Experimental Report BM02_ESRF septembre 2016

Objective: During ESRF beamtime, *in situ* uniaxial tensile tests (continuous loading and cycle loading) were performed to evaluate the mechanical properties of harmonic design Ti and Ti alloys sintered by Spark Plasma Sintering (SPS). The aim is to use the intrinsic crystallographic phase and, mechanical phase selectivity of X-Ray Diffraction (XRD) to measure during *in situ* loadings the evolution of diffraction peaks (position, FWHM and intensity) in order to determine the main mechanisms of deformation and to establish a link between the microstructure and the mechanical properties. Those experiments have been performed on samples with a specific microstructure, namely harmonic design microstructure, which is designed to solve the dilemma ductility-strength in metallic materials. The harmonic microstructure design has a heterogeneous microstructure consisting of a controlled and specific topological distribution of fine and coarse grains. This microstructure is heterogeneous on micro-scale but homogeneous on macro-scale[1]. The Figure 2[1] illustrates this microstructure:

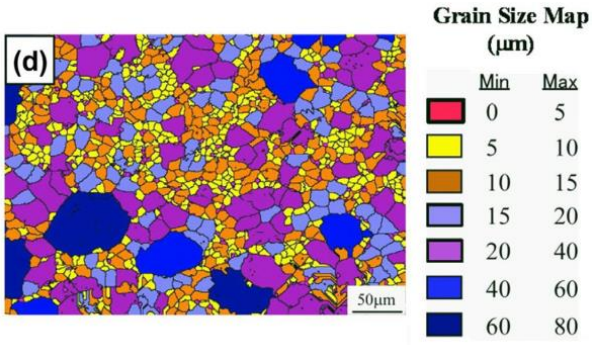

Figure 1: EBSD grain size map of a Ti harmonic metal. Coarse grains are surrounded by fine grains.[1]

Experimental setup:

Figure 2: Experimental set up at the beamline BM02 at the ESRF

The Figure 1 described the experimental setup composed by:

- A FReLoN 2D detector
- A CCD camera : pixelfly (with telecentric objective magnification x 1)
- A tensile machine Deben 5kN and the dog-bone samples (gage length of approximately 6mm x 1mm x 2mm)

Conditions:

Energy: 20 keV Size of the beam: 1000 x 300 μm^2
Detector: FReLoN Acquisition Time = 1 sec $\Omega = 10°$ $\delta \approx 10°$ and $\mu \approx 4°$
PixelSize1: 5.19e-05 PixelSize2: 5.19e-05 Rot1= 0.066772759393
Rot2= -0.180039758244 Rot3= 4.54654291373e-06 m Poni1: 0.00913355541215 m Poni2: 0.0368817153293 m Distance detector-sample: 26.3045 cm

The macroscopic strain or true strain is measured by Digital Image Correlation (DIC) technique thanks to a pixelfly CCD camera fixed on top of the sample to capture the sample surface images (fig.1). The reason to use DIC is related to the analysis of the deformation of the sample independently to the deformation of the tensile device or slips into the grips.

The size of the beam is large in order to probe large amount of grains. Indeed, the average grain size of the shell is about 5 μm and 25 μm for the core. During the tensile test, a 2D pattern was recorded approximately every 1 second. This choice is due to the requirement to get as much information as possible during the elastic domain, the elasto-plastic transition, and the plastic domain.

Sample Name	Phases	DIC analysis	XRD analysis	SEM	TEM
Ti64-MM-4	α and β	done	Started	Done but to be completed	To be completed
Ti64-MM25h-Prep-800-10m-6-	α and β	-	Started	Done but to be completed	-
Ti-IP-X	α	Done	Done	Done	-
Ti-JM-X	α	Done	Almost done	Done	-
Ti-MM-100h-Tilop-800-30m-2	α	Done	Almost done	Done	-
Ti-MM-100h-Tilop-800-30m-2-continu	α	-	difficult	-	-
Ti-MM-100h-800-30m-continu-2	α	-	difficult	-	-
Ti-MM-100h-800-30m-continu	α	-	-	-	-
Ti-JM3-800-30mn	α	-	-	-	-
Ti-IPb-800-30m		-	-	-	-
Ti64-IP-Tilop-800-10m-2	α and β	-	-	-	-
Ti64-IP-Tilop-3	α and β	-	-	-	-

Table 1: Samples deformed at BML02 during continuous uniaxial loading.

The table 1 described the samples deformed in situ during X-ray synchrotron measurements. Basically they are pure Ti and Ti-6Al-4V alloys elaborated by different metallurgical routes providing different microstructures. We have performed both continuous uniaxial loading and cyclic loading-unloading tests. The strain rate was fixed as small as possible ($2x10^{-3}$ s^{-1}).

I First results on pure titanium samples elaborated by SPS

The pure Ti samples were the first set of Ti alloys which have been analysed. Figure 3 displays the plot of the applied stress as a function of the macroscopic strain (measured by DIC) for the three different microstructures and for the continuous uniaxial loading. As expected from our preliminary ex situ laboratory measurements, a significant increase of mechanical properties is well established if we compare the Ti-IP (the Ti without any modification of the Initial Powder) with the Ti-JM and Ti-MM (both Ti-JM and Ti-MM are constituted with powders that have been milled). Moreover, there is no significant evolution of the ductility.

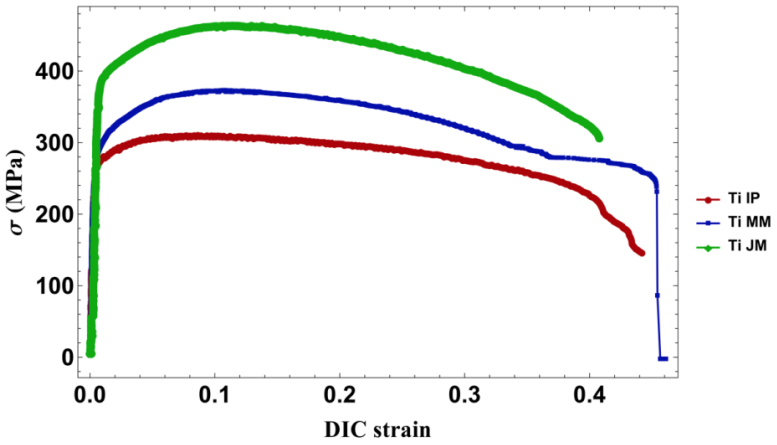

Figure 3: pure Ti stress-macroscopic strain curves during in situ XRD measurements.

Figure 4 is a magnification related to the first part of the tensile test. It displays the elastic domain. As one can see, the DIC strain measurements are rather scatter for the TiJM and TiIP. This scatter is currently under analysis, but does not induce any significant change on the complete test (fig.3). If we cannot find any solution to improve the analysis, we will determine the elasto-plastic transition with a relatively large uncertainty, but this is not the main objective of the present experiments (based on maximum strength, maximum elongation, elongation at necking).

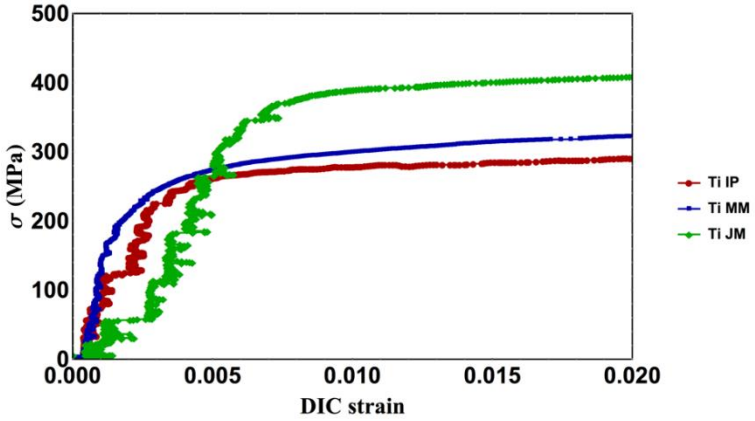

Figure 4: Zoom of the tensile curve from 0 to 0.02 of macroscopic strain

Such scatter of the strain values is due to the small size of the sample (and hence the small region of interest of the images (see ex: fig.5) and, maybe to some small vibration of the pixel camera installed on the BM02 goniometer. Figure 5 shows 2 typical images of a TiMM dog bone specimen used for the DIC analysis.

Figure 5: Picture of the gauge length of the tensile sample of the Ti MM at the initial state (left) and after the failure (left))

The Figure 6 displays a magnification of the images of Figure 5. The fact that the resolution is relatively poor and the brightness of the sample is changing during the experiments could also be taken into account for the strain analysis.

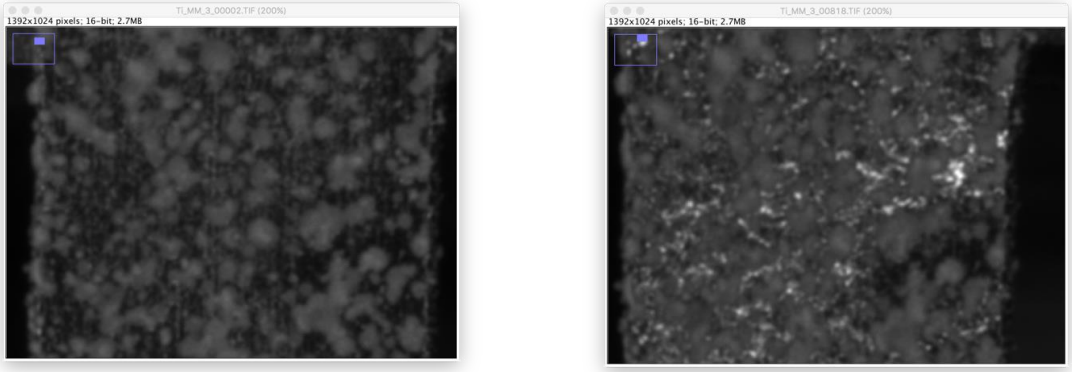

Figure 6: magnification (x20) of the gauge length of the Ti MM at the initial state (left) and after the failure (left))

A typical 2D diffractogram of the pure Ti-MM is represented in the Figure 7. As easily observed, the Debye-Scherrer (DS) rings are spotty. This discontinuous intensity distribution is related to the coarse grains of our microstructures (large coherent diffracting domains).

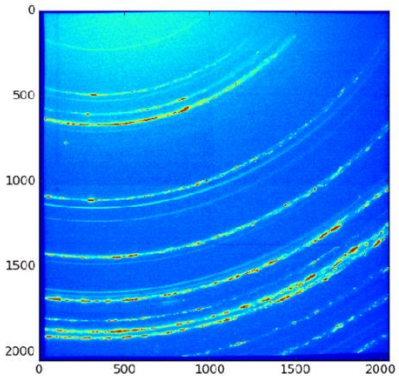

Figure 7: 2D diffractogram of a pure Ti MM at the initial state (σ close to 0 MPa)

During the plastic regime, the DS rings become continuous (Figure 8) and a texture evolution can be seen around the necking, i.e. maximum strength (fig.3). This is obviously related to a better distribution of the coherent diffracting domains induced by the strong plastic activity (defects introduced by the plastic deformation process such as dislocations and twins).

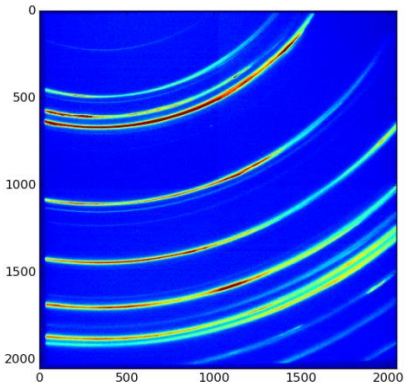

Figure 8: 2D diffractogram of a pure Ti MM at a deformed state (σ close to 350 MPa)

The Figure 9 illustrates a typical 2theta vs intensity diffractogram obtained after a caking of about 9° (performed with pyFAI) of the 2D pattern. The Ti-α (hexagonal close packed) is the only phase which composed our bulk Ti samples. The others peaks are related to the TiO2 powder that we add at the surface of our dog-bone sample to perform in situ calibration and check the sample drift during straining.

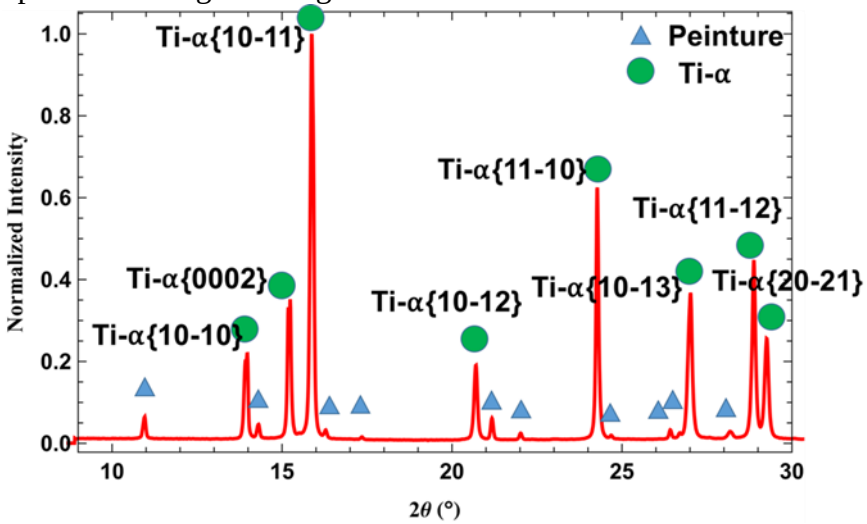

Figure 9: Diffractogram of a Ti MM after integration of the 2D diffractogram.

The Figure 10 displays the evolution of the diffractograms during straining of the Ti-α {0002} and Ti-α {10-10} peaks. As clearly shown, the change of the intensity of the peaks evolves oppositely, indeed the Ti-α {0002} integrated intensity increases while the integrated intensity of the Ti-α {10-10} decreases significantly.

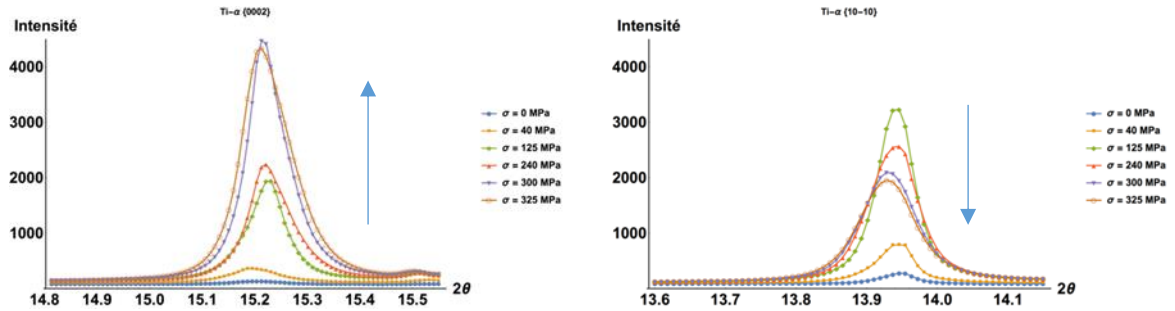

Figure 10: Evolution of the diffractograms of the Ti-α {0002} and Ti-α {10-10} peaks of Ti MM for different applied stresses.

Such evolution is attributed to a texture evolution induced by a mechanism of twinning, namely twins of type {10-12} which induced a rotation of about 85 degree of a part of the diffraction volume. The literature[2] already reports such mechanism during plastic deformation (Figure 11):

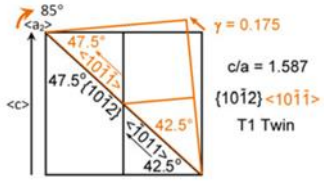

Fig. 1—The geometry of a T1 $\{10\bar{1}2\}\langle 10\bar{1}\bar{1}\rangle$ extension twin in Ti, in which the resulting c-axis is rotated 85 deg about the $\langle a\rangle$ axis common to both the twin and the parent (the a_2 direction points perpendicular to and into the page, and the twinned orientation is rotated 180 deg about the twin plane normal).

Figure 11: Geometry of the extension twin {10-12} in Ti (also called tensile twin).

Because of the discontinuity of the DS rings, the diffraction strain analysis is quite difficult. This DS rings discontinuity was expected because of the large grain size, but we have thought before experiment that it would be much less pronounced thanks to large beamsize and small incident angle. Anyway, a compromise must be found. Obviously a very large caking reduces the effect of the spottiness of the DS rings while a small caking allows having information in a specific direction in the reciprocal space in order to have a reliable measurement of the elastic strains. To succeed, around 9° of caking was considered. We obtain satisfactory "Intensity vs 2θ" curves (Fig. 6) and monotonous evolution of lattice strains as shown in Figs. 12 and 13. Moreover, Figure 12 clearly shows the effect (and also the requirement) of the correction on the lattice strain. The blue curve related to the TiO2 diffracting planes should be ideally equal to zero all along the tensile test. Hence, it is necessary to correct the apparent lattice strain measurement as illustrated on the Ti-α{0002} peak.

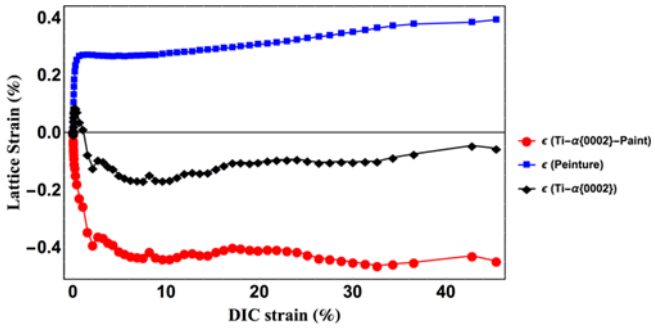

Figure 12: Effect of the correction of the lattice strain thanks to the TiO2 peaks.

Figure 13 displays the evolution of the lattice strains for multiple orientations of the Ti crystallographic planes. As expected, depending to the considered orientation, some families of crystallites deform more than others due to elastic anisotropy and exhibit an elasto-plastic transition at different applied strain. This is mainly due to their orientation with respect to the tensile direction and to the stress that should be applied to induce their plastic deformation (critical resolved shear stress). The Ti-α{0002} is softest than all the other crystallographic orientations as expected.

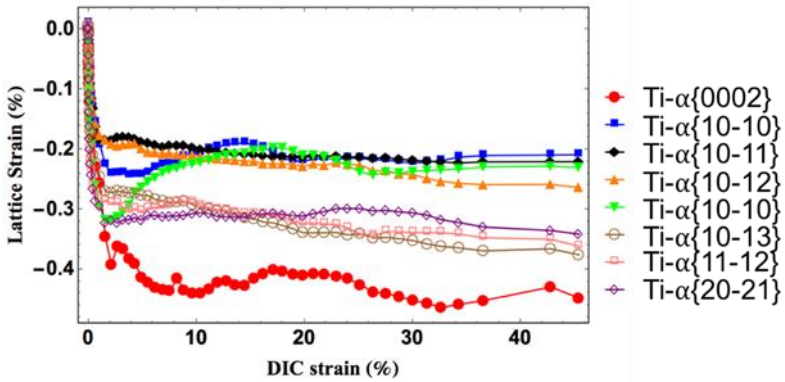

Figure 13: Evolution of the lattice strain of several crystallographic orientations of the Ti MM versus true strain.

II results on Ti-6Al-4V samples

The Japanese partners had a big problem with their Spark Plasma Sintering apparatus a few weeks before beamtime. Hence, the samples have been produced in a private society (outsourcing) and the harmonic design on the first sample that we have observed by scanning electron microscopy seems to be not very well established. Investigations should be done to confirm whether the samples are harmonic or not for that microstructure. XRD and DIC analyses have not yet been performed, Scanning Electron Micrograph is under progress.

III Conclusion:

To start the analysis, we have focused only on the pure Ti and interesting results seems to have been obtained. A deeper analysis is currently under progress. Concerning Ti-6Al-4V, the analysis is under progress and we have some problem with one of the three microstructures. The cyclic testings have not yet been analysed.

The results are still being investigated. Difficulties due to the spotty DS rings seems to be override to perfectly succeed to obtain the lattice strain evolution during the continuous tensile tests of pure Ti. The texture evolution has been measured and will help to capture the deformation mechanisms related to the different microstructures.

Three communications were done with the first analysis of pure Ti samples:

1) **Invited conference** at the international Symposium Soft/Hard 2017 (6th International Symposium on Functionalization and Applications of Soft/Hard Materials) (Biwako-Kusatsu, Japon), Speaker: T. Sadat, university of Poitiers (20 au 22 Janvier 2017).

2) **Best Poster award** at the international Symposium Soft/Hard 2017 (6th International Symposium on Functionalization and Applications of Soft/Hard Materials) (Biwako-Kusatsu, Japon) : "Pure Ti, mechanical properties and in situ tensile test under Synchrotron X-Ray Diffraction". S. Kosuga, university of Poitiers and Ritsumeikan university (20 au 22 Janvier 2017)

3) « Titane élaborés par Frittage Flash : étude mécanique couplée à la Diffraction des Rayons X » T. Sadat, P.O. Renault, P. Godard et al., 10-12 avril 2017, conférence annuelle plasticité, Rennes, France

References

[1] K. Kurokawa et al., Application of High-pressure gas milling process to pure Titanium for harmonic structure design, Advances in Materials and Processing Technologies, 2016,2(2),202-208.

[2] T. R. Bieler et al., In situ characterization of twin nucleation in pure Ti using 3D-XRD, Metallurgical and Materials Transactions A: Physical Metallurgy and Materials Science, 2014, 45, 1, 109-122