| ESRF | **Investigation by in-situ nano-tomography of cavities nucleation and growth in light alloys during high temperature deformation** | **Experiment number**: MA3507 |
|---|---|---|
| **Beamline**: ID16B | **Date of experiment**: from: 05/05/2017 to:08/05/2017 and 01-02/07/2017 | **Date of report**: |
| **Shifts:** 12 shifts | **Local contact(s)**: Julie Villanova | *Received at ESRF:* |


**Names and affiliations of applicants** (* indicates experimentalists):

Julie Villanova: ESRF
Richi Kumar : ESRF/Laboratoire SIMAP Université Grenoble Alpes
Pierre Lhuissier : Laboratoire SIMAP Université Grenoble Alpes
Luc Salvo : Laboratoire SIMAP Université Grenoble Alpes


**Report:**

**1. Overview**

In the context of fossil resources depletion and environmental challenges for reducing greenhouse gases emission, finding solutions for saving energy in particular in the transportation sector has become vital. This driving force for lightening the structures pushes for the use of light alloys such as magnesium or aluminium based alloys. Forming of such alloys is highly dependent on the crystallographic structure, on the micro-structure (grains size, grains shape, texture, intermetallics...) and on the loading conditions (temperature, strain rate, uni-axial tension, uni-axial compression...). However there are voids or cavities nucleation, growth and coalescence during high temperature deformation which, due to degradation, limits the forming of light alloys.
The study we report here aimed to understand the nucleation and growth of cavities of those light alloys to prevent damage during high temperature forming. Then, the purpose of the experiment was to carry out *in-situ* 3D nano-imaging on Aluminium and Magnesium alloys during their deformation at high temperature.

**2. Measurement/data**

The experiment took place in May2017 and July at ID16B beamline (the experirment has been split in two session in order to optimize the in situ set up if needed and increase the chances of success of such experiment). It used the nano-tomography set up and the high temperature furnace available at the beamline. The beam has been aligned in order to have a nano-beam of 50 nm with high flux (5.6 x$10^{11}$ph/s) with an energy of 17.5keV. One in situ measurement consists of imaging at different resolutions the alloy sample that is mounted in a tensile device (ie under constrain) (Figure1(a)) and heated in the high temperature furnace at 400°C. Indeed, while imaging high temperature deformation, it was required to image the notch for computing mechanical deformation parameters and examine deformation in real time, which meant low resolution imaging. But at the same time imaging at high resolution was also imperative, in order to image nucleation and growth of creep cavities. Multiscale acquisition was the ideal approach for this. Thanks to the conic beam, changing the distance of the sample with respect to the detector / focused X-Ray beam, changes the field of view and the consequent pixel size of acquisition, which allows multi resolution imaging. Employing this capability, a set of Low Resolution (LR) radiographs with a field of view of 826 × 826 × 697 $\mu m^3$, pixel size of 645nm followed by a set of High Resolution (HR) radiographs with a field of view 128 × 128 × 108 $\mu m^3$, pixel size of 100nm were acquired by moving the sample between the focused nanobeam and the detector (Figure 1 (b)). The motion of the furnace was synched with that of the sample, and consequently it also moved along with the sample. A total of 10 samples have been succesfully scanned.

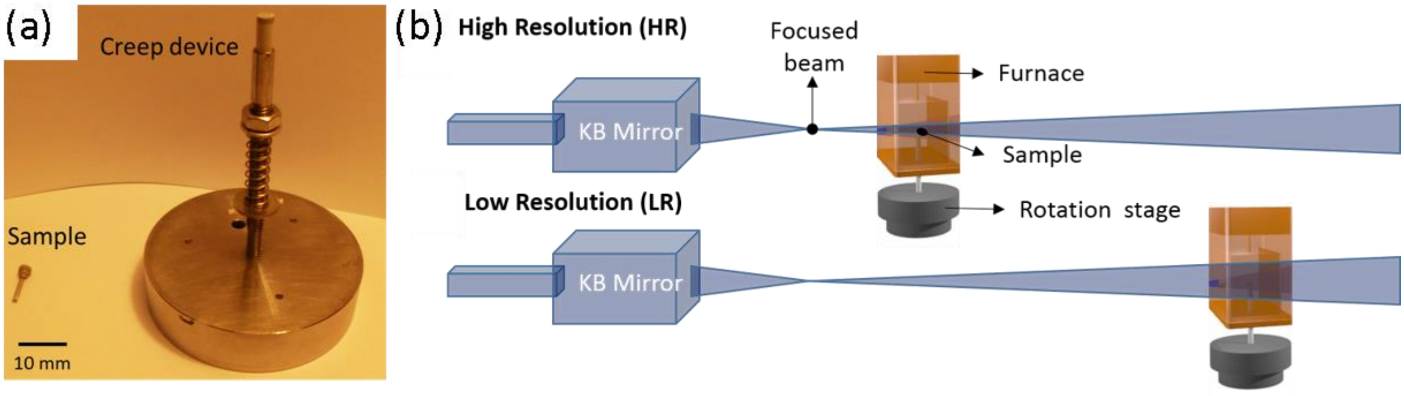


*Figure 1: (a ) The tensile device as it has been mounted on the rotatoin stage  (b) Setup of the in situ multi-reoslution X-ray nanotomogrpahy at ID16B.*

## 3. Results

The 3D volumes reconstructed has been analysed using classical image analysis and digital volume correlation to track the nucleation and growth of the cavities in the samples as it is illustrated in Figure 2. Quantitative results show that in the Aluminium alloys the cavities initially grow by diffusion, while the growth mechanism changed to plasticity near failure. For the magnesium alloys, pre-existing cavities grow into complex shapes. Moreover, during deformation, each cavity's shape evolves in a different manner. A shape based classification of evolution of cavities has proposed to understand this. As a result four evolution types emerged and it has seen that one cavity generally grew by a combination of several evolution types.

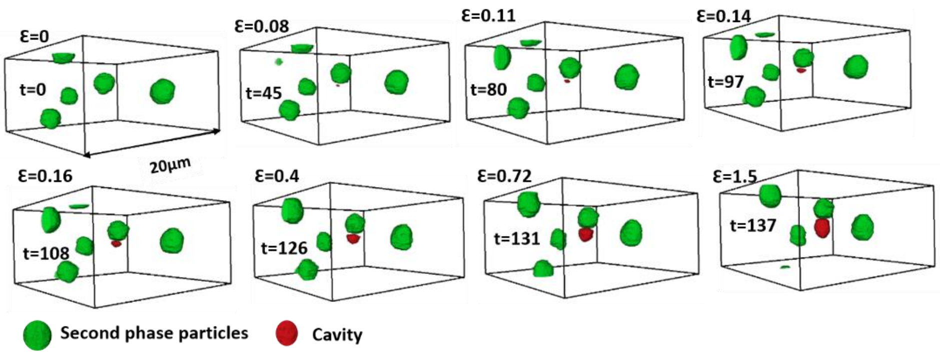


*Figure 2: 3D rendered view of nucleation and growth of cavity (red) alongside a second phase particle (green) as the AC5 sample strains ($\mathcal{E}$) with time (t, in minutes)*

All the results have been published in the Manuscript of the PhD thesis of Richi Kumar [1]  as well as in a paper in Acta materialia [2]. A second paper is under preparation and will be submitted in the coming month [3].

## 4. Conclusion

In summary, successful in situ X-ray nanotomogprahy experiment  has been performed on light alloys samples during high temperature deformation. Besides, providing experimental data in regime that has not been reported previously, this work gives precedence for in situ nano-imaing experiments in damage investigation of materials. Further improvements have to be made in order to have better and reproducible results. As for example, improve the control of the strain rate by developping a new motorised mechanical device allowing strain / pull on the sample at a constant rate.

## 5. References

[1] PhD manuscript of Richi Kumar, defended 29/10/2019 : http://www.theses.fr/2019GREAI065

[2] R. Kumar, J. Villanova, P. Lhuissier, L. Salvo, In situ nanotomography study of creep cavities in Al-3.6-Cu alloy, Acta Mater. 166 (2019) 18–27. https://doi.org/10.1016/j.actamat.2018.12.020.

[3] R. Kumar, J. Villanova, J-J Blandin, P. Lhuissier, L. Salvo, Elementary growth mechanisms of creep cavities in superplastic AZ31 alloy revealed by in situ nanotomography characterization, to be submitted to Acta Mater.