Long Term Project Report MA-1876 : half first year report

"Use of coherency and large field of view for fast in situ multi-resolution imaging"

The aim of this long term project is to extend the material science knowledge and instrumentation by the development of high speed in situ multi resolution absorption and phase tomography. The LTP started in june 2013, the 6 shifts were used in October 2013 and the next 6 shifts will be used in February 2014.

1. Beamtime Used

Scheduling period	Beamline(s) Used	Shifts Used	Summary of results obtained
2013 /II	ID19	6	- Validation of technical developments on real experiments (live 2D section, live 2D correlation, live control and interaction) - Investigation of competitive nucleation of intermetallics and aluminium primary phase during solidification of Al-Si-Fe alloys - Destabilisation of air/liquid interface in reinforced foams - Controlled multi-resolution characterisation of fibre reinforced composites - Damage during continuous tensile experiments on metallic / amorphous composites

2. Resources Provided by User team (financial, personnel, technical...)

Staff

- July – October 2013 : One post doc : Michelle Alvarez (SIMAP) based 60% at ESRF
- October 2013 – January 2014 : one post doc : Rémi Daudin (SIMAP) based at 60% at ESRF
- June 2013– January 2014 : two researchers Pierre Lhuissier, Luc Salvo (SIMAP) 10% on the LTP
- June 2013– January 2014 : Two technicians Franck Peloux et Xavier Bataillon (SIMAP): 4 weeks
- June 2013– January 2014 : One technician Emmanuel Garre (SVI) : 3 weeks
- June 2013– January 2014 : One researcher H. Leclerc (LMT) : 1 week

Technical :

- Development of a high temperature tensile /compression device for fast in situ experiments (SIMAP)
- Modification of a high temperature induction furnace with vacuum or gas for fast in situ experiments (SVI).
- Specific user friendly macros for fast in situ acquisition with PCO DIMAX camera and fast reconstruction

- Live control development of fast in situ tomography : 2D live correlation and slice visualisation

Financial :

In the project financial investment have been planned concerning GPU blades, Cameras and sample environment. At the moment only sample environment investment have been used. Discussion will be conducted with ID19 about the GPU blades and cameras.

3. **Technical and Scientific Milestones Achieved** (in relation to the milestones identified in the original proposal).

During these first months of the LTP that started in june 2013 we decided to use only 6 shifts in october 2013 in order to test during real experiments the various technical developments on which we focused. We will first present these technical developments that mainly match the milestone of the LTP proposal. The scientific applications were selected in order to match the technical development and the chosen set up for this session (U 17.6, PCO DIMAX, multi-resolution optic, sample environment …) rather that strictly following the planned experiments.

Task 1 : Technical development

Data acquisition : Live 2D section (*online 2D visualization in less than 1s*) P.Lhuissier, R.Daudin, (SIMaP)

Implementation of a python code and development of an acquisition macro: The macro records the sinograms in a prescribed folder. The python code computes the center of rotation from the first sonogram or use a prescribed value. Then it generates dynamically the .par files and submits the reconstruction to PyHST. The python code also launches an ImageJ plugin that waits for the reconstructed slices and automatically insert them in a stack. The current implementation delivers approximately one slice every 5 seconds. Another version of the acquisition macro and of the python code provides simultaneously a stack of reconstructed slices and a stack of 2D radios for a prescribed angle.

Required improvements : To be finished in February 2014.

- Faster reconstruction : It might be that performing the same process on a local machine and not on nice is sufficient. Otherwise it means a direct implementation of the python code as part of PyHST in order not to initialize and launch PyHST for each slice.
- Faster and more robust visualization : it doesn't necessarly means to avoid ImageJ, use of a local machine might also be sufficient.

Data acquisition : Live 2D correlation (*online 2D correlation to monitor tensile experiments*) H.Leclerc (LMT), P.Lhuissier, R.Daudin (SIMaP)

Implementation of a 2D correlation code and development of an acquisition macro : The macro takes radiographs for a prescribed angle and records them in a prescribed folder. The correlation code computes the mean axial strain and display dynamically a graph of the strain as a function of the scan number. The current implementation delivers approximately one value every 5 seconds.

Required improvements : To be finished in February 2014.

- More robust and user friendly correlation. The correlation is robust for one that well knows how to tune the parameters depending on the observed material. But a simple user is stuck to the already prescribed parameters and thus cannot obtain consistent results.
- Faster reconstruction : It might be that performing the same process on a local machine and not on nice is sufficient. Further tests need to be performed in order to measure the time of the acquisition and of the correlation.

Data acquisition : Multi-resolution 1 camera (*user friendly multi-resolution for fast acquisition rate around 10s using PCO edge or DIMAX camera*) E. Boller (ESRF), L. Salvo, P. Lhuissier, M. Alvarez (SIMaP),

We performed development of acquisition macros that allows interactive selection of resolution during experiments and easy resolution change during the. The macros allows to target a specified point (X,Y,Z) in the sample for local tomography. Change between resolutions takes approximately 10 seconds. This was tested on the PCO DIMAX camera. See EZRT experiments in task 2.

Required improvements : To be finished in February 2015.

- Simultaneous motion of the motor to reduce the time to change the resolution (slits, objectives, focus, positions …).
- User friendly improvements : Some checks on motors positions in order to avoid mistakes and selection of the point for higher resolution tomography by click in a 3D volume (requires fast 3D reconstruction)

Sample environment :

Development of a high temperature tensile machine (*controlled atmosphere up to 1600°C*) P. Lhuissier, F. Pelloux, X. Bataillon, L. Salvo (SIMaP)

Work have been done on a tensile / compressive machine using inductive heating (not under controlled atmosphere for the moment) : this can handle 200 N and a slip ring is mounted at the top allowing motor control and thermocouple control. Figure 1 shows the set up that will be tested next session in February.

Required improvements : To be finished in February 2015

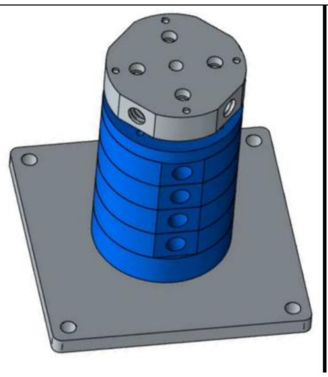
- Design another set up for tensile experiments that need controlled atmosphere

Modification of a high temperature furnace (*controlled atmosphere up to 1600°C*) E. Garre, E. Gouillart (SVI), E. Boller (ESRF)

This task was scheduled later on the planning but it starts. The aim was to modify the induction furnace developed for ID15 by ESRF so that continuous rotation can be possible. Specific slip ring (2 fluid input and output) has been selected and bought (MPCI society) and the design has been modified to adapt the XY motors also (see figure 2a and 2b). Furthermore a double wavelength pyrometer has been bought to control temperature. This set up is available for the MR tomo for the moment and will be tested in march during the shutdown and in real experiments in june 2014.

Required improvements : To be finished in February 2015

- Design another set up for similar to this one with the HR tomo if the use on the MR tomo is not satisfying for 3D imaging with 1 micron optic.

		
Figure 1 : high temperature tensile set up	Figure 2a : initial ESRF furnace	Figure 2b : slip ring below the furnace with 2 fluid input/output

Volume reconstruction : Backprojection algorithm and paganin algorithm (*faster reconstruction on multi-GPU blades*) E. Gouillart (SVI) , L. Salvo, P. Lhuissier (SIMaP)

This was already developed by A. Mirone, P. Tafforeau in PyHST2 for non multi-edf scans. At the moment the development that have been performed for multi-edf scans (obtained from PCO DIMAX) is to be able to perform a fast slice reconstruction with various parameters and select the good ones for full reconstruction.

Required improvements : To be finished in September 2014

- Full compatibility between non multi-edf macros and multi-edf macros for fast slice reconstruction : this will ensure the use of GPU PyHST implementation of Paganin algorithm

Volume reconstruction : Helicoidal reconstruction (*provide and adapt existing multi-GPU code for helix-reco from the lab to PyHST*)

Nothing performed on this point. To be finished before September 2014.

Task 2 : Scientific applications

Elaboration : Intermetallic Al-Fe-Si (*influence of cooling rates and additional elements on the nucleation and growth of AlFe5Si intermetallics*) L. Salvo, P. Lhuissier (SIMaP), E. Boller (ESRF).

The recycling of cast aluminium silicon alloys often lead to an increase in the level of Iron content which promotes the formation of beta Al-Si-Fe intermetallics. Until now in situ tomography was performed with a time resolution that is too long to clearly established if intermetallics nucleate before or after the primary aluminium phase scans about 40s.

Thanks to recent developments (ID19 refurbishment) scan time was reduced to be able to reach solidification speed close from industry one and phase nucleation. We performed scans with various acquisition time (1.4s , 0.7s and 0.2s!) with an optic of 1.1µm and the PCO DIMAX camera thanks to the U17.6 Thanks to the recording temperature near the sample it was possible to launch the scan in the region of interest just before any phase could appear in this Al-10%Si with 1.35% Fe. This is a bit tricky at high acquisition speed since the camera can only store 40 scans and thus the time window for imaging can be very small (less than 1 seconds for a scan with 0.2s !!). it seems that depending on the level of iron in the small sample we can observe all the theoretical features : alpha phase then beta, alpha phase and beta together, beta then alpha phase (see figure 3 and 4). This is the first time such nucleation is observed in 3D with such a high temporal resolution.

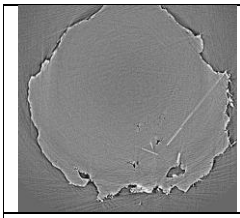


Figure 3 : white beta phase nucleates before alpha phase

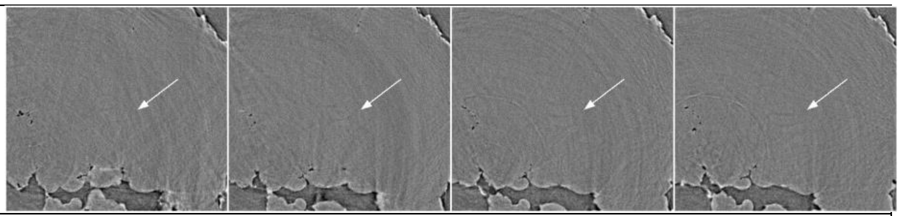


Figure 4 : 4 consecutive slices in the volume scanned at 0.2s : in this case we can see alpha phase nucleating before beta phase (near the white arrow)

Foams : X-ray analysis of paper fibers in foam A. Meagher(HZB)

The foams were produced from water with 1% dissolved surfactant and 1% fibers. The rayon fibers had an average diameter of roughly 20 microns. Data regarding pine fiber characteristics is not currently available. The foam samples were imaged with a beam intensity of 35 KV with the camera located at 300 mm distance from the sample. 2880 projections were taken for each tomography. The objectives used during the experiments were 10x and 20x. Each tomography was a region of interest tomography. Two experiments were performed

- Variation of fiber distribution with distance from the foam liquid interface for both the synthetic and natural fibers.
- Temporal evolution of synthetic fibers within the foam. This was performed by imaging the same region of foam every 5 minutes over the course of an hour.

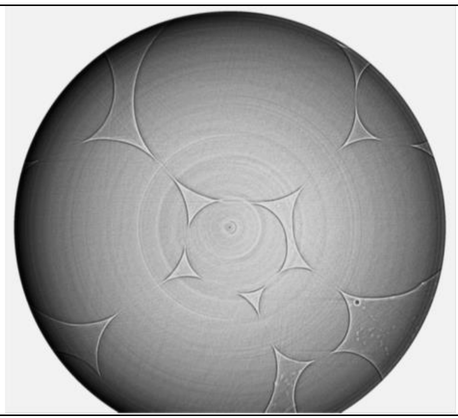


Figure 5 : 2D section of the foam : fibers can be seen in the top right of the image

A reconstructed image of a foam sample stabilized with Rayon fibers is seen in the figure 5. The fiber are seen in the liquid phase of the foam in the top right of the image. The presence of the fibers is seen to alter the surface of the bubbles, resulting in almost 'rough'. We have seen in some of the reconstructions that the fibers appear to be more prevalent within the foam near the foam liquid interface.

Composites, multi-resolution : S. Gerth (EZRT), S. Zabler (LRM) :

After the adaption of the measurement macros for multi resolution measurements the stem of roses was measured in three different resolutions, 2.75µm/voxel, 1µm/voxel and 0.5µm/voxel. In the first implementations, the center in all three measurements was the same, but we tuned the setup to do a higher resolution and a shift of the center at the same time. So it was possible to measure the whole stem in the lowest resolution and afterwards only the more interesting outer ring of the stem. This is shown in figure 6

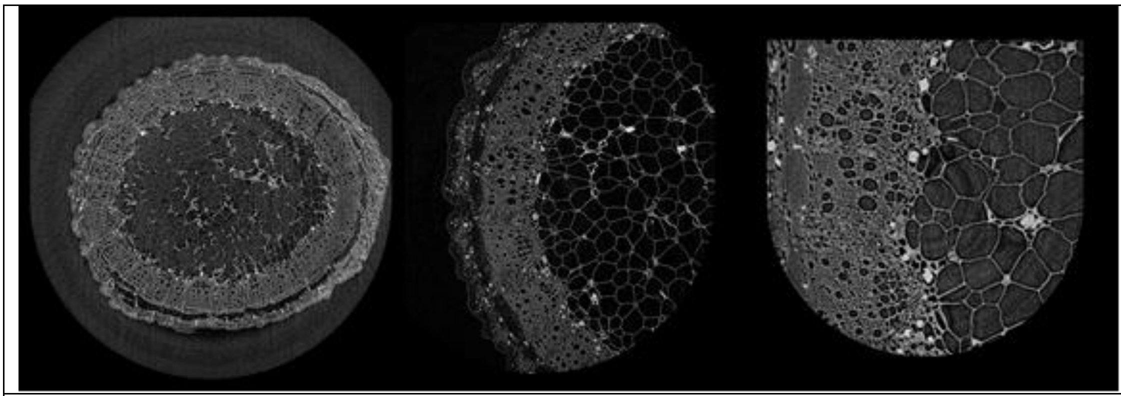


Figure 6: Stem of roses in 2.75µm, 1µm and 0.5µm resolution (from left to right).

The second sample system was piece of CFK. To observe the coarse as well as the fine structure of the CFK multi resolution measurements were conducted as well, but this time without a translation of the center of magnification due to the regular arrangement of the CFK.

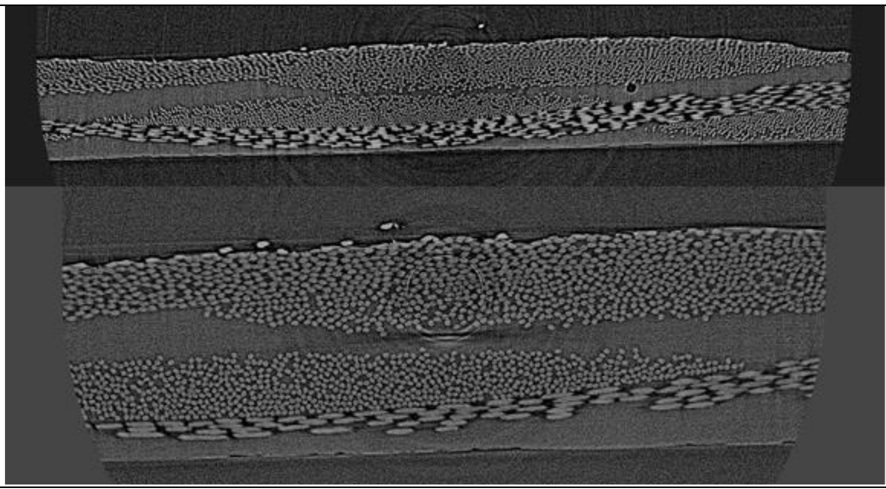


Figure 7: CFK measured in different resolutions of 1µm and 0.5µm

Damage at room temperature: composite (*influence of size and volume fraction of particles on crack growth*) A. Ferré, E. Maire, J. Adrien (MATEIS)

Amorphous crystalline composites with different volume fraction were prepared by spark plasma sintering (SPS) of mixing of aluminium and amorphous metallic glass (Zr57Cu20Al10Ni8Ti5) powders with similar particle size (around 50µm). After SPS, composites were extruded at the SIMAP Laboratory. Two kind of composite were studied. The first one was a mixing between aluminum matrix (99% pure) and metallic glass inclusion with 1% volume fraction. The second one was a mixing between aluminium magnesium (Al-Mg 5083) and metallic glass with a volume fraction from 1% and 4%. Two different behavior were observed during the in situ tensile experiments at room temperature with the PCO DIMAX camera.. Damage in matrix and decohesion between inclusions and matrix were observed in case of aluminium matrix (Al99%). For aluminium magnesium (5083), we observed few decohesion and few particles crack as we can see on Figure 8a and 8b. The aluminium magnesium matrix (5083) is harder and less ductile than aluminium (99%), that's why we could see particles crack in Al-Mg.

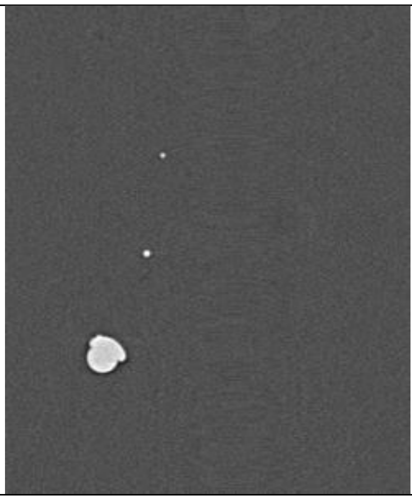


Figure 8a : initial 5083 based composites with 1% amorphous 50µm particles (in white)

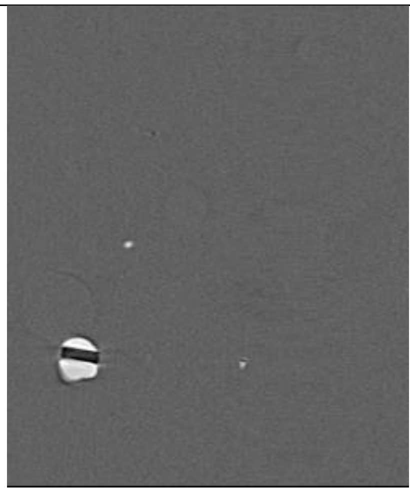


Figure 8b : particle cracking just before rupture in the 5083 based composites of figure 8a.

Task 3 : Dissemination

development of user friendly GUI in Qt, linking to SPEC language of ESRF, E. Boller, E. Papillon (ESRF), M. Alvarez, R. Daudin, P. Lhuissier, L. Salvo (SIMaP).

At the moment we defined the main macros that are needed when users want to perform in situ fast tomography with PCO DIMAX. All these macros have defined and user friendly parameters. A user can now define a text file with all the sequence of acquisition he wants to perform. The user can easily switch to any sequence during acquisition. This text file is working now and was tested in the multi-resolution experiments and solidification experiments.

Required improvements : To be finished in September 2014

- Make a full documentation in the ID19 wiki of all the macros available
- Define a GUI based no these text file

4. List of publications directly resulting from beamtime used for this Long Term Project

No publication have been done so far since these are preliminary results but some of them will be presented at the international conference 3DMS (TMS) in 2014