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Crystallographic analysis of functionally graded titanium-molybdenum alloys with DED-CLAD® process

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Abstract

Construction Laser Additive Direct (CLAD®) is a blown powder additive manufacturing process in which complex and functional parts can be created layer-by-layer. This process is characterized by a direct injection of metallic powder under the laser beam and now allows the manufacturing of custom-made part such as functionally graded materials (FGM). Microstructures and crystallographic orientation will depend on the chemical composition of the deposited layer as well as the process parameters and the part dimensions.
This article describes microstructures and crystallographic orientations of functionally graded titanium-molybdenum alloys with different chemical composition variation and different shape configurations. Microscopy observation and EBSD analysis provides information about behavior of such materials used in biomedical applications.

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Keywords: Functionally Graded Materials; Titanium-Molybdenium alloys; Additive Manufacturing; Direct Energy Deposition process; Differential injection system

1. Introduction

Nomenclature	
AM	Additive Manufacturing
FGM	Functionally Graded Materials
DED	Direct Energy Deposition
CLAD®	Construction Laser Additive Direct
EBSD	Electron Backscatter Diffraction

For some years now, AM processes have been rapidly developed. Contrary to machining processes which are based on removing material, AM processes consist to create a part layer-by-layer by adding material. These processes are used in several fields such as automotive, aerospace and biomedical sector for large parts manufacturing or repairing [1].

Irepa Laser had developed its own process called CLAD®. This blown powder process, where process parameters are numerous, allows the manufacturing of very complex parts thanks to 5-axis machine (3 translations, 2 rotations). CLAD® is today adapted to manufacture functionally graded materials. These new materials have been developed in 1984 to obtain high-performance heat-resistance materials [2]. FGM consist of a modification of the chemical composition during the part manufacturing. This leads to mechanical, thermal and microstructural variation along one or more space direction. Blown powder processes are suitable to perform FGM because the powder is directly injected under laser beam.

Today, the possibility to manufacture custom-made parts with specific properties is became a real asset. *In-situ* manufacturing also allows to respond to the unavailability of specific alloy in the market.

This article present study on functionally graded Ti6Al4V-Mo alloys. In biomedical area such as orthopedic application,

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the most widely used alloy is Ti6Al4V thanks to its outstanding combination of strength, corrosion resistance and biocompatibility. However, the presence of Al and V may cause long-term health problem such as Alzheimer's diseases or peripheral neuropathy [3,4]. Combined with a non-allergenic and non-toxic beta-stabilizer such as molybdenum, Ti6Al4V with a good alloying element concentration provides lower Young's modulus than α-phase. Ti6Al4V-Mo alloys have been already studied with a maximum Mo content of 25wt.%. However, behavior of this couple of material when molybdenum varied from 25% to 100% is not fully established.

2. Materials and methods

DED-CLAD® process was used to manufactured FGM samples with two different materials: Ti6Al4V (hereafter 'Ti6Al4V' will be referred to as Ti64) and molybdenum. The variation of the chemical composition along the walls manufacturing is control by the use of two powder feeder. By adjusting the rotating speed of each powder feeder, the powder ratio can be set for each material to meet the required chemical composition. Both materials are mixed before being injected in the laser beam and deposited onto the Ti64 substrate.

The CLAD® process developed by Irepa Laser is equipped with a 2 kW laser diode (fiber Ø600 µm, λ=980 nm). The coaxial nozzle in which the metallic powder is injected (US Patent n°5418350) was adapted to work with refractory material such as molybdenum and to be resistant to high temperature (>2000°C). The manufacturing processes were carried out under argon gas due to affinity of titanium alloys with oxygen. For each deposition of different chemical composition, process parameters were adjusted.

The initial particles size distribution of Ti64 and Mo powders ranging from 45 to 90 µm. However, due to the high difference in melting point temperature between both powder (T_f(Ti64)=1670°C; T_f(Mo)=2617°C), Mo powder was sieved to keep particles size distribution from 45 to 75 µm only. The particles size distribution of both powders is reported in table 1.

Table 1. Particles size distribution of Ti64 and Mo powders.

Size distribution (µm)	D10	D50	D90
Ti64	57,9	79,6	109,2
Mo	44,9	60,6	81,5

Three types of sample are presented in this study:

- a single-track width wall manufactured with an increase or a decrease of 25% between each gradient (ie 5 FGMs)
- a single-track width wall manufactured with an increase or a decrease of 20% between each gradient (ie 6 FGMs)
- a two-track width wall manufactured with 4 gradients of chemical composition

To perform microstructural and crystallographic analyzes in the transversal direction, samples were cut, mechanically polished and etching with a Kroll solution. The microstructures were observed by optical microscopy and scanning electron microscopy (Zeiss Supra 40). Crystallographic analyses were carried out by EBSD with a Jeol 6490 equipped with a tungsten filament. EBSD data were then processed with AZtech (Oxford Instrument, HKL Technology).

3. Results and discussion

The modification of the chemical composition along the wall manufacturing leads to a microstructural evolution (fig.1), as explained previously by Schneider-Maunoury et al. [5]. Martensite needles are visible at higher magnification (fig.1-A). The initial α'-phase present in the first 100% Ti64 deposition disappears to take place to a β-phase dominant from 25% Mo. Authors have demonstrate that molybdenum operates as a beta-gene element which avoid the martensitic transformation [6,7,8].

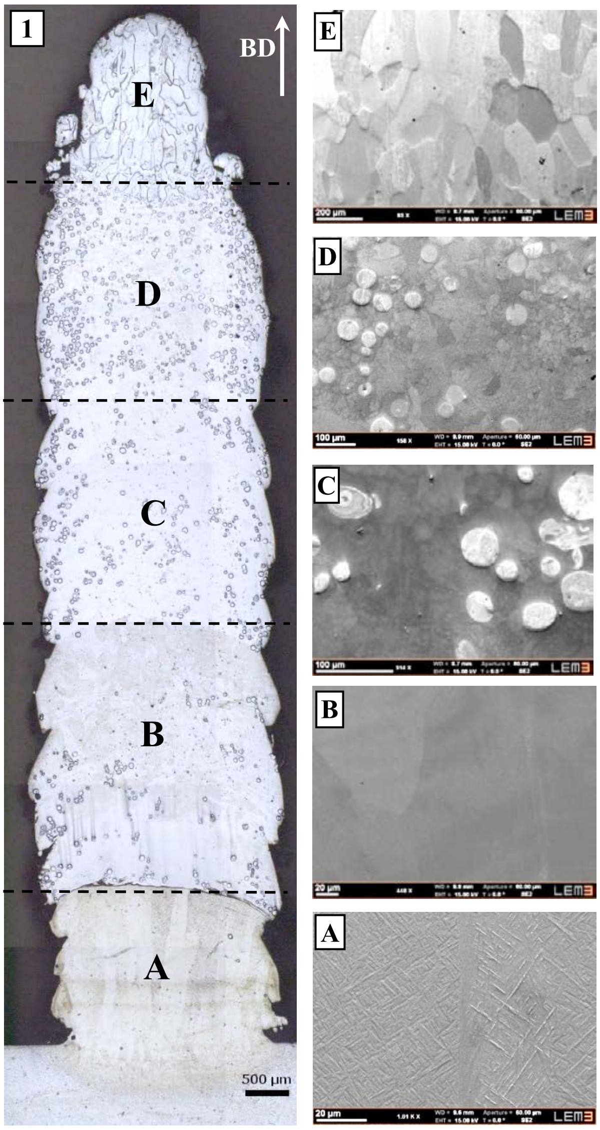

Fig. 1. (1) Optical microstructure; (A-E) Microstructures of Ti64-Mo alloys with Mo content from 0% to 100% Mo and a variation of 25% Mo.

This evolution of chemical composition also leads to a crystallographic modification from the substrate to the top of the wall (grain size, grain shape, crystallographic orientation), as shown in fig.2. Authors have demonstrated the influence of process parameters such as scanning velocity and hatch spacing as well as scanning strategy on the morphological grains [9].

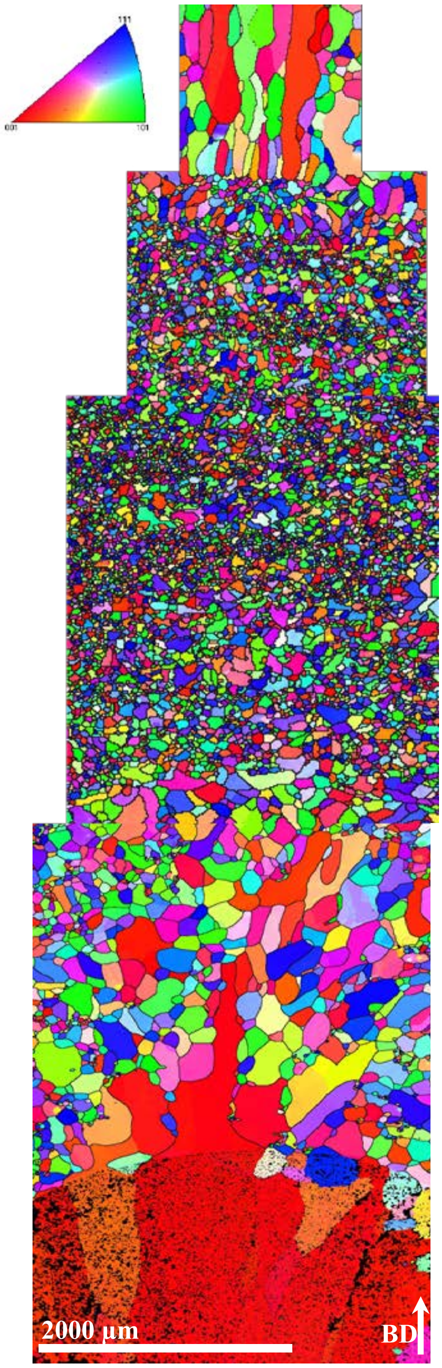

Fig. 2. EBSD analysis of Ti64-Mo alloys with a variation of 20% Mo content.

The β-phase grain structure of 100% Ti64 deposition was reconstructed from the α-phase orientation map thanks to Merengue 2 software which is based on Burgers relationship. This first graded deposition composed by 100% Ti64 present large columnar β-grain grows parallel to the build direction with a strong fiber texture $<100>_{\beta}$, as confirmed by Antonysamy [10]. This specific orientation of Ti64 alloys is found in all the additive manufacturing process. By adding molybdenum up to 20% content, a significant change of morphology is observed marked by the appearance of coarse equiaxed grains. Gäumann and Hunt explained the CET (Columnar to Equiaxed Transition) by the equiaxed grains nucleation and growth ahead of the moving solidification interface when there is a region of undercooled liquid [11,12]. Then, the more content of molybdenum into the deposition, the smaller the equiaxed β-grains. Any crystallographic orientation is observed in the x% Ti64 - y% Mo deposition (20% < x; y < 80%).

Fig.2 also reveal the presence of lines of very small equiaxed grain (around 15 µm diameter) at regular interval corresponding to one-layer thickness. As explained previously by Schneider-Maunoury et al., these lines may result from the remelting of the previous layer, which lead to a change in grains morphology at the interface between two deposited layers [5].

Crystallographic analysis of sample manufactured with two tracks width is shown in fig.3. This sample was manufactured by two tracks width deposition with a round trip strategy for each layer and 30% overlapping, as shown in fig.4.

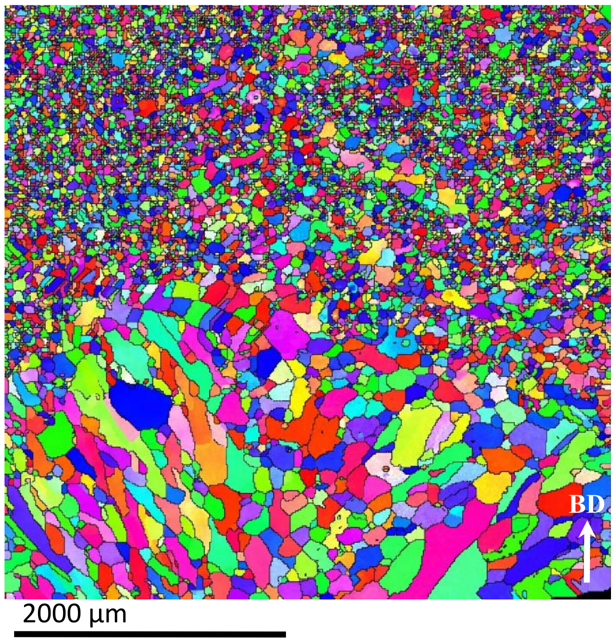

Fig. 3. EBSD analysis on the width of the wall at the interface between 65% Ti64 - 35% Mo (at the bottom) and 40% Ti64 - 60% Mo (at the top).

EBSD map shows a deposition composed by beta grains only. Depending on the chemical composition of the deposition, the beta grains present different morphologies. Indeed, on the bottom of the map, coarse elongated grains are found near the face of the wall whereas the overlap zone is composed by smaller equiaxed grains. The elongated grains have an inclination of 50° with an orientation toward the edges of the wall. The top of the map shows a modification of grain shape due to the alloy composition. Grains are equiaxed on the entire width of the wall although they are bigger in the overlapping zone (around 140 µm diameter compared to 50-80 µm diameter for grains outside the overlapping zone).

The presence of equiaxed grains in the overlapping zone is due to the partial remelted of the first deposited track which leads to recrystallisation phenomenon. Moreover, the orientation of elongated grains in 50° toward the edges of the wall is explain by the thermal gradient involved by the laser heat source as well as the convection effect between the molten pool and the air which contribute to cooling track.

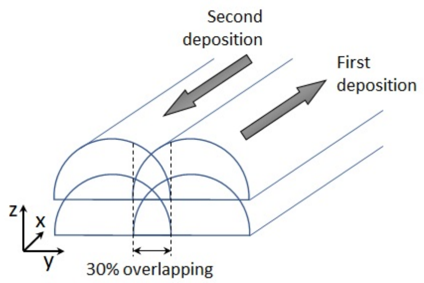

Fig. 4. Scheme of scanning strategy for two tracks width deposition.

4. Conclusion

The manufacturing of FGM with DED-CLAD® process has been demonstrated in this study. Ti64-Mo alloys have been successfully manufactured with different variation in chemical composition (20% or 25% steps between each graded deposition) and different shape configurations (single or two tracks width deposition). The variation from 0% to 100% of molybdenum content is easily controlling with blown powder processes thanks to multiple powder feeder. Results show that microstructure, grains morphology and crystallographic orientation depends on the chemical composition, as well as the dimensions of the part.

Acknowledgements

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