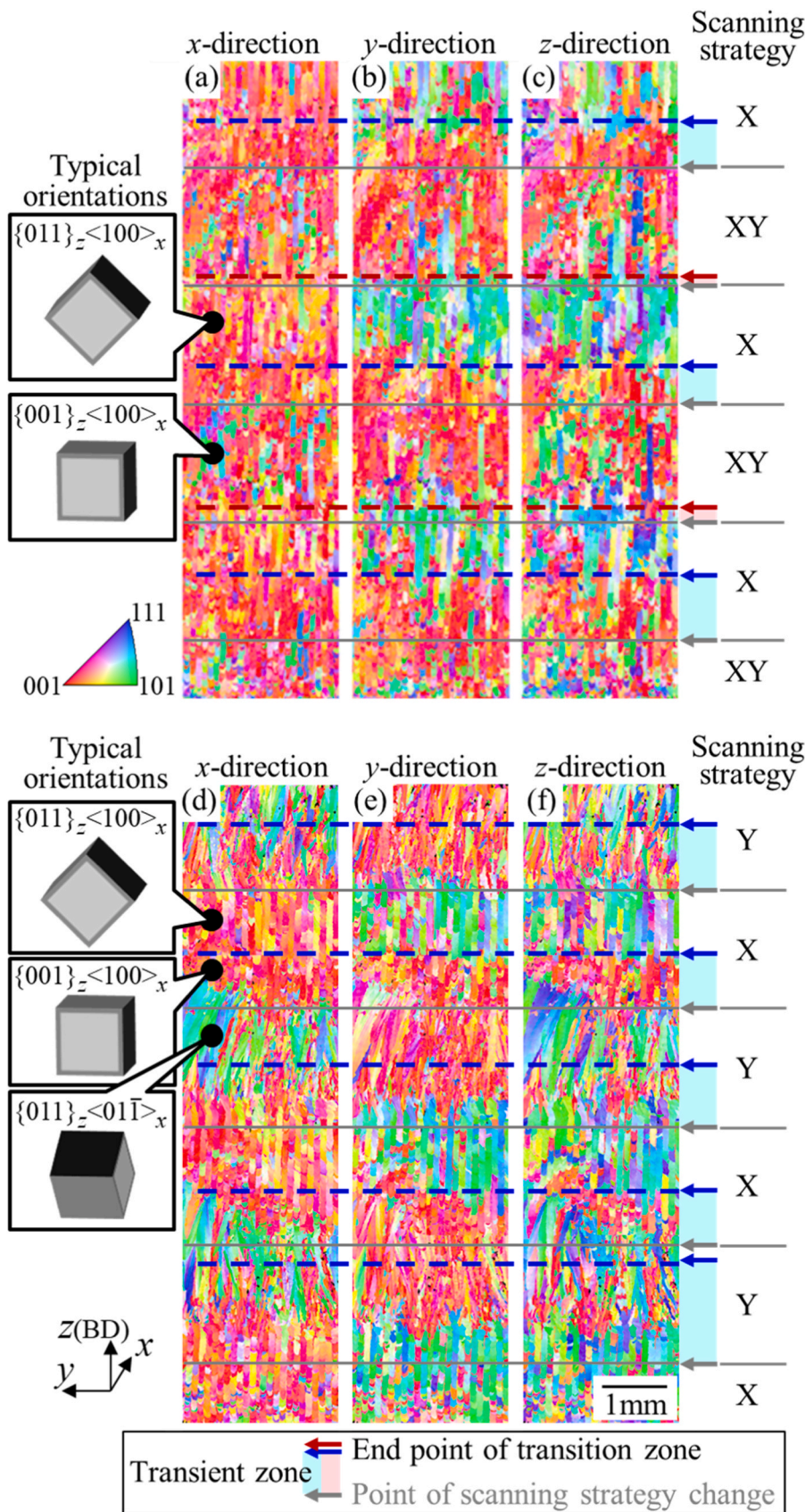
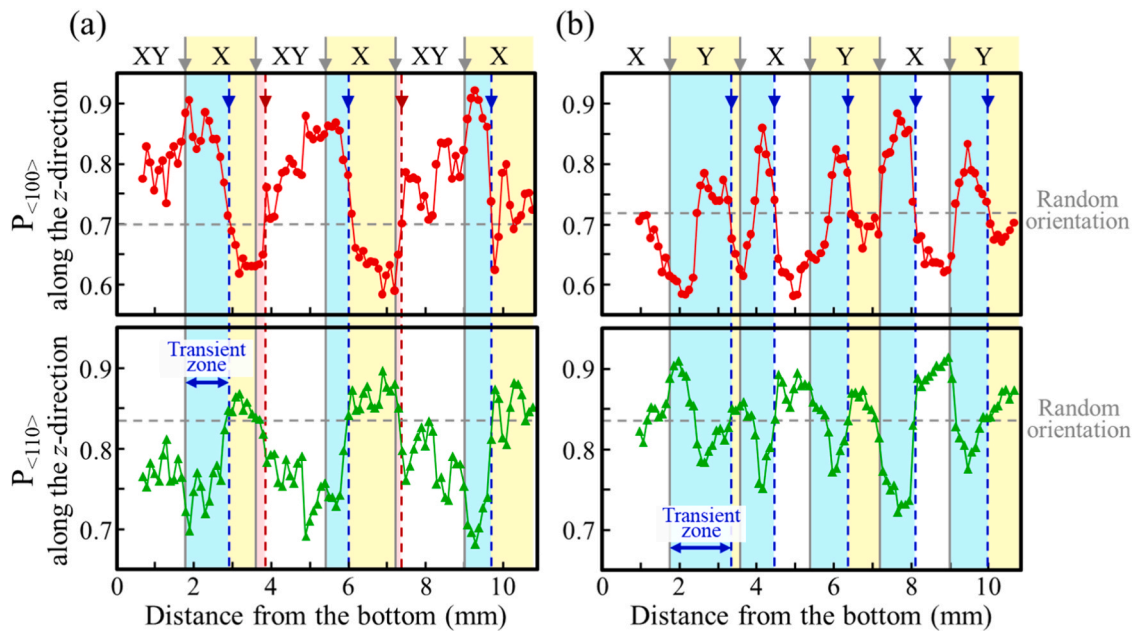
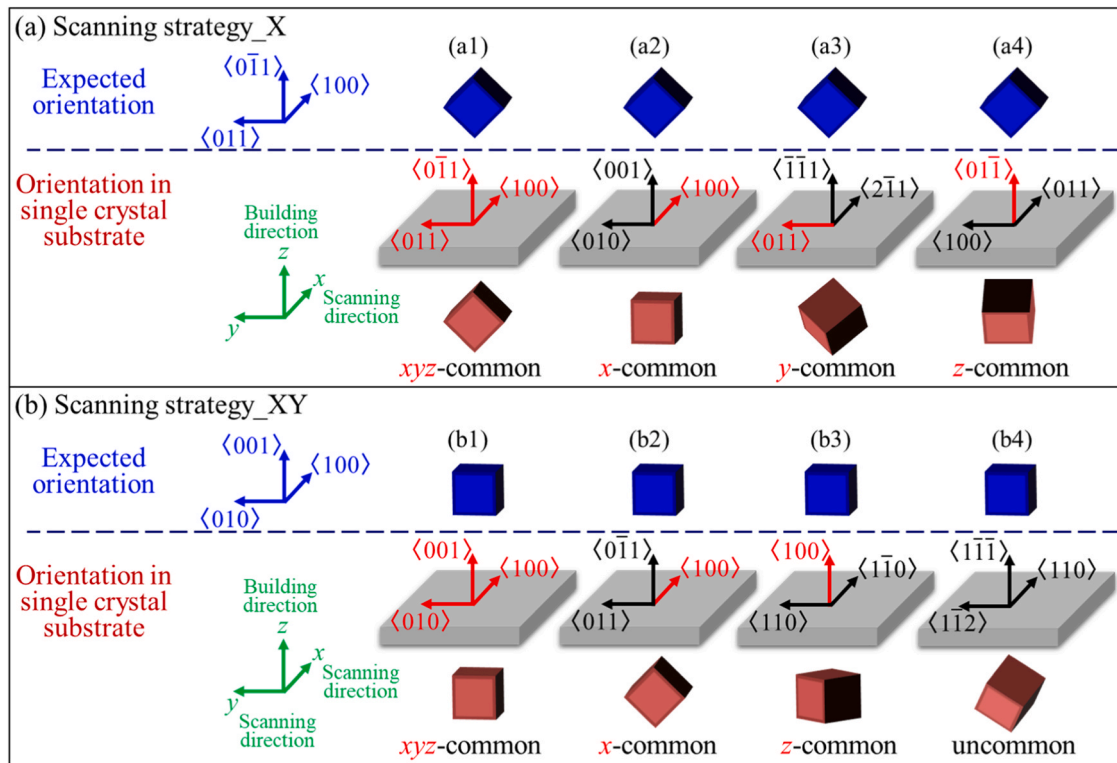


Fig. 2. Crystal orientation maps showing the variation in texture by changing the scanning strategy during the fabrication of the samples. (a–c) Alternative change in the SS\_X and SS\_XY and (d–f) alternative change in the SS\_X and SS\_Y. The observation was conducted on the yz-plane and the crystal orientation colors indicated those along the (a, d) x-, (b, e) y-, and (c, f) z-directions, respectively. The positions where the scanning strategy was changed are indicated by horizontal gray lines, and the positions where the resultant change in texture occurred are indicated by blue and red dashed lines.



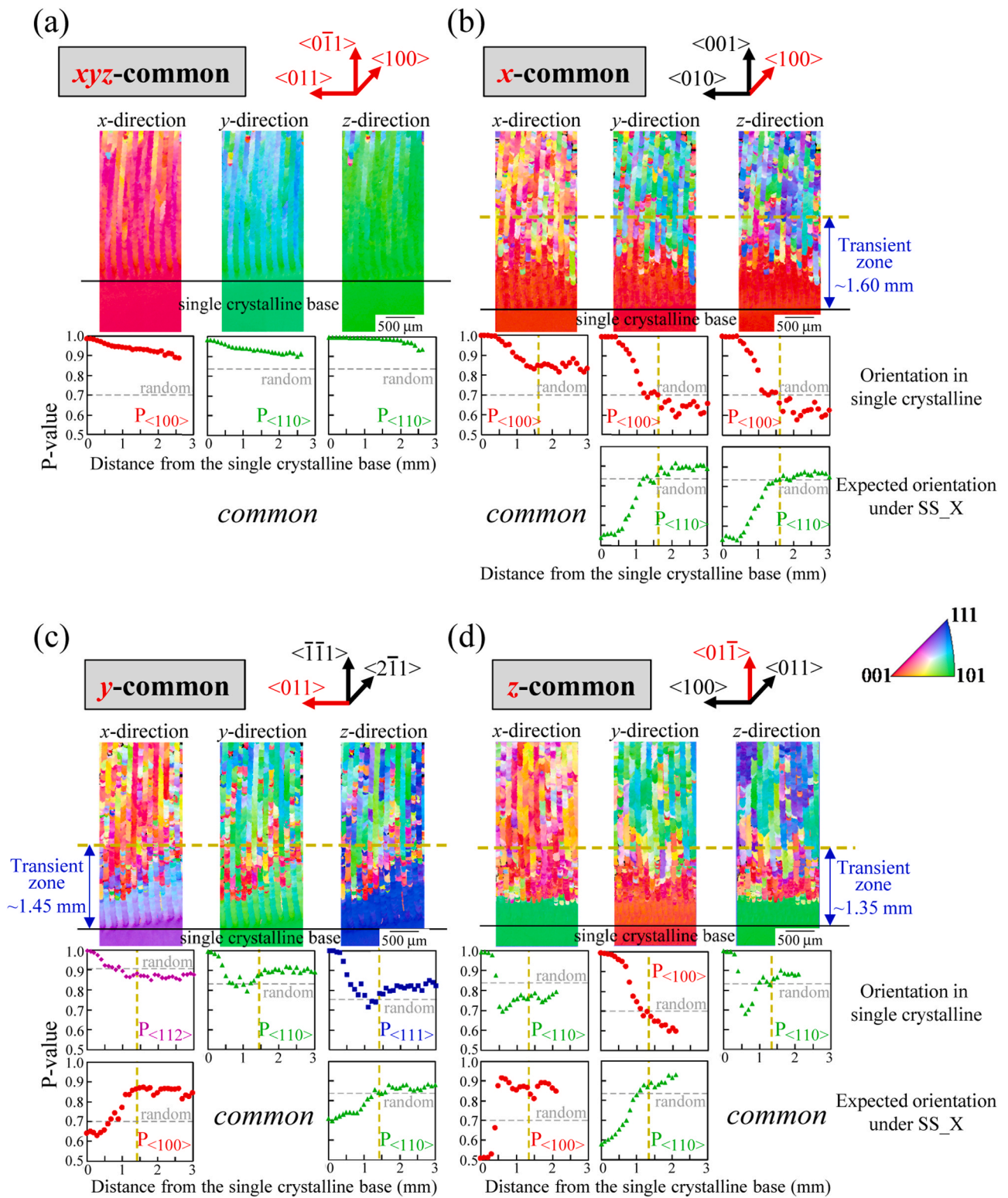
**Fig. 3.** Variation in the degree of crystal orientation alignment  $P_{\langle 100 \rangle}$  and  $P_{\langle 110 \rangle}$  along the z-direction in the following samples: (a) SS\_X/SS\_XY and (b) SS\_X/SS\_Y. The positions where the scanning strategy was changed are indicated by vertical gray lines, and the positions where the resultant change in texture occurred are indicated by blue and red dashed lines.



**Fig. 4.** Eight different geometries of the single crystal used as a base plate in the experiment to clarify the variation in the epitaxial growth behavior of the columnar cells during the LPBF process.

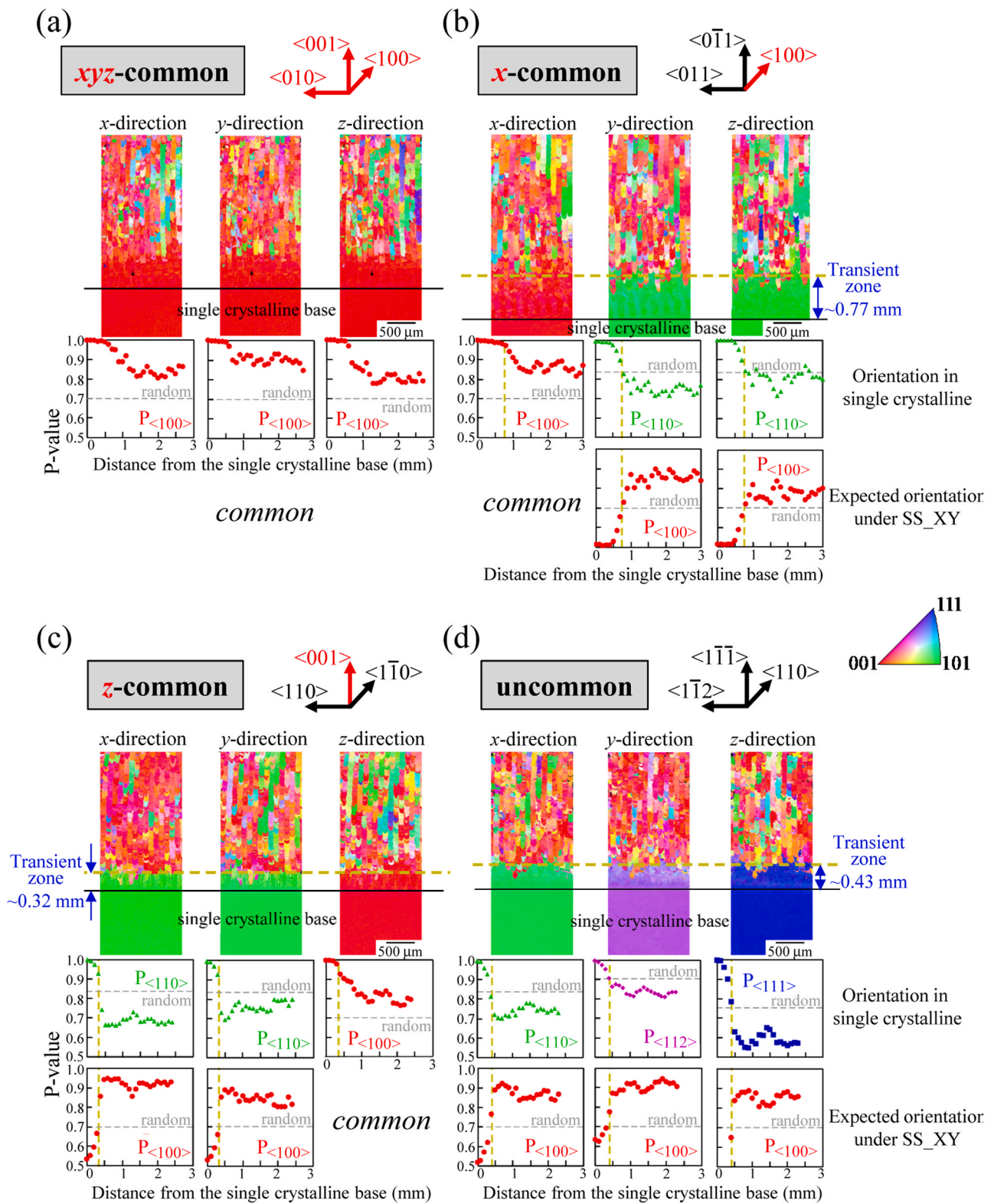
the following two important findings. 1) The transient zone width is wider in the x-, and y-common samples than those in the z-common samples, in both the SS\_X and SS\_XY fabrications. Comparing between the x-, and y-common samples, transient zone width is wider in the x-common sample. 2) In the case where the laser scanning directions (x-direction in SS\_X and x- and y-directions in SS\_XY) were parallel to the

$\langle 100 \rangle$  direction in the substrate, the crystal orientation tended to be inherited over a certain distance, otherwise the crystal orientation switched rapidly, such that the  $\langle 100 \rangle$  orientation was parallel to the scanning direction(s). This conclusion was quantitatively confirmed by demonstrating the variation in the transient zone width as a function of the deviation angle of the crystal orientation along the BD (Fig. 7(a)) or



**Fig. 5.** Crystal orientation maps showing the variation in the developing texture depending on the geometry of the crystal orientation of the starting plate in the fabrication with SS.X. The starting plates are (a) xyz-common, (b) x-common, (c) y-common, and (d) z-common single-crystalline plates, respectively. The microstructure observation was conducted on the yz-section, and the crystal orientation along the x-, y-, and z-directions are displayed, as indicated in the figure. Below the maps, the corresponding variations in  $P_{\langle 100 \rangle}$ ,  $P_{\langle 110 \rangle}$ ,  $P_{\langle 111 \rangle}$  and/or  $P_{\langle 112 \rangle}$  are also displayed.





**Fig. 6.** Crystal orientation maps showing the variation in the developing texture depending on the geometry of the crystal orientation of the starting plate in the fabrication with SS\_XY. The starting plates are (a) xyz-common, (b) x-common, (c) z-common, and (d) uncommon single-crystalline plates, respectively. The microstructure observation was conducted on the yz-section, and the crystal orientation along x-, y-, and z-directions are displayed, as indicated in the figure. Below the maps, the corresponding variations in  $P_{\langle 100 \rangle}$ ,  $P_{\langle 110 \rangle}$ ,  $P_{\langle 111 \rangle}$ , and/or  $P_{\langle 112 \rangle}$  are also displayed.

**Table 1**

The difference in the width of the transient zone of the texture measured in Figs. 5 and 6, which is varied depending on the geometry of the crystal orientation of the starting plate and scanning strategy.

SS_X	xyz-common	x-common	y-common	z-common
Difference in angle along z-direction (BD) (deg.)	0	45	35.3	0
Difference in angle along scanning direction (deg.)	0	0	35.3	45
Transient zone width (mm)	–	1.60	1.45	1.35
SS_XY	xyz-common	x-common	z-common	Uncommon
Difference in angle along z-direction (BD) (deg.)	0	45	0	55
Difference in angle along scanning direction (deg.)	0	0	45	35
Transient zone width (mm)	–	0.77	0.32	0.43

the laser scanning direction(s) (Fig. 7(b)) between the single-crystalline substrate and the expected  $\{011\}_z<100>_x$  or  $\{001\}_z<100>_x$  textures. In the case of SS\_XY, since there are equivalent two scanning directions, the smaller values of the two are plotted as the deviation angles for the scanning direction. The width of the transient zone decreased as the deviation angle increased with respect to the scanning direction (Fig. 7(b)). Conversely, the difference in the deviation angle along the BD exhibited no correlation with the width of the transient zone (Fig. 7(a)).

#### 4. Discussion

##### 4.1. Formation of the transient zone and the stability of the crystallographic texture in LPBF

In both the SS\_X and SS\_XY fabrications, the transient zone width is wider when the laser scanning direction is parallel to the  $<100>$  orientation in the substrate as typically shown in Figs. 5(b) and 6(b). In this case, the crystal orientation tended to be inherited over a relatively longer distance and subsequently transitions to the expected texture for the scanning strategy selected. When the laser scanning direction deviates from  $<100>$  in the substrate, the crystal orientation switched rapidly, so that  $<100>$  orients in the scanning direction(s) (Figs. 5(c and d) and 6(c and d)). These findings strongly demonstrate that the crystallographic  $<100>$  orientation along the “scanning direction” stabilized within a relatively short time and lead the texture formation in the LPBF process. The stability of the texture in which  $<100>$  was parallel to the scanning direction was significantly higher than that of other textures irrespective to the scanning strategy (SS\_X or SS\_XY). This insight is completely different from the frequently seen explanation that the crystal orientation along the z-direction (BD) determines the texture evolution in LPBF [25–27]. Actually, the initial crystal orientation along the building direction has less effect on the texture stability in the case of LPBF process. The stability of the crystallographic orientation is

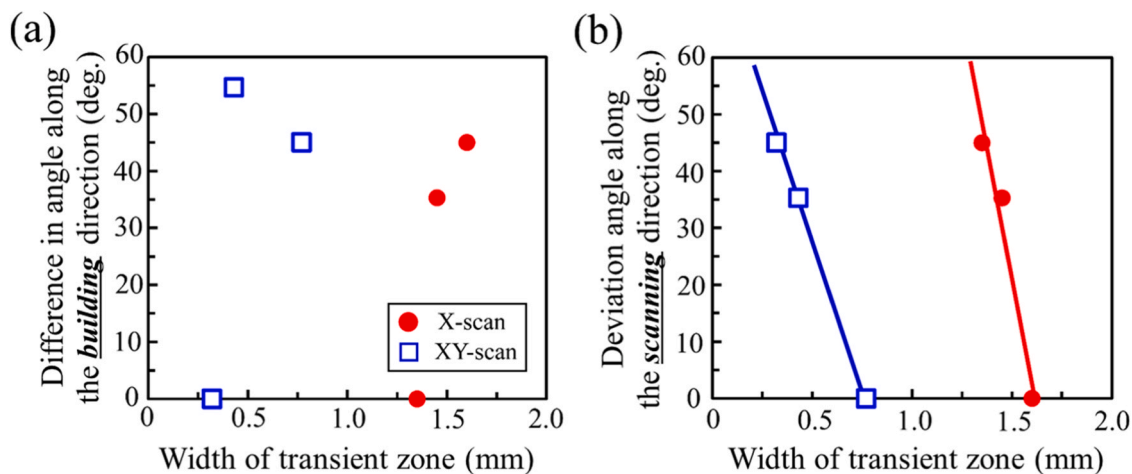
determined by the orientation relationship (difference) between the scanning direction and the  $<100>$  direction in the pre-solidified underneath portion that acts as the substrate.

The transient zone width was narrower in the SS\_XY samples than in the SS\_X sample. In the case of SS\_XY, the two-dimensional cellular growth alternatively occurs on  $yz$ - (for X-scan) and  $xz$ -planes (for Y-scan). As a result, the  $<100>$  shows strong tendency to orient in the normal directions on the two planes (i.e., the x- and y-scanning directions), which simultaneously fix the alignment of the remaining  $<001>$  to the z-direction (BD). As a result, the  $\{001\}_z<100>_x$  texture in SS\_XY evolves more rapidly than the  $\{011\}_z<100>_x$  texture in SS\_X.

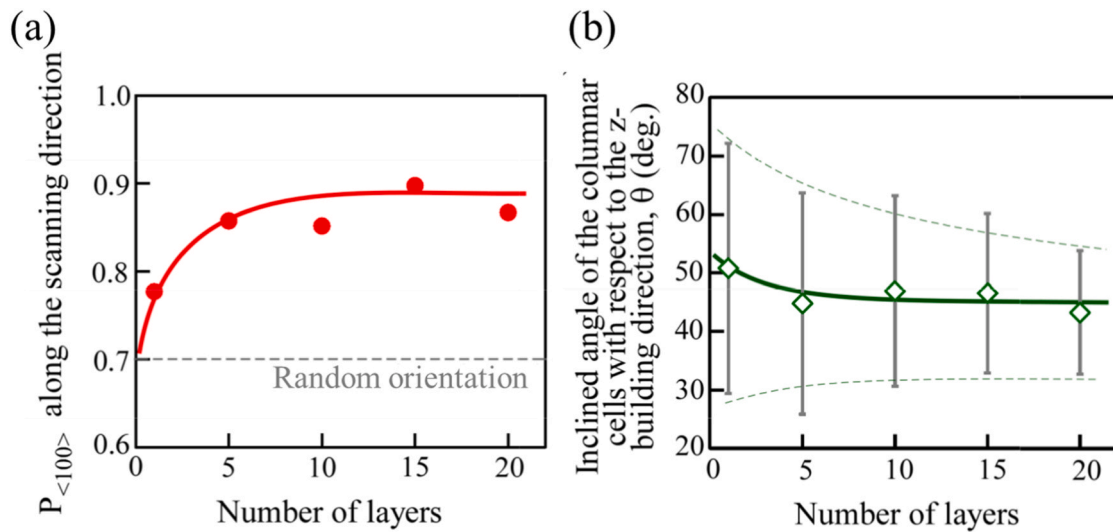
The high stability of the  $\{001\}_z<100>_x$  texture under the SS\_XY would largely influence the transient zone formation in the fabrication with the alternative change of SS\_X and SS\_Y (Figs. 2(d–f) and 3(b)). The alternative change of SS\_X and SS\_Y was well reproduced by the seeding experiment using the combination of SS\_X and single-crystalline substrate with z-common orientation (Fig. 5(d)). The  $\{001\}_z<100>_x$  texture that is unexpected for the SS\_X and SS\_Y, while expected for SS\_XY was evolved following to the switch of the scanning strategy. In the alternative change of SS\_X and SS\_Y, SS\_XY is locally established which is made up of only two layers at the switching point of scanning strategy. Even this locally established SS\_XY domain induced unexpected  $\{001\}_z<100>_x$  texture owing to its high stability.

##### 4.2. Evolution mechanism of the crystallographic texture depending on the scanning strategy, considering the competitive and epitaxial crystal growth in the melt pool

The  $<100>$  orientation in the substrate being parallel to the laser scanning direction(s) was demonstrated to be important for the stability of crystallographic texture evolution during LPBF. In order to understand the crystallographic stability not only in the laser scanning direction but also in the other directions that are typically considered in



**Fig. 7.** Relation between the width of the transient area and the difference in the crystal orientation angles in the base single crystal and the developed  $\{011\}_z<100>_x$  or  $\{001\}_z<100>_x$  texture in SS\_X or SS\_XY, respectively, focused along the (a) building direction (BD), and (b) scanning direction.



**Fig. 8.** (a) Variations in  $P_{\langle 100 \rangle}$  and (b) the inclined angle of the elongation direction of the columnar cells with respect to the  $z$ -building direction ( $\theta$ ) with the number of stacking layer, measured on the  $yz$ -plane of the sample fabricated by SS\_X. The fabrication was conducted on a pure  $\alpha$ -Ti plate with the hcp structure to minimize the influence of the epitaxial growth impacted from the starting plate.

the LPBF process, the texture transition behavior was analyzed at the initial stage of the deposition. The LPBF fabrication was performed with a simple SS\_X. The variations in  $P_{\langle 100 \rangle}$  along the  $x$ -scanning direction (Fig. 8(a)) and the inclined angle ( $\theta$ ) of the elongation direction of the  $\langle 100 \rangle$ -oriented columnar cells with respect to the BD (Fig. 8(b)), were measured as a function of the number of stacking layers. The  $yz$ -cross-section that is perpendicular to the  $x$ -scanning direction was used to appropriately analyze the cellular angles. The fabrication was conducted on a pure  $\alpha$ -Ti plate with the hexagonal close-packed (hcp) structure to avoid the influence of the epitaxial growth from the base plate.

The variation in  $P_{\langle 100 \rangle}$  shown in Fig. 8(a) demonstrated that the  $\langle 100 \rangle$  orientation along the  $x$ -scanning direction rapidly stabilized within the first few layers of deposition. On the other hand, the inclined angle of the columnar cells exhibited a wide distribution between  $\sim 30^\circ$  and  $\sim 70^\circ$  at the initial stage, and gradually converged in the  $\pm 45^\circ$  direction as the number of stacking layers increased. Consequently, the  $\{011\}_z \langle 100 \rangle_x$  texture stabilized and epitaxial growth occurred, as reported in the previous paper [14]. These results strongly indicated that at the initial stage of deposition, the crystal orientation in the scanning direction was first stabilized, then the orientation ( $\langle 100 \rangle$ -oriented cell growth direction) within the melt pool cross-section was secondary determined. The inclined angle of the columnar cells of  $\pm 45^\circ$  is considered to be advantageous for the reduction of interfacial energy at the center of the melt pool, where the solidification front of the right and left sides encounters. Therefore, the orientation perpendicular to the  $x$ -direction is adjusted so that the crystallographic misorientation in the right and left sides of the melt pool reduces, leading to the  $\pm 45^\circ$  cells from BD and  $\langle 110 \rangle // z$  orientation.

In the SS\_XY, in contrast, the fact that  $\langle 100 \rangle$  orientation in the scanning direction is important as similar to the previous situation would lead to competition for crystal orientations. When the scanning direction was changed from X-scan (the texture where  $\langle 100 \rangle // x$ -direction and  $\langle 011 \rangle // y$ -direction are stable) to Y-scan (the texture where  $\langle 100 \rangle // y$ -direction and  $\langle 011 \rangle // x$ -direction are stable), the  $\langle 011 \rangle$  orientation in the pre-solidified layer formed by X-scan and  $\langle 100 \rangle$ -orientation generated by Y-scan competed with each other in the  $y$ -direction. The crystal orientation was determined through the competition of crystal growth. Since  $\langle 100 \rangle$  grows faster [23],  $\langle 100 \rangle$  could remain in the  $y$ -direction, whereas  $\langle 011 \rangle$  was eliminated. In SS\_XY, the competition also occurred in the  $x$ -direction, resulting in the texture in which  $\langle 100 \rangle$  oriented in  $x$ - and  $y$ -directions, and resultantly the

$z$ -directions as well.

#### 4.3. Application of the crystallographic texture control

By exploiting the feature that metal AM enables the scan strategy to be altered in a site-selective manner within a part, a promising methodology for enhancing the functionality of structural parts is to three-dimensionally collocate elements with different crystallographic textures in order to tailor the mechanical properties as desired. To achieve this, it is necessary to superimpose an element with different crystallographic orientations on the underneath layer. Such a strategy must be of great help to functionalization of biomedical devices such as orthopedic and/or dental implants because the implant devices should adopt to the complicated stress applied in vivo with simultaneously suppressing a stress shielding [28] on surrounding bone tissues. It is desired to tailor mechanical properties e.g., Young's modulus depending on the location in the part and the direction. The findings regarding the texture stability obtained in this study are beneficial to achieve the series stacking of elements with different textures in the vertical (building) direction. By increasing the strength of the texture formed, it is expected that the transient zone width can be predicted uniformly. Strengthening the texture is one of the challenges in future research. In addition, the investigation of the texture stability in other alloy systems is needed to utilize this strategy in a wide range of industries.

## 5. Conclusions

In this study, the stability of the crystallographic texture during LPBF fabrication was investigated. The following conclusions were drawn:

- (1) In SS\_X in which the  $\{011\}_z \langle 100 \rangle_x$  texture formed,  $\langle 100 \rangle$  was first stabilized in the laser scanning ( $x$ -) direction. Within the  $yz$ -plane in the melt pool cross-section, the  $\langle 100 \rangle$ -elongated cell gradually aligned in the direction of  $\pm 45^\circ$  from the BD, resulting in reduced interface energy in the melt pool center.
- (2) In SS\_XY in which the  $\{001\}_z \langle 100 \rangle_x$  texture formed,  $\langle 100 \rangle$  and  $\langle 011 \rangle$  competed to grow in the laser scanning direction. Because of the more rapid growth rate in the  $\langle 100 \rangle$  direction,  $\langle 100 \rangle$  remained in both the scanning directions ( $x$ - and  $y$ -directions), and thus the remaining  $\langle 001 \rangle$  was fixed in the  $z$ -direction.

- (3) The stability of the crystallographic texture varied depending on the orientation in the substrate. In the case where the laser scanning direction was parallel to the  $\langle 100 \rangle$  direction in the substrate, the crystal orientation was likely to be inherited. Interestingly, however, the crystallographic orientation along the z-direction (BD) in the substrate did not impact the stability of the texture. Thus, the  $\langle 100 \rangle$  orientation in the scanning direction determined the texture formation, irrespective of SS\_X or SS\_XY.
- (4) Using a single crystal as a seeding substrate, it was possible to produce a single crystalline region under the LPBF process. The crystallographic orientation in the seed must be selected by considering the stability of the texture depending on the scanning strategy.

The formation of the crystallographic texture during LPBF fabrication is largely determined by the relationship between the laser scanning direction(s) and crystal orientation in the underneath solidified layer. This is peculiar to layer-by-layer fabrication, which is impacted by both the influence of the underneath layer and the solidification behavior in the melt pool itself. It is expected that functional devices, such as biomedical implants, with a configuration of differently textured portions will be created by carefully considering the stability of the texture.

#### CRediT authorship contribution statement

**Takuya Ishimoto:** Methodology, Validation, Investigation, Data curation, Writing - review & editing. **Koji Hagihara:** Investigation, Data curation, Writing - original draft, **Kenta Hisamoto:** Investigation, Data curation, Visualization, **Takayoshi Nakano:** Conceptualization, Writing - review & editing, Supervision, Project administration, Funding acquisition.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.addma.2021.102004](https://doi.org/10.1016/j.addma.2021.102004).

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