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Microtexture developed during Laser Metal Deposition (LMD) of Ti-6Al-4V

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Abstract

Additive manufacturing of metallic objects has attracted significant attention in a broad range of sectors including aerospace, defense and biomedical. Whereas one of the main advantages of this technique is, from a manufacturing point of view, its versatility, a critical issue is the control of the microstructure that plays a key role on the consistency of the properties. However only a limited number of studies have yet focused on the thorough description of the microstructure by additive manufacturing techniques. The aim of this work was thus to analyze the microstructure of bulk samples prepared by additive manufacturing of Ti-6Al-4V (Ti64) powder by mean of the CLAD® technique (laser blown powder processing) developed at IREPA LASER. The very high cooling rate resulting from this process induced the formation of the martensitic α' phase in the form of thin needles. Using Merengue 2 software developed at LEM3, the analysis of the martensitic phase orientation by EBSD enables the reconstruction of the primary β -phase that formed at high temperature. Microstructural observations performed at various locations within the samples revealed marked heterogeneities in the microstructure. After the first deposited layer characterized by fine and equiaxed β grains, the β grains located in the central region of the samples tend to grow in a direction perpendicular to the deposited layer plan with a coarse columnar structure. By opposition, the grains on the sample side were found to grow at angle of about 60° from the deposited layer plan. The main difference with others additive manufacturing techniques is that, only a weak crystallographic texture was detected in these samples prepared by CLAD®.

Keywords: LMD; additive manufacturing; TA6V; texture

1. Introduction

Since a decade, the understanding on laser rapid manufacturing technologies has rapidly increased and the developers found applications in various industrial sectors for parts manufacturing or repairing. IREPA LASER has developed its own specific tools for the manufacturing of meso-scale and macro-scale metallic and functional 3D parts from CAD files (*Vetter, 1997; Sankaré, 2007*). This process, named CLAD® (Construction Laser Additive Direct), is based on laser cladding, and involves the deposition of melted powders into a melt pool for 3D component manufacturing. The process parameters in additive manufacturing are numerous and the general properties (geometry, mechanical) of the manufactured parts are directly related to the operating conditions and to the materials used. For example, the properties of common AISI 316L samples made by LMD (Laser Metal Deposition) have been studied and are well understood (*Dutta Majumdar et al, 2005; Vilar, 2001*), but the influence of process parameters on the characteristics of the manufactured samples, i.e. microstructure, is not clearly established.

To the authors knowledge, the microstructure description and evolution of titanium alloy Ti64 have been investigated for different additive manufacturing techniques, which are mostly powder bed processes techniques: Electron Beam Melting (EBM) (Al-Bermani *et al.*, 2010 ; Antonysamy *et al.*, 2013 ; Murr *et al.*, 2009), Selective Laser Melting (Thijs *et al.*, 2010 ; Antonysamy, 2012 ; Simonelli *et al.*, 2012), or wire feed technique (Baufeld *et al.*, 2011 ; Brandl *et al.*, 2012). No literature has been found for powder blown processing for this alloy. This paper aims at investigating the microstructure and texture obtained by powder blown additive manufacturing process and at underlining the main differences with other additive manufacturing technologies.

2. Material and experimental setup

The laser deposition tests were carried out on a 5 axis LMD machine designed by IREPA LASER (fig. 1 (a)). The substrate was a Ti6Al4V plate with 5mm thickness. The process developed by IREPA LASER is a blown powder additive manufacturing process, called CLAD® process, with a laser as a heating source. In this process, a material in a powder form is injected in a coaxial nozzle (US Patent n° 5418350). The powder is carried by a transport gas. At the output of the nozzle, the powder jet is shaped by a secondary gas (fig. 1(b)) in order to concentrate the powder density. The powder is then heated and melted by a laser source before to be deposited on a substrate. The addition of deposited layers creates the manufactured object.

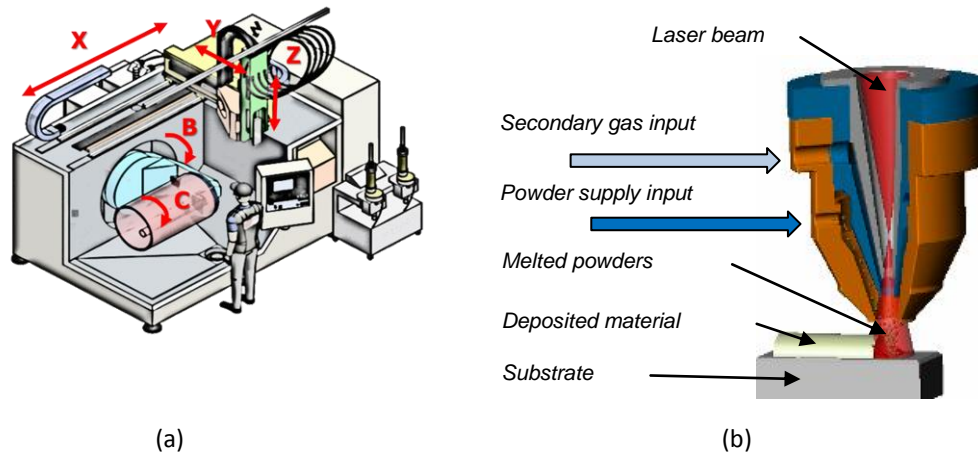


Figure 1: (a) the 5 axis CNC workstation (b) the coaxial laser cladding nozzle patented by IREPA LASER

This machine is equipped with a 500 W diode laser (200 μm diameter and 1070 nm wavelength) from IPG LASER. This machine is equipped with 5 axis, with travels of $X1500 \times Y800 \times Z800$ mm for the linear axes, and a swiveling (B) and rotating (C) movement for the part (Fig. 1a). In this configuration, the nozzle works always vertically, because gravity affects the powder jet stream. Two nozzles are integrated into this machine, and work alternatively for the manufacturing of the parts. The first nozzle (called macroCLAD 10Vx) makes deposition tracks with a width in the range of 1-1.2 mm, and the second nozzle (called macroCLAD 24Vx), equipped with a different optical system, makes tracks width in the range of 2-2.4 mm. The machine is equipped with a gas enclosure, with a volume of 12 m^3 specially adapted for the manufacturing of Ti alloys parts.

The titanium powder used for this study was plasma atomized Ti-6Al-4V grade 23 from AP&C with a 45-90 μm diameter. A cube with 20 mm edge length has been built. The distance between the nozzle and the deposited track was 35 mm. The sample has been made under controlled atmosphere with less than 20 ppm O_2 and less than 100 ppm H_2O . The macroCLAD 10Vx nozzle was used for part manufacturing. The laser power was 425 W, the scan speed was 2000 mm/min and the powder flow was 2.1 g/min. The resulting track dimensions were 0.94 mm for the track width and 0.27 mm for the track height.

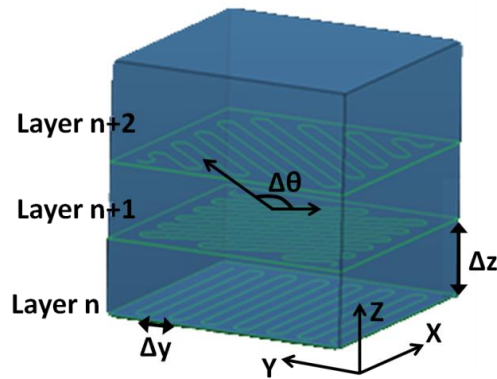


Figure 2: Building strategy

The construction strategy consisted of a filling of the inside with parallel tracks with an overlap of 30% (i.e $\Delta y=0.7\text{mm}$). Finally, around each layer a contour track is deposited (fig. 2). The height between each layer, Δz , was the height of one track: 0.27mm . The track deposition direction between two consecutive layers was rotated by $\Delta\theta=113^\circ$ in order to reduce the content of residual stresses.

The macrographic analysis was performed with an optical microscope Olympus BX61 and reconstruction multimapping with Stream Motion software. The microscopic images were taken on two different JEOL scanning electron microscopes: a JSM-6490 (with a W-gun) and a JSM-6500F (with a field emission gun). Grain orientations were determined by electron back-scattered diffraction (EBSD), using AZtech (Oxford Instruments, HKL Technology) for data acquisition. The reconstructions of the primary β grains were performed with the Merengue 2 software developed by the LEM3 (Fig. 3). The reconstruction procedure is similar to that described by *Germain et al 2012a* and is performed in three steps. In a first step, crystallographic domains (grains) are identified using an Anti-Leak Grain detection (*Germain et al 2014b*). In a second step, neighboring domains related by the Burgers orientation relationship (with a 3° tolerance) to a common parent orientation are sought. Those which satisfy some quality criterions (set by trial and error) are called fragment and their parent orientation is then known. Starting from fragments, Merengue 2 gathers all domains related by the Burgers relationship (within a tolerance of 4°) to the same parent. This last step called “enlargement” allows the determination of the parents orientation of the remaining domains.

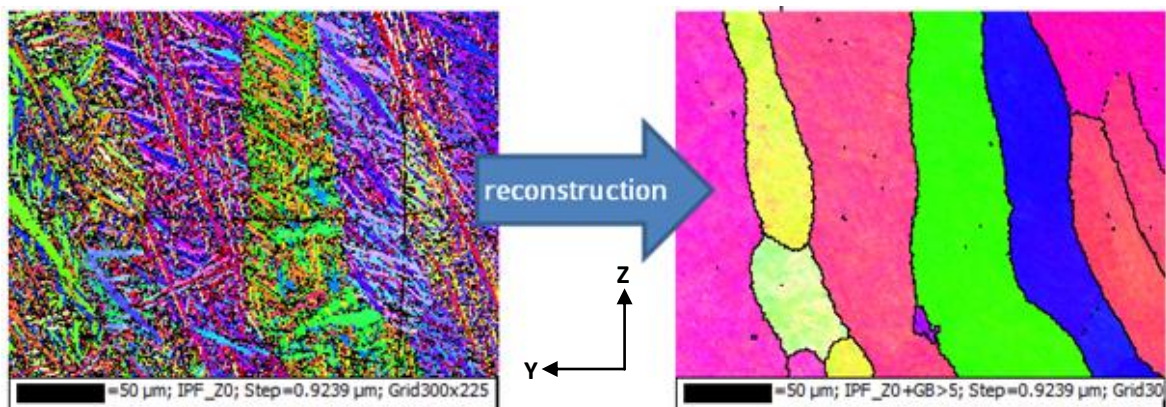


Figure 3: Example of a picture from the center of the YZ sample face before and after reconstruction

3. Experimental results

The sample in the as produced condition is shown Fig.4 a. It has then been cut for metallographic observation (Fig. 4 b) both in longitudinal and transversal directions.

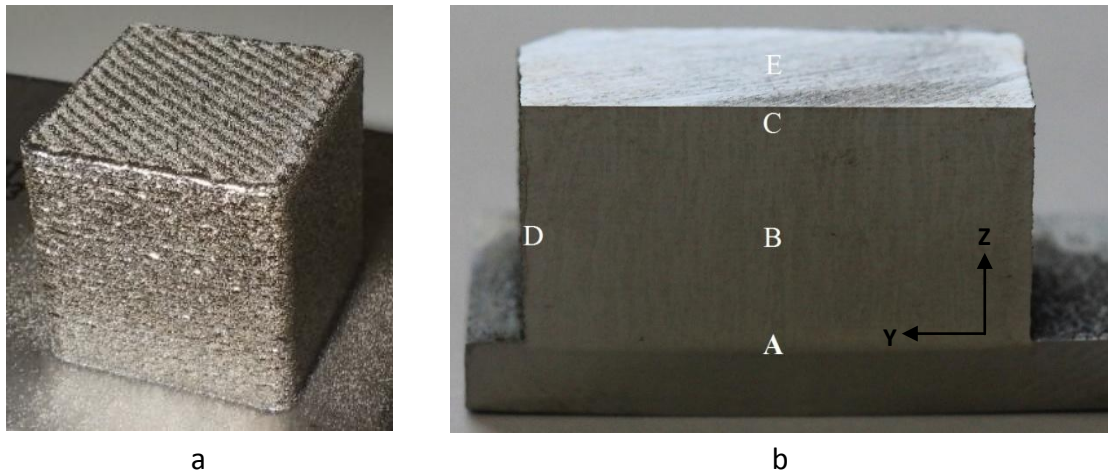


Figure 4: a. Sample as produced. b. Center of the sample after cutting with the letters corresponding to the analyzed zone (A=Bottom ; B=Center ; C=Top ; D=Side ; E=XY face)

On the macrograph (Fig.5), a very coarse structure can be observed. It corresponds to the columnar β grains which are up to several millimeters long. At a higher magnification, the martensite needles can be observed in the primary β grains. The boundary between the substrate and the deposited layers is clearly distinguishable. This is due to the brutal change in microstructure: the micro-grains of the rolled plate suddenly grow at the border which delimits the molten zone at the bottom of the image. It is also possible to see horizontal bands corresponding to deposited layers with a $270 \mu\text{m}$ thickness.

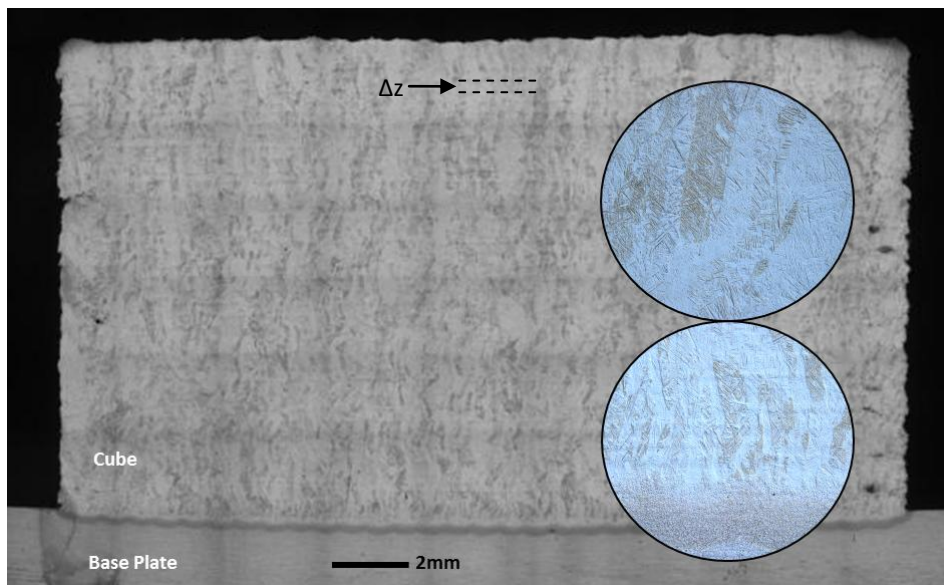


Figure 5: Multimapping picture of XZ face in the center of the sample. The columnar grains can be seen in the enlargements

The reconstructed EBSD maps of primary β grains at high temperature show significant heterogeneity (fig. 6 and 7). Indeed, as previously seen in fig. 5, the region close to the support presents small size grains coming from the plate and medium-size columnar grains coming from the deposited layers (Fig 7A). These columnar grains have grown up to several millimeters in the central area of the sample before decreasing at the top of the sample, in the last deposited layer (Fig 7C). At the edges, a grain growth along a direction forming an angle of 60° with the z axis is observed (Fig 7D). This angle rapidly deviates to align with the Z-direction when the grains are moving away from the edge to the center of the sample. The columnar grains were observed in cross section by analyzing the XY plane (Fig 7E).

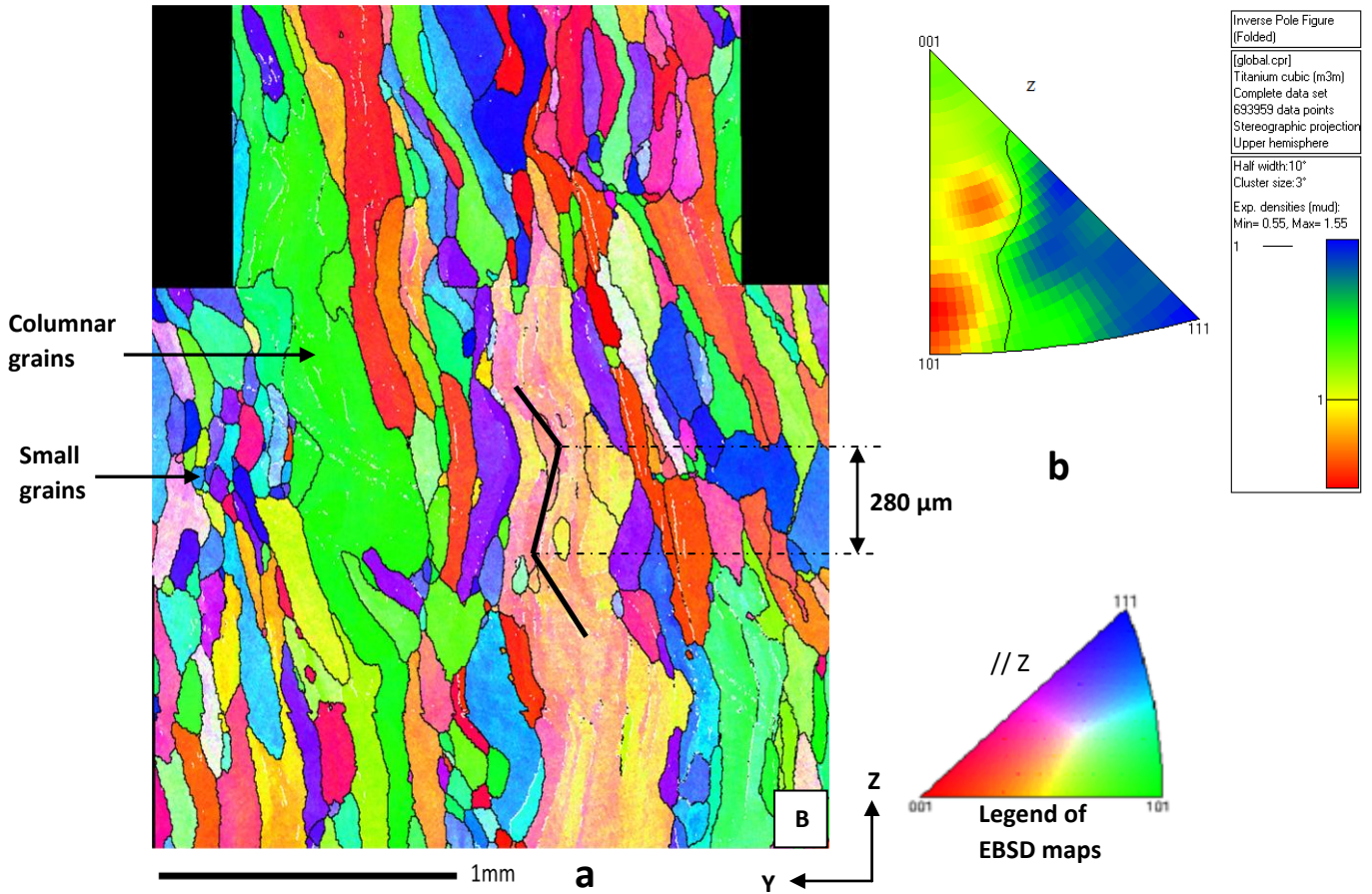


Fig 6 : a. EBSD Map corresponding to the area B as shown in the fig.4b (grain boundaries: black > 10° ; white > 6°) and b. Inverse Pole Figure (IPF) adds up all maps of this paper (150 grains).

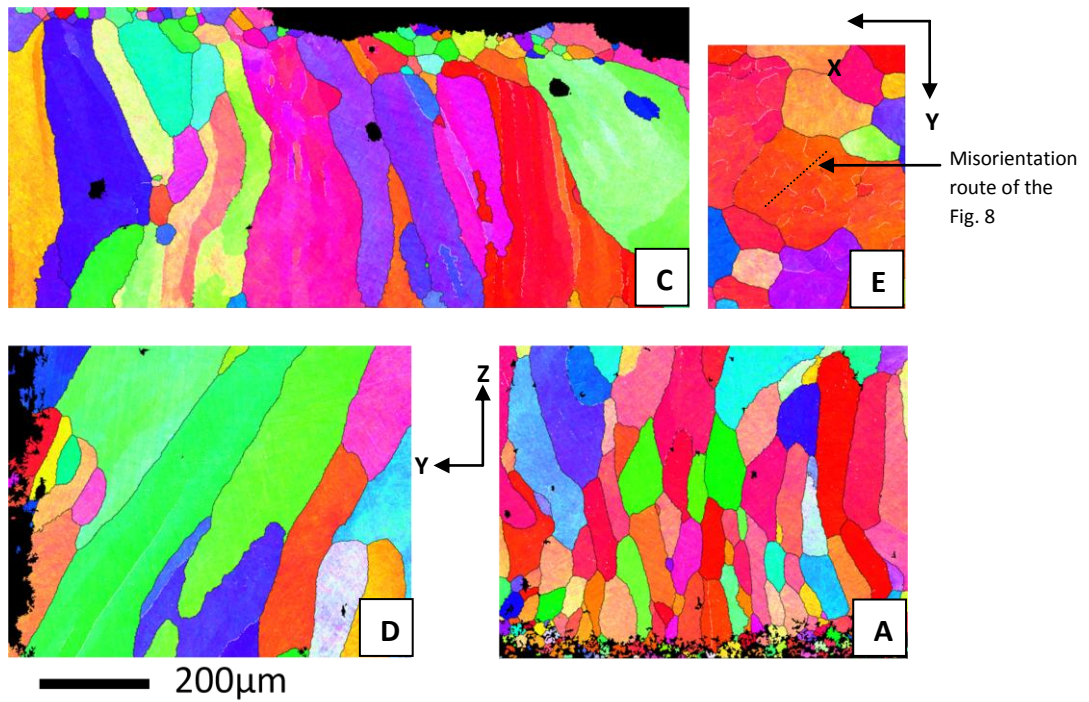


Fig 7 : EBSD Map corresponding to the areas A, C, D and E as shown in the fig.4. The E map is in the XY plan. The legend is previously given in the Fig.6

4. Discussion

The final structure of the sample is composed of α' martensite needles like in parts manufactured by SLM (Selective Laser Melting), contrary to the EBM which gives a fine α structure (Murr *et al*, 2012). This is due, in the case of the CLAD method, to rapid cooling caused by the argon gas which continuously circulates around the sample. This martensitic structure is homogeneous throughout the piece, in the center as at the edges.

The columnar grains in Ti64 additive deposition has been observed by many authors as mentioned earlier. They are due to the re-melting of the previously deposited layers as well as the thermal gradients (Al-Bermani *et al*, 2010). As the last layer does not undergo this series of thermal processing, it produces a small grains structure corresponding to the powder melted then rapidly solidified.

In the central area, the columnar grains present a herringbone pattern. It is interesting noting that the distance between each turn is equal to the thickness of a deposited layer (Fig. 6). This shows that the grain shape is strongly dependent on the deposition strategy. This relation is the subject of an on-going work. CLAD method gives a high heterogeneity in terms of β microstructure and texture for the primary beta grains. Indeed, usually in additive deposition, the grains have their $\langle 100 \rangle$ direction perpendicular to the deposition direction. This particular orientation due to thermal gradients, is found in all additive manufacturing process and has been observed by all the authors mentioned above. In the case of the CLAD method and with the construction strategy of rotating tracks by 113° between each layer, no texture was measured which means that this process produces a weak texture (IPF Fig.6). Besides, beta grains exhibit substructures which misorientation profile is shown in Figure 8.

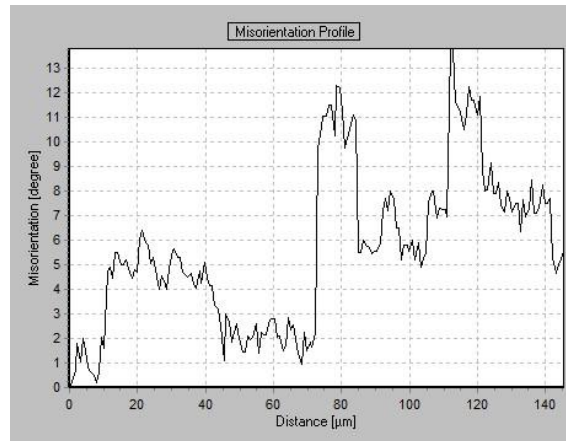


Fig 8 : Misorientation profile into a columnar grain (the route is visible in the Fig. 7E)

However, from the microstructure point of view, small grains are trapped in the matrix formed by the columnar grains which is a new result compared to the studies undertaken so far with other techniques such as bed powder or the electron beam. Due to their size and their shape (Fig 6), these small grains could be either unmelted or partially melted powder particles. Further testing are underway to confirm these hypotheses.

The structure inclined at about 60° located around the edges is due to the direction of thermal gradients as explained by Thijs *et al*. (2010) and is the result of the contour deposition as shown by Antonysamy *et al* (2013) for Ti-6Al-4V produced by EBM.

5. Conclusion

CLAD process was used to fabricate Ti-6Al-4V pieces. Rapid cooling of the molten zone results in a $\beta \rightarrow \alpha'$ martensite transformation throughout the sample.

Reconstruction of the primary beta grains shows a heterogeneous structure comprising central columnar grains parallel to the growth axis with an herringbone pattern. These primary beta grains measures up to

several millimeters in the building direction. Some small grains, which could be unmelted or partially melted powder particles, are found on the sample edges and are trapped between the previously described grains.

Unlike other additive manufacturing techniques such as EBM or SLM in which the axis $\langle 100 \rangle_{\beta}$ is parallel to the sample z axis (build direction), the CLAD® process doesn't reveal any significant texture. To explain this phenomena, more information is needed and it is considered to correlate the deposition strategy with primary formed β -grain shape and the mechanical properties of the pieces in future studies.

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Microtexture de Ti-6Al-4V développée lors d'une fabrication par dépôt de poudre fondue par laser

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Résumé

La fabrication additive de pièces métalliques fait l'objet d'une considération accrue dans un large éventail de secteurs tels que l'aérospatial, la défense et le biomédical. Alors que l'un des principaux avantages de cette technique est sa polyvalence, une question cruciale reste en suspens : peut-on contrôler la microstructure via les paramètres de fabrication, sachant que celle-ci joue un rôle clé sur les propriétés physiques des matériaux ainsi mis en forme ? En effet, il existe un nombre limité d'études portant sur la description détaillée de la microstructure et les textures obtenues sur des échantillons mis en forme par des techniques de fabrication additives. Le but de ce travail est donc d'analyser la microstructure d'un échantillon préparé par dépôts successifs de poudre Ti-6Al-4V fondue par laser au moyen de la technique CLAD® (Construction Laser Additive Directe) développé à l'IREPA LASER. Les vitesses de refroidissement très élevées obtenues pendant la construction induisent la formation de la phase martensitique sous la forme de fines aiguilles. En utilisant un procédé mis au point au LEM3, l'analyse de l'orientation de la phase martensitique par EBSD permet la reconstruction de la phase bêta primaire qui s'est formée à haute température. Les observations microstructurales effectuées à divers endroits dans l'échantillon ont révélé des hétérogénéités marquées dans la microstructure. Après la première couche déposée, caractérisée par des grains fins équiaxes, les grains bêta primaires situés dans la région centrale des échantillons ont tendance à se développer dans une direction perpendiculaire au plan de la couche déposée avec une imposante structure colonnaire. Sur les bords de l'échantillon, les grains croissent avec un angle d'environ 60 ° par rapport au plan de la couche déposée, angle dû au phénomène de transfert thermique. La principale différence avec les autres techniques de fabrication additive est qu'une faible texture cristallographique apparaît dans les échantillons préparés par CLAD®.