ESRF	**Experiment title:** Development of versatile microstructures for steels and metastable Beta Ti-alloys	**Experiment number**: MA-2909
Beamline: ID22	**Date of experiment**: from: 21-07-2016 to: 25-07-2016	**Date of report**: 24.09.2017
Shifts: 12	**Local contact(s)**: Carlotta Giacobbe and Andy Fitch	*Received at ESRF:*

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Report:

Objectives and expected results

To provide the continuous and univocal evolution of the phase transformation kinetics during thermal treatment of metastable β titanium alloys and steels produced by additive manufacturing. To perform quantitative phase analysis of the diffraction patterns to determine the variations in phase volume fractions and lattice parameters and evaluate: a) the effect of the heating rate / holding temperature on the phase transformation sequence, b) the evolution of the crystal structure in terms of the phase transformation mechanisms (e.g. diffusion-driven processes), and c) the crystallographic relationships between the lattice structures of stable and metastable phases. The results obtained were complemented with atom probe tomography to link the obtained structural information of phases with the evolution of their 3D architecture and element partitioning. Fundamental rules based in these investigations will be established for the design of new, optimized processing paths for metastable β titanium alloys and steels produced via additive manufacturing. The results obtained have been published in:

P. Kürnsteiner, M. Wilms, A. Weisheit, P. Barriobero-Vila, E. Jägle, D. Raabe, Massive nanoprecipitation in an Fe-19Ni-xAl maraging steel triggered by the intrinsic heat treatment during laser metal deposition, Acta Mater. 129 (2017) 52-60.

Results and conclusions

High energy X-ray diffraction was carried out at ID22 using an energy of 70 keV. Metastable titanium alloys as well as steel samples affected by the intrinsic heat treatment during laser metal deposition were investigated ex situ as well as in situ by capsuling the specimens in argon atmosphere using capillaries. An air blower was

employed to reproduce thermal profiles at isothermal temperatures. The thermocouple was located next to the position of the incoming X-ray beam (slit size of 0.5 × 0.5 mm). During spinning the specimens at 300 rpm, image sequences of complete Debye-Scherrer rings from the bulk of the samples were recorded in transmission mode using an image-plate detector Perkin Elmer XRD 1611. Instrument parameters of the diffraction setup were obtained using a LaB6 powder standard. Azimuthal integration of the intensity along the Debye-Scherrer rings was performed using the software Fit2d. Quantitative phase analysis of the diffraction patterns was carried out by the Rietveld method as implemented in the software Maud.

Samples of 5 mm diameter and 10 mm length of commercial β-quenched metastable β titanium alloys Ti-5Al-5V-5Mo-3Cr (Ti-5553), Ti-5Al-2Sn-2Zr-4Mo-4Cr (Ti-17), operating nowadays in modern aerospace applications (e.g. landing gears and gas turbine engine components), were investigated together with model alloys Ti-45Nb and Ti-18Mo to consider the effect of alloying in simpler binary systems. Moreover, a model maraging steel alloy with Fe, Ni and Al, showing a pronounced response to the intrinsic heat treatment (IHT) imposed during a Laser Additive Manufacturing (LAM) was investigated. IHT occurs owing to the layer-by-layer build-up of additively manufactured parts where the deposited material experiences a cyclic re-heating in the form of a sequence of temperature pulses. Using this methodology it was possible to produce a maraging steel that is intrinsically precipitation-strengthened by an extremely high number density of 1.2×10^{25} m^3 NiAl nanoparticles. Atom Probe Tomography was capable of quantifying the high number density of nanoparticles present in specific layers of the LAM sample, while high energy X-ray diffraction carried out at ID22 permitted to identify the precipitate phase as NiAl (Fig. 1).

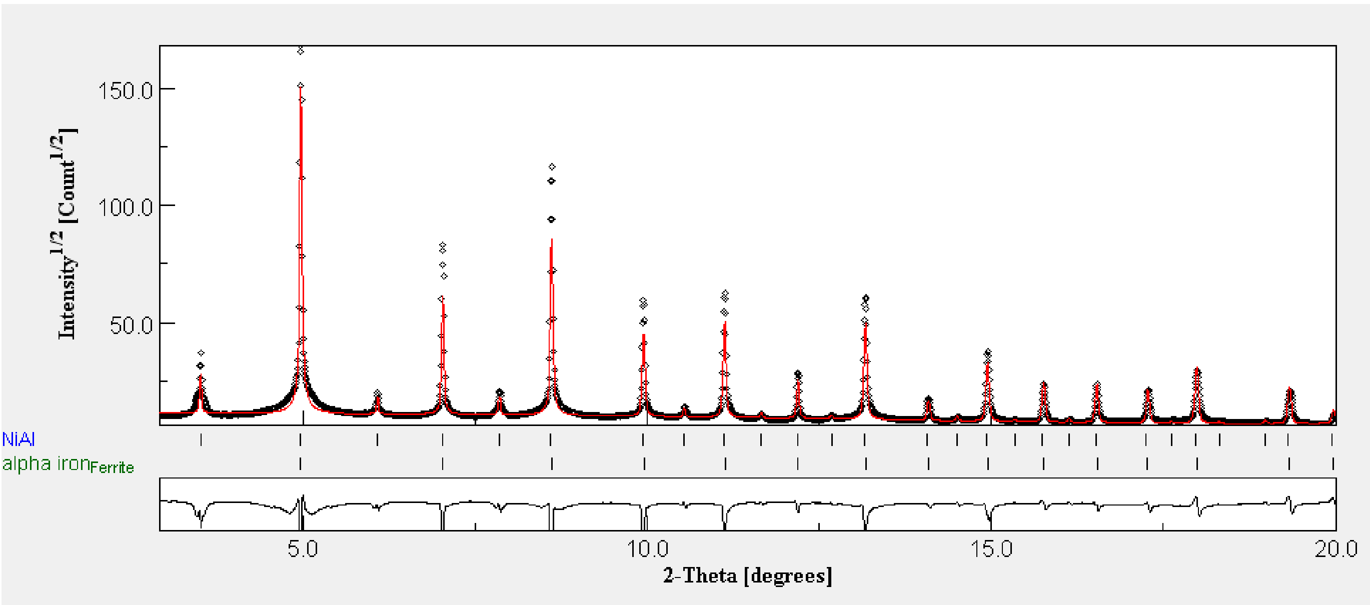

Fig.1. XRD spectrum of a representative 2θ range from a sample layer containing ferrite and NiAl.