ESRF	**Experiment title:** Microstructure optimization of titanium alloys produced by additive manufacturing	**Experiment number**: MA-3006
Beamline: ID16A	**Date of experiment**: from: 20-04-2016 to: 25-04-2016	**Date of report**: 10.09.2017
Shifts: 15	**Local contact(s)**: Julio Cesar Da silva and Peter Cloetens	*Received at ESRF:*

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Report:

Objectives and expected results

To determine the evolution of the 3D architecture of phases as well as the influence of diffusion-driven element partitioning on the phase transformation kinetics of Ti-alloys undergoing decomposition of martensite during thermal treatment. To provide knowledge-based thermal treatment optimisation for the processing advance of titanium alloys produced via selective laser melting (SLM) –a powder bed-based 3D printing technique capable to manufacture near net-shape titanium components with complex geometries (not achievable by conventional forming methods), with manufacturing cost reductions estimated up to 50%. The investigations at ID16A aim at linking the morphological evolution of phases with studies of phase transformation kinetics using in situ high energy synchrotron X-ray diffraction –previously carried out by the proposers– which revealed the sequence of martensitic decomposition in titanium alloys as function of temperature and heating rate. By doing so, the proposers intend to validate/correct/modify a correct control of thermal profiles during solidification and additional heat treatments (or both) necessary to obtain microstructures with stable architectures of α and β phases that result in an improvement of fatigue resistance in the popular α+β Ti-6Al-4V (Ti-64) alloy produced by SLM, which accounts for more than 50% of the titanium market and leads AM of titanium alloys. The results obtained have been published in:

*P. Barriobero-Vila, J. Gussone, J. Haubrich. S. Sandlöbes, J. Silva, P. Coetens, N. Schell, G. Requena, Inducing stable α+β microstructures during selective laser melting of Ti-*6Al-4V *using intensified intrinsic heat treatments, Materials 10(3) 268 (2017).*

Results and conclusions

Micro-cylinders of about 16 µm diameter × 18 µm height were prepared for holographic X-ray computed tomography (HXCT) from post treated SLM samples as well as from representative microstructural regions highly affected by the intrinsic SLM heat treatment (IHT). The IHT aims at producing Ti-64 by SLM using a combination of porosity-optimized processing parameters based in our previous studies, with a tight hatch distance of the scanning laser during SLM, leading to longer exposure time laser-material, and decomposition of brittle martensite providing stable α and β phases (desired for structural applications).

Phase contrast was required since the microstructural components of the Ti-64 alloy do not produce sufficient absorption contrast. The samples were illuminated with a magnifying X-ray cone beam of 33.6 keV focused by Kirkpatrick–Baez mirrors using the zoom HXCT approach. 2500 projections with an image size of 2048 × 2048 pixels and a field of view of 20.5 × 20.5 μm^2 were recorded between 0 and 180° employing a CCD camera at four sample-to-focal-point distances (5.54, 5.72, 6.45 and 7.95 mm) for efficient phase retrieval of the holotomographic reconstruction. The experiments were performed in an ultra-high vacuum atmosphere and the acquisition time was ~1 s/projection resulting from the sum of exposure and read-out times. 3D segmentation of phases was performed by local threshold based on grey-level distribution after image conversion from 32 to 8 bits. The reconstructed volume has a voxel size of 10 × 10 × 10 nm^3.

HXCT (Fig.1) reveals that —for a region highly affected by the IHT— the β matrix consists of a thin (≤70–100 nm) and highly interconnected continuous 3D network throughout the investigated volume (blue). This indicates that the IHT applied during SLM induces the formation of a β matrix percolating through the α lamellae. The impact in the mechanical performance of the alloy is that homogeneous distributions of slip transfer between phases can occur across the incoherent α/β interphase.

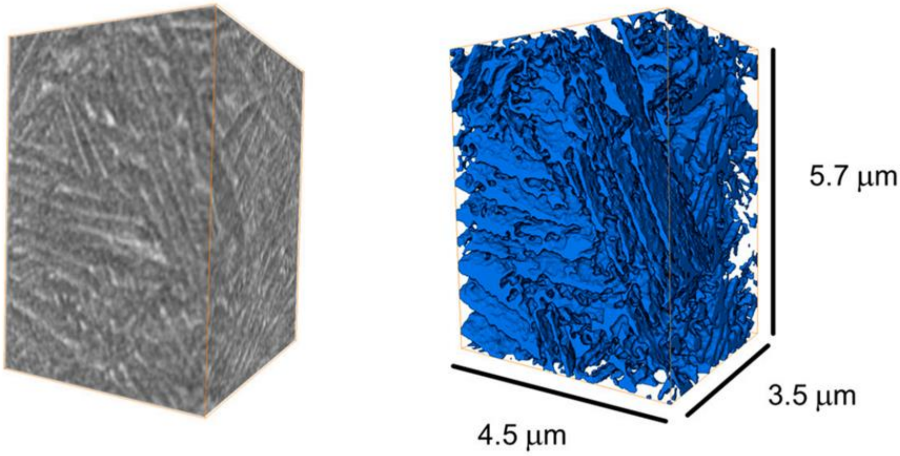

Fig.1. Illustrative perspective of an interconnected network of β matrix (in blue) segmented from its associated holographic X-ray computed tomography (HXCT) reconstruction (voxel size = $(10\ nm)^3$. It corresponds to a representative volume of the microstructure of SLM-produced Ti-6Al-4V highly affected by the intrinsic heat treatment.