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On the Application of X-ray Microtomography in the Field of Materials Science

By . ric Maire, Jean-Yves Buffière, Luc Salvo, Jean Jacques Blandin, Wolfgang Ludwig, and Jean Michel Létang

The principle of the tomography technique and the different possible set-ups, which can be used to obtain medium-(10 μm) and high-(1 μm) resolution, three-dimensional, non-destructive images, are shown in this paper. Illustrations are made of the applications of the technique in the field of materials science. Examples are given for medium-resolution images of metallic foams and model metal matrix composites that are reinforced with spherical particles. High-resolution examples are shown for aluminium alloys. For low-absorbent materials we show that the phase contrast obtained using synchrotron radiation can provide a valuable solution. The quantitative use of these images, coupled with in-situ tensile tests or used for the simple analysis of the initial microstructure of several structural materials, is also described.

1. Introduction

This paper describes the activities of several laboratories that are members of the CNRS research federation "Matériaux de structure et propriétés d'usage" in the region Rhône-Alpes (FR MAT SPU) in the field of materials characterization by means of X-ray microtomography. This technique is non-destructive and permits to obtain three-dimensional (3D) images of the interior of a material. It shows all the microstructural features (other phases, inclusions, cracks, pores, ...) inducing a modification of the attenuation or of the optical phase along the path of an X-ray beam. It has been used in medicine for about 30 years with a typical resolution of around 300 micrometers. Recently, third-generation synchrotron radiation facilities became available. When working at the first of these new sources, the European Synchrotron Radiation Facility (ESRF) in Grenoble, it was possible to improve the resolution to the micrometer level. This allows the extension of the observation of the microstructure of materials in 3D. More precisely, when coupled with a tensile stage, the microtomographic equipment installed at ESRF on beamline ID19 has permitted to obtain 3D images of the microstructure of the materials as well as their evolution under load.

The aim of this paper is to illustrate the activities carried out in the framework of the FR MAT SPU, which has brought the "Région Rhône-Alpes" to a leading position in this field. Thanks to the studies performed in the last three years, X-ray microtomography has now proved to be a very powerful tool

[*] *Dr. E. Maire, Dr. J. Y. Buffière*
CNRS GEMPPM bat. St. Exupéry
INSA de Lyon, 20 av. A. Einstein
F-69621 Villeurbanne (France)
E-mail: eric.maire@insa-lyon.fr

Dr. W. Ludwig
ESRF, BP20
F-38043 Grenoble Cedex (France)

Dr. L. Salvo, Dr. J. J. Blandin
Institut National Polytechnique de Grenoble (INPG), Génie
Physique et Mécanique des Matériaux (GPM2) ENSPG-VJF,
BP46, F-38402 Saint-Martin d'Hères (France)

Dr. J. M. Létang
CNDRI bat. St. Exupéry
INSA de Lyon, 20 av. A. Einstein
F-69621 Villeurbanne (France)

to investigate the microstructure and the deformation mechanisms of various structural materials, which range from foams and alloys to high performance composites. The principle of the technique will be presented with an emphasis on recent improvements. Qualitative and quantitative illustrations obtained in collaboration between different laboratories of the federation will be given each improving our understanding of basic problems in the field of materials science.

2. Principle of the Technique

2.1. Basis

2.1.2. X-ray Radiography

X-ray radiography physics is based on the Beer-Lambert law (Eq. 1), which gives, for a given beam, the number of transmitted photons N_1 of energy E in terms of the number of incident photons N_0 of energy E and the corresponding attenuation coefficient μ of the sample along that beam:



Luc Salvo is a lecturer at INP Grenoble where he teaches Materials Science and Materials Selection. His main interest in research are Materials and process selection and the study of relationship between microstructure and mechanical properties (in semi-solid materials, metal foams and superplastic alloys). Microstructure is characterised using high resolution X ray microtomography

in collaboration with the European Synchrotron Radiation Facility in Grenoble (ESRF)



Wolfgang Ludwig has performed his thesis at the European Synchrotron Radiation Facility (ESRF) and obtained his PhD degree in Physics from the University of Munich. He has been working on 3D neutron and X-ray micro-imaging and he currently holds a post-doctoral fellow position in the X-ray microscopy group at the ESRF.



Jean-Michel Létang received the PhD degree in signal and image processing from the INP-Grenoble in 1993. He is currently a lecturer at the INSA of Lyon, and a research associate in the CNDRI laboratory. His research interests include 3D-tomography, X-ray imaging and 3D image processing.

$$\frac{N_1}{N_0} = \exp \left[- \int_{S \in \text{ray}} \mu(s) ds \right] \quad (1)$$

The attenuation coefficient μ varies along the beam and depends on the local composition of the sample and the photon energy. In the photoelectric domain μ typically follows a law of the type:

$$\mu(x,y,z) = K \rho \frac{Z^4}{E^3} \quad (2)$$

where K is a constant, ρ and Z are the density and the atomic number of the investigated material, respectively, and E is the energy of the incident photons. For a polychromatic beam, the Beer-Lambert law (Eq. 1) has to be integrated over the whole spectrum.

This attenuation law is the basic rule to explain the contrast observed in the X-ray radiograph of a bulky material because each point of a detector placed behind the sample is situated in front of a different path. If the material is heterogeneous, the integral value of $\mu(x,y,z)$ varies also with the beam orientation, in particular x , y , and z .



Jean Yves Buffière is a lecturer at INSA Lyon where he teaches Materials Science and Materials Selection within the Industrial Engineering Department. His main interest in research is the study and modelling of plasticity and damage of metallic micro-heterogeneous materials during monotonic or cyclic deformation. During the last 5 years, in collaboration with the European Synchrotron Radiation Facility in Grenoble (ESRF) he has been working on the application of high resolution X ray tomography to the characterisation of structural materials.

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Jean-Jacques Blandin is currently researcher at the Centre National de la Recherche Scientifique. He is working at the Institut National Polytechnique de Grenoble (INPG). His research topics are essentially high temperature deformation and damage of materials, with a particular attention given to superplastic deformation. These activities, which include grain refinement procedures via thermomechanical treatments, mechanical testing and microstructural characterisation, are carried out mainly on aluminium and magnesium alloys.



Eric Maire received the PhD degree in materials science from Insa de Lyon in 1995. He is currently a research associate in the GEMPPM laboratory at Insa. His research interests include 3D tomography applied, mechanical properties of heterogeneous metals.

2.1.2. X-ray Tomography

Radiography is a projection of a high amount of information on one single plane and the resulting image can be difficult to interpret if the microstructural feature complexity along the thickness of the sample is important. Tomography overcomes this drawback by combining the information of many radiographs, each being taken with a different orientation of the sample in front of the detector. When the angular step between each radiograph is small enough it is possible to compute the local value of the attenuation coefficient at each point of the sample from the complete set of radiographs. This reconstruction is performed with an appropriate algorithm based on the filtered back-projection.^[1]

2.2. Setup

Different setups can be used. They all comprise an X-ray source, a rotation stage on which the object is fixed, and an X-ray detector. The easiest way to get digitized images is to directly use a two-dimensional radioscopic detector composed of a fluorescent screen that transforms the X-rays into visible light, which are then transferred by appropriate optic lenses to a cooled CCD camera (lines and rows of sensitive elements). Every tomographic setup has to be properly calibrated: the 3D geometry must be reliable and accurately known and some constraints must be fulfilled (number of projections, location of the rotation axis, sample size, ...). The crucial point in applying tomography to materials science is the achievable spatial resolution. Its limit value is mainly governed by the available photon flux at the level of the sample and by the setup itself as described in the two following sections. For medical tomography (2D scanner) this limit resolution is of the order of 300 μm . Materials scientists wishing to see and distinguish features with a size of the order of 1 μm had to develop appropriate tools.

2.2.1. Medium Resolution Microtomography

For a limit resolution of the order of 8 μm (medium resolution), a cone-beam system can be used with a classical microfocus X-ray tube as the source. Such a device has been for instance assembled in the CNDRI laboratory at INSA de Lyon. With this diverging geometry (see Fig. 1a), the magnification can easily be tuned by changing the spatial position of the sample between the source and the detector in this case. The maximum resolution is mainly due to the size of the microfocus, which introduces a blur in the projected image. This focus size has to be as small as possible in order to get the best resolution. However, the focus size highly depends on the power delivered by the X-ray tube: the lower the power, the better the focus size. Thus, for a realistic analysis (i.e., acceptable acquisition time) the reconstruction resolution is a trade-off between the focus size and the delivered X-ray power, to prevent the flux at the level of the sample to become too low. In such a laboratory

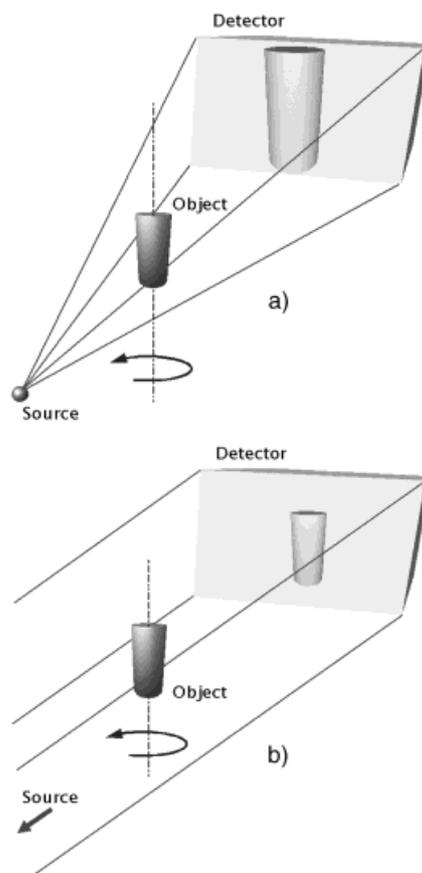


Fig 1. Scheme of the tomography technique with a diverging (a) and a parallel (b) geometry.

setup, the X-ray source is polychromatic, which may introduce artifacts due to beam-hardening and does not allow a quantitative reconstruction of the absorption coefficient without prior calibration.

2.2.2. High-Resolution Microtomography

It was shown in the previous section that setups using X-ray tubes are limited because of the flux delivered by this kind of source. The best quality images in terms of signal-to-noise ratio and spatial resolution allowing high-resolution microtomography nowadays are obtained on instruments located in synchrotron radiation facilities. The origin of the X-rays is here completely different. Synchrotron radiation is produced by ultra-relativistic electrons in a storage ring when they are accelerated by a magnetic field. The X-rays are emitted in a narrow cone tangentially to the trajectory of the electrons (i.e., tangentially to the storage ring). Such a source can be found for instance at the ESRF in Grenoble. The X-ray beam produced is very interesting for microtomography because of its many original features,^[2] the main of which being the very high intensity of the X-ray beam and the monochromatic character of the beam (this suppresses for example the beam hardening effect and allows for quantitative reconstructions). The X-ray beam can be assumed to be parallel in such a device (see Fig. 1b).

The ID19 beam line at ESRF is devoted to high-resolution imaging and features an energy-tunable (7–100 keV) X-ray beam of low divergence ($\sim 1 \mu\text{rad}$). The dedicated microtomographic setup consists of a precision mechanical sample stage (rotation and translation are possible) combined with a fast, high-resolution detector system. The latter consists of a fluorescent screen (YAG:Ce), which transforms the X-rays into visible light, and microscope optics to project the image on the cooled 1024^2 CCD camera, with a dynamic range of 14 bits, a fast readout (0.22 s/frame), and low-noise electronics ($3 e^-/s$). This unique combination allows a resolution of the order of $1 \mu\text{m}$ to be obtained routinely.

2.3. Phase Contrast

The refractive index for X-rays deviates slightly from unity and an X-ray beam is modulated in its optical phase after passing through a sample or through different constituents inside the sample. A difference in phase can be induced between two parallel rays passing on each side of an interface between two constituents of the studied material. This phase difference induces a contrast, which is added to the classical attenuation contrast. A spatial redistribution of the photons occurs due to deflections (more generally due to Fresnel diffraction). This is normally considered as a nuisance in attenuation imaging. However, it can also make a phase imaging technique possible. This imaging mode is based on simple propagation.^[3] The occurrence of contrast can be understood as due to interference between parts of the wavefront that have suffered slightly different angular deviations. The overlap between parts of the wavefront is only possible after propagation over a certain distance. The spatial coherence of the X-ray beams delivered at ESRF ensures good contrast in the interference patterns.

Phase imaging is in general much more sensitive than absorption imaging. This is crucial when the attenuation contrast is too small, for instance to observe light materials, such as polymers, but also to distinguish absorbing constituents with similar X-ray attenuation.

The setup is the same as for attenuation microtomography. Phase sensitivity is obtained by increasing the sample-detector distance. Phase imaging can be used straightforwardly in a qualitative way, mainly useful for edge detection. It has also been shown^[4] to be an excellent way of improving the detection of cracks even in their early development stage (i.e., when they are not widely opened). The detection of isolated features smaller than the spatial resolution is possible thanks to the interference pattern they produce. A more quantitative approach, holotomography, has been implemented to extract the quantitative distribution of the phase in two-dimensional projection images, then to turn it into 3D reconstructions. The phase-retrieval procedure is based on a few radiographs, recorded at different distances with respect to the specimen, and can be combined with microtomography. It then allows to reconstruct at each point of the sample the decrement with respect to unity of the refractive index. This decrement is

essentially proportional to the electron or mass density of the material. This leads to a different source of contrast compared to the standard attenuation contrast and allows, for instance, to separate constituents with similar attenuation.

3. Selected results

3.1. Metallic Foams

The novel, low-density, metallic cellular materials, such as aluminum foams, show potential for use in light-weight structures. One of their interesting properties is the amount of energy absorbed during the deformation, which is directly related to the way the material collapses in compression. X-ray tomography has recently emerged as a powerful technique capable of giving a non-destructive picture of the interior of the structural materials including foams.^[5] We have used this technique to study the deformation modes of commercial foams during compression tests.

The setup used was the medium-resolution ($40 \mu\text{m}$) tomograph developed at the CNDRI laboratory. The sample was scanned for microtomographic analysis in its initial state. It was then compressed for two increasing values of remnant strain (true strain measured after unloading). The internal microstructure was imaged using microtomography at these two steps after removal of the compression load. We then have a picture of the interior of the sample at three values of the remnant true strain in compression: 0, 0.02, and 0.06.

A 3D view of the analyzed sample is shown in Figure 2. The cell walls are clearly imaged and the 3D structure can be analyzed. However, it is quite difficult to visualize the deformations with this kind of representation especially in the case of closed cells for which the outer walls hide the inner ones. The qualitative results can also be shown in the form of 2D reconstructed slices, which are easier to analyze. Figure 3 (a and b) shows a set of 2D images of the same zone of the sample numerically extracted from the volume at 0 to 6% of remnant strain respectively. These slices are parallel to the compression axis, which is vertical on the Figure. One can

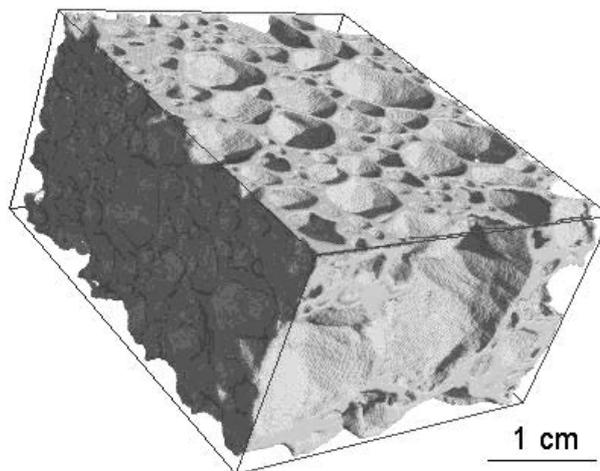


Fig 2. 3D representation of a metallic foam.

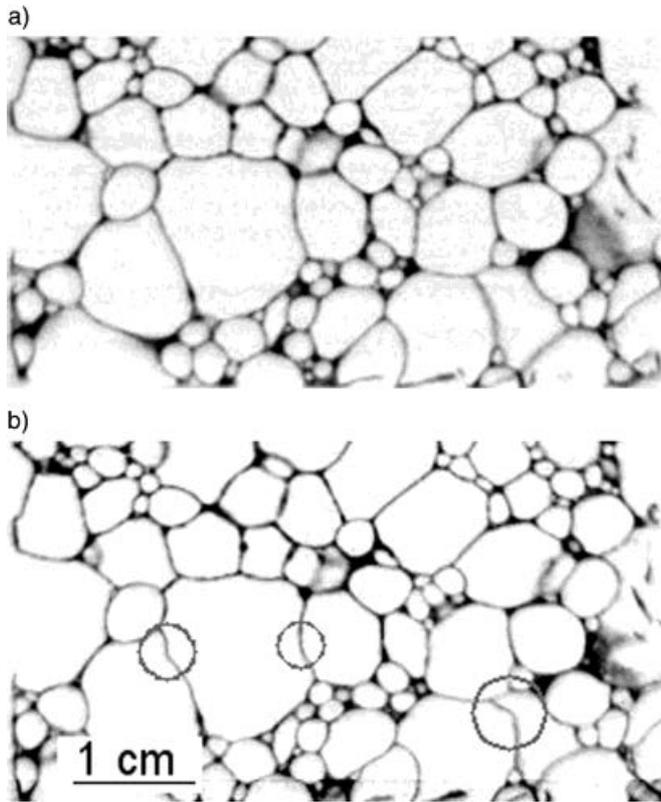


Fig 3. 2D reconstructed slice extracted from the set of tomographic results showing the evolution of the internal structure of a metallic foam during compression: a) initial state; b) for a plastic strain of 0.02 in compression

clearly observe that the deformation mechanism is due to the local buckling of several walls such as the ones surrounded by circles on the picture. The buckled walls are all located in a band perpendicular to the compression axis.

The tomographic images of such materials can be used to automatically generate meshes, which can be calculated by finite element codes. These are valuable tools to investigate the amount of stress distributed in each cell wall and help to explain the collapse of these materials in compression.^[5] However, the meshing method, which has been used so far in this direction, leads to very large models for which the calculations are then difficult to perform on standard computers. New efforts are now being carried out trying to reduce the size of the models of the actual microstructures using beam or shell elements.

3.2. Damage During in-situ Tensile Tests

A testing device has been especially designed^[4] to allow mechanical tests to be performed in situ during microtomography experiments. The device, mounted directly on the turntable of the synchrotron beamline or of the laboratory tomograph, includes a polymer tube, which transfers the forces from the top to the bottom grip. This tube gives negligible attenuation of the X-rays under each view angle and then allows a tomographic scan to be recorded under load.

This setup has been used to study the extension of damage in heterogeneous systems like SiC/Al metal matrix composites with a resolution of 6 μm . As mentioned in Section 2.3, phase contrast was of crucial help because SiC particles and Al matrix X-ray attenuation is nearly the same. The particles were distinguished in the reconstructed images by the detection of the edge of their interface with Al. Phase-contrast fringes also improved the detection of the particle cracks. This study has permitted to show that the evolution of damage in the bulk of the material, though qualitatively similar, was strongly different, in a quantitative way, to that at the surface. The amount of broken particles was nearly twice as big inside the sample as at the surface.^[4]

Another important study in the field of materials science concerns the growth of cavities in ductile matrices. A lot of analytical and numerical models have been proposed to theoretically describe this problem. However, such models usually describe local processes that are difficult to compare with actual experimental measurements. We produced model composites to study this problem with a good control of experimental and microstructural parameters. The chosen system was a pure Al matrix reinforced with 4% of ZrO_2 spherical particles.^[6] High-resolution (2 μm) tomography experiments were carried out on this system during tensile tests. It was observed that the fraction of particles de-bonded during the extrusion is relatively large in the bulk of the material (about 50%). The fraction of interfaces pre-damaged during extrusion is more important than the fraction of interfaces damaged during the tensile test. It is important to notice that primary surface scanning electron microscopy (SEM) observations of the sample showed much less pre-damaged interfaces, which underlines clearly that surface preparation can modify the microstructure of materials especially in the case of hard particles embedded in a soft matrix. The pores in the reconstructed blocks present a gray level, which is markedly different than that of the particles or of the matrix. By selecting this gray level the porosity in the studied 3D block could easily be visualized. The outer shape of the pores could be rendered in 3D by virtually removing the voxels v corresponding to the other phases. A visualization of the porosity detected using this technique is given in Figure 4. This 3D rendering, which corresponds to a state with an important plastic deformation, clearly shows the porosity growth and the coalescence that occur in the material. The amount of porosity as a function of the true strain could also be extracted easily from the processed 3D images. This study has been carried out both globally (i.e., on the total studied 3D images) and locally (i.e., on single voids near isolated particles). The initial global volume fraction of porosity was very low (about 0.08%) and for the sixth deformed state (the sample being already necked) it was found to be equal to 1.95%.

Comparisons have been carried out between the experimental value of the porosity measured with this technique and standard predictions of a Rice and Tracey type expression. These comparisons showed that the standard models give upper bounds for the prediction of local porosity but strongly overpredicts the value of the global porosity.

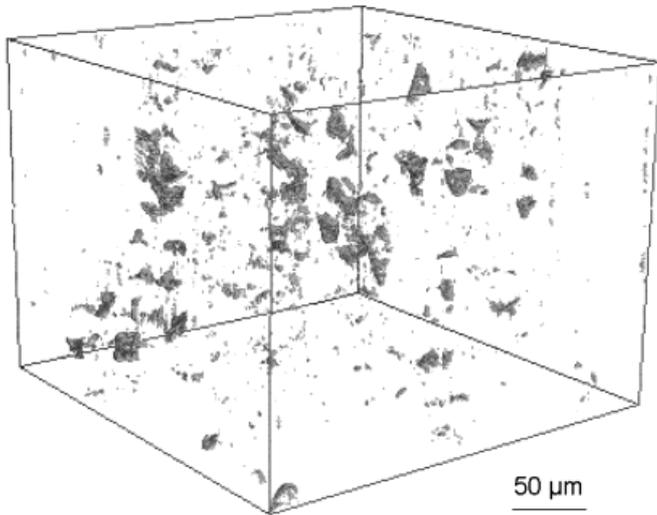


Fig. 4. 3D representation of the porosity in a model Al-ZrO₂ composite for a large value of the plastic strain.

3.3. Semi-solid Forming

Semi-solid forming is an alternative process to liquid, high-pressure die-casting in which a material in the semi-solid state (generally Al-Si alloys) is cast using an injection machine.^[7] Semi-solid forming requires a specific microstructure, which is often referred to as globular, in order to have good mold-filling and to produce sound parts without any liquid segregation. From a fundamental point of view, it is important to know the microstructural evolution, which takes place in the semi-solid state to reach this microstructure, and the rheology of such a solid-liquid mixture under shearing during the injection process. Both require microstructural characterization and it has been shown that 2D observations might not be sufficient to fully characterize these microstructures.^[8-10] Indeed, one of the main important features during forming is the connectivity of the solid phase, which is not easy to measure in 2D even if some attempts have been made using stereological relationships.^[11] The first 3D observations of semi-solid materials were made mainly on model alloys, using serial cuttings and 3D reconstruction, with a resolution of 10 micrometers at best. We have been using X-ray microtomography at ESRF to characterize the microstructure of industrial materials at a resolution of 2 μm. This characterization was performed after the material had been isothermally held in the semi-solid state and quenched to study the microstructural evolution, which took place during the treatment.

These experiments were carried out using quenched samples of A357 aluminum alloys at room temperature. In order to have a good contrast between the solid phase and the eutectic mixture (which was liquid in the semi-solid state) the holotomography mode described in Section 2.3 was used.^[12] A typical 3D representation is shown in Figure 5. It can be seen that the microstructure consists of globules of the solid phase and it is then possible to extract some interesting characteristics of this microstructure. Figure 6 shows the entrapped liquid in the microstructure of Figure 5, i.e., the liquid completely embedded in

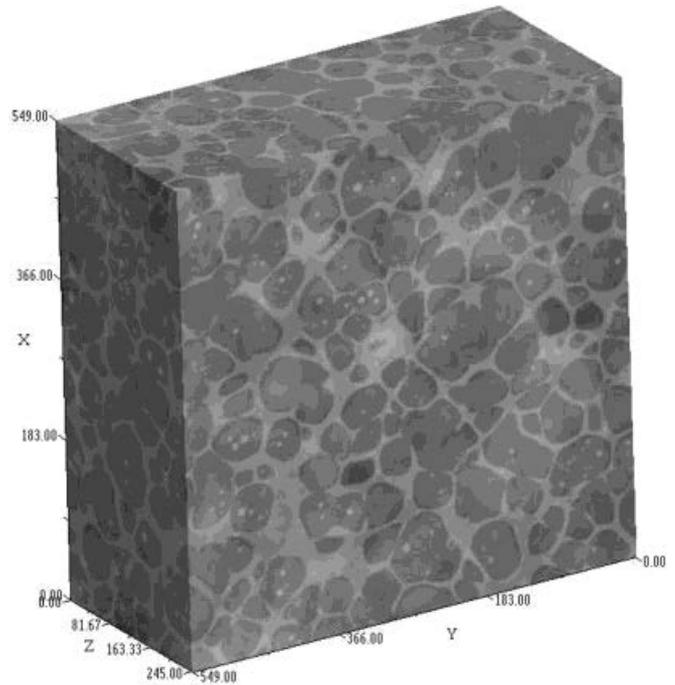


Fig 5. 3D representation of a A357 alloy, volume = 1mm × 1mm × 0.5 mm.

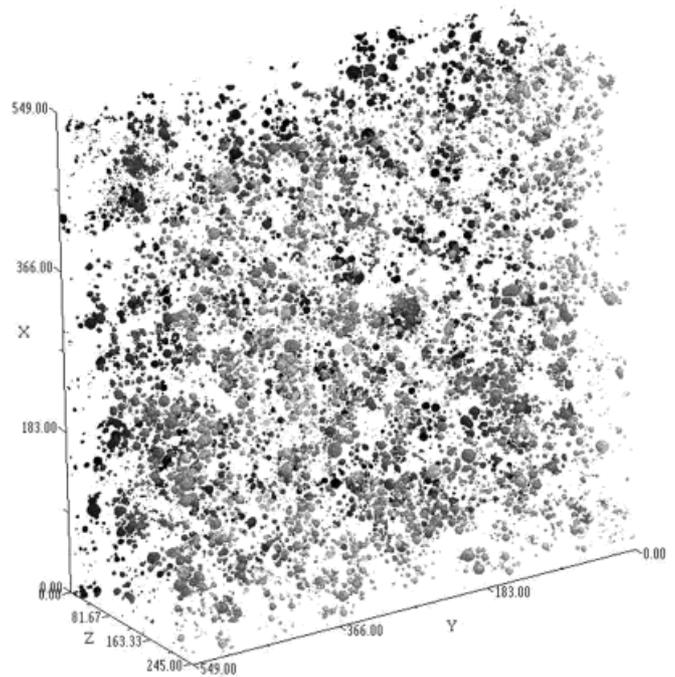


Fig 6. 3D representation of the entrapped liquid in the alloy of Fig. 5: volume = 1mm × 1mm × 0.5 mm.

the solid phase. This liquid is not so easy to characterize in 2D since it is hardly distinguishable from the interglobular liquid. Tomography allows to get the total liquid volume fraction, the volume distribution, the morphology of this entrapped liquid, and its 3D location. For this particular microstructure, the fraction of entrapped liquid is 0.8 %, the average volume of the liquid pockets is 800 μm³, and the average sphericity is 0.9, which means that the entrapped liquid pockets are nearly spherical.

3.4. Superplasticity

Superplasticity is frequently defined as the ability for a polycrystalline material, deformed at high temperature, to reach very high values of the elongation before rupture. It is associated to the predominance of grain-boundary sliding (GBS) as the main mechanism of deformation. Superplastic forming (SPF) concerns particularly titanium and aluminum alloys and major applications have been developed in the aeronautical industry. In the case of single-phase materials, such as Al alloys, superplastic deformation can induce severe damage by cavitation through the microstructure, leading to premature fracture but also to a reduction of service properties of the alloy after forming. Today, industrial SPF overcomes this difficulty by forming components under superimposed pressure. A better understanding of the fracture mechanisms during superplastic deformation is then needed to avoid the use of such a complex procedure.

The complex shape of the cavities formed during SPF makes the counting of these cavities difficult to carry out by standard metallography. Experiments were then performed at ESRF to assess the actual shape and number of the formed cavities. The beam energy was set to 17.5 keV to study an Al-Mg alloy, deformed in superplastic conditions ($T = 525^\circ\text{C}$) at different strains. An example of a 3D reconstructed image of the population of cavities through the alloy is shown in Figure 7. This image corresponds to an elongation of about 170% (strain rate of 10^{-4}s^{-1}), for which a mean cavity volume fraction close to 1% was measured. For this elongation, the majority of the cavities are isolated through the microstructure, although a little amount of connections between cavities could be detected. From such images, several parameters can be measured

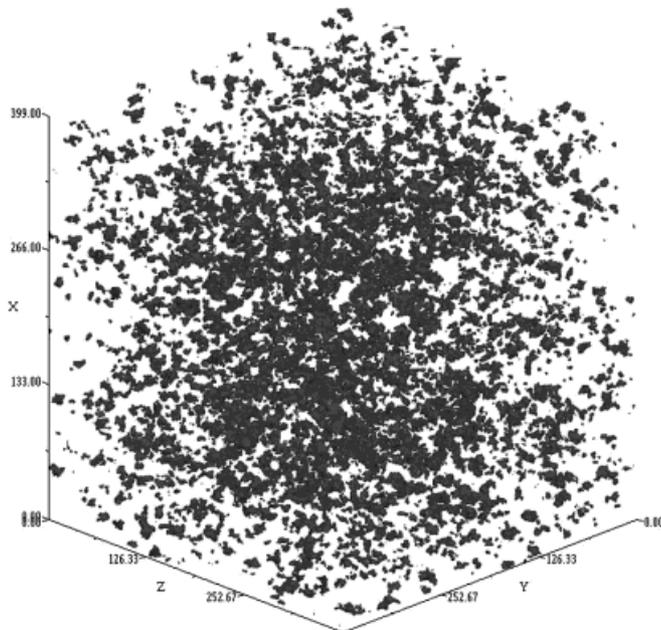


Fig 7. 3D reconstructed volume showing the cavities induced by superplastic deformation of an Al-Mg alloy (cavity volume fraction $\approx 1\%$).

directly, such as the cavity volume fraction C_V , the number of cavities n_V per mm^3 , or the volume V of each cavity. Data about the shape of the cavities can also be obtained.

Data about the damage process (i.e., nucleation or coalescence of the cavities) are frequently obtained from the variation of the number of cavities with strain. For the studied Al-Mg alloy, it was shown^[13] that the variation of n_A (number of cavities per unit area) with strain, deduced from conventional SEM observations, continuously increased. It was also shown that n_V (number of cavities per unit volume) deduced from microtomography measurements decreases when the strain is increased. To clarify this point, 2D sections were generated from the reconstructed volumes, since from each volume it is possible to choose a family of planes to reconstruct 2D sections. For each section, the apparent number of cavities per unit area could then be measured. These measurements confirmed the tendency observed by SEM investigations (increase of n_A with strain). Such differences in the variations of n_A and n_V with strain are attributed to the complex shapes of the cavities after large superplastic strains, due to the predominance of grain-boundary sliding. In consequence, some cavities, which appear apparently disconnected in section observations, are in fact connected through the volume. These results confirm the difficulty to directly interpret data deduced from 2D observations.

3.5. Grain-Boundary Imaging

If polycrystalline aluminum is brought into contact with liquid gallium (melting point 30°C) the aluminum grain boundaries are replaced by microscopic ($0.1\text{--}2\ \mu\text{m}$) liquid gallium layers. High-resolution X-ray microtomography allows to observe this phenomenon (grain-boundary wetting) in situ and in the bulk of coarse-grained Al polycrystals^[14]. Figure 8 shows a tomographic slice of a cylindrical sample of 99.996% Al after application of liquid gallium to the sample surface. One observes a network of wetted grain boundaries (white corresponds to higher absorption). Obviously there are also "missing" grain boundaries, which have not been wetted by liquid gallium. A more detailed analysis, using a new 3D X-ray diffraction technique^[15] reveals that these boundaries correspond to low-angle grain boundaries.

The described wetting phenomenon can be used to visualize and analyze the three-dimensional grain structure in the bulk of polycrystalline Al alloys: in this case one intentionally uses liquid Ga as a contrast agent to reveal the position of the grain boundaries in the bulk of the material. Using this technique (grain-boundary imaging) it was possible, for instance, to analyze the propagation of short fatigue cracks in cast Al alloys^[16] and their interaction with microstructural features (i.e., grain boundaries, inclusions, ...). Combined with a 3D X-ray diffraction technique this methods opens interesting new possibilities for the quantitative analysis of fatigue mechanisms in bulk material.

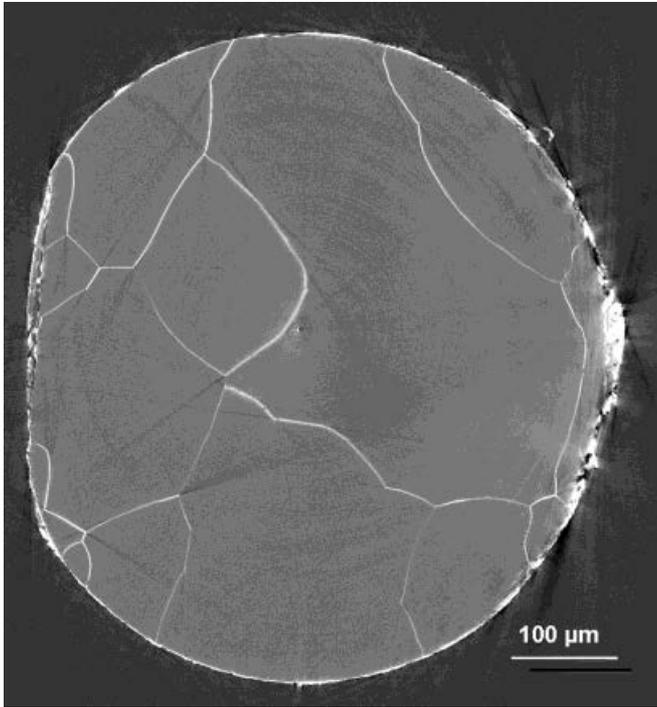


Fig 8. 2D reconstructed slice showing the grains structure of pure aluminum wetted with gallium.

4. Concluding remarks

The first part of this paper describes the microtomography technique and the specifications for medium and high resolution, which are required for materials science applications. The microtomography technique has been used to investigate the microstructure and damage mechanisms in several materials for which key materials science problems are still to be solved. The studied systems were composed of very different materials, such as a metallic foam, particular metal matrix composites, and alloys. In each case, microtomography permitted us to visualize clearly the initial microstructure at a suitable resolution and when coupled with in-situ tensile or compression experiments, it gave important information about the internal structure and its evolution during the deformation of the studied samples. Tomography allows a better description of the shape of the different features of the microstructure (inclusions, pores, and grains). It also shows quantitative differences between what can be derived from surface observations and what is actually happening in the bulk of the studied materials. This experimental breakthrough should lead to a real improvement in the modeling capabilities applied to this class of materials. This paper also shows that different laboratories, all members of the FR MAT SPU, are involved in the recent development of this technique in collaboration with X-ray specialists and taking the opportunity of using the exceptional ESRF X-ray source. This has

brought the "Région Rhône-Alpes" to a leading position in the field of three dimensional images applied to materials science.

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