Number 1

Psiché

Solidification of an Al-Cu Alloy (4 w% Cu) using X-ray Microtomography

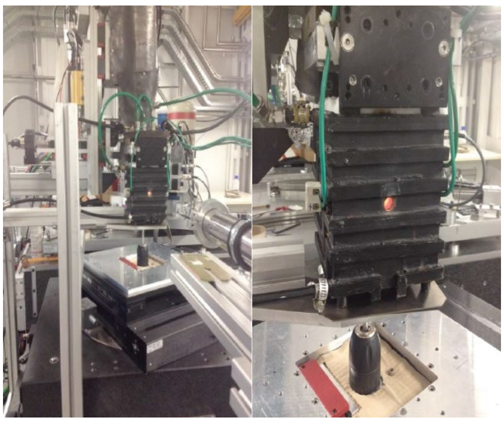
When it comes to high performance materials for industry (e.g. automotive, aeronautics, petrochemical), 3D visualization of the internal microstructure during manufacture or service proves to be an essential tool in the characterization of complex phenomena such as crystallization, phase transitions, microcracking, stress corrosion, and high temperature creep.

Challenge:

Improvements in the quality of X-ray beams, detectors and reconstruction algorithms, as well as the development of *in situ* sample environments enable the study of the micro-structure of materials subjected to extreme and evolving conditions (e.g. mechanical, thermal). This is performed non destructively using *in situ* microtomography with high-speed data acquisition. The observation and quantification of dendrite growth during the solidification of an Al-Cu alloy carried out in this study highlights the great potential of technique to address a wide range of industrial issues.

SOLEIL's solution:

The PSICHE beamline, dedicated to X-ray microtomography imaging and high-pressure X-ray diffraction experiments, offers two modes: monochromatic (a single narrow bandwidth beam energy), providing maximum contrast or "pink" beam (with wide spectral bandwidth) for high-speed data acquisition (~1-10 seconds per scan). Equipped with a large hollow rotation stage allowing users to install sample environment equipment inside the stage (e.g. furnaces, compression cells, tensile testing machine) for *in situ* and real-time measurements of samples. The beamline also allows for further increased tomography data acquisition speed (< 1 second per scan) as well as variants in which tomography is combined with diffraction.



Experimental set-up: integration of the furnace* (operating in the 300-1500°C temperature range) on beamline PSICHE

High-speed, high-temperature *in situ* data acquisition using X-ray microtomography on PSICHE.

Solidification experiments have been carried out on a cylindrical sample (2.6 mm diameter) of an Aluminium-Copper alloy (~4% Cu) encased in a hollow alumina rod which was fixed to the tomography stage (for rotation and translation) and placed in a furnace allowing the passage of X-rays. After reaching its melting point (~660°C), the sample is slowly cooled at 5°C/min, down to 580°C. A series of tomograms were recorded, each lasting 5 s and containings 900 projections collected using a sCMOS Hamamatsu camera (FOV~2.6 mm x 2.6 mm) in pink beam mode every 30 s (30 scans in total), with a 1.3 µm pixel size.

Results obtained:

Dendrite coalescence appears to be the dominant mechanism for dendrite growth (figure 1). In contrast with the works of Limodin et al. 2009[1], the scientists did not observe any small dendrite arms re-merging for the benefit of larger arms by Ostwald mechanism, which is probably related to higher cooling speed. Special attention was given to the processing of tomographic data in order to extract local speeds in 3D for dendrite tips (Vtip). In fact, for a given undercooling, dendrite arms are expected to grow according to product Vtip x Rtip = constant (Gibbs et al., Sci Rep 2015[2]; Daudin et al., Acta Mat 2016[3]). Figure 2 shows that this relation seems to be verified in this experiment.

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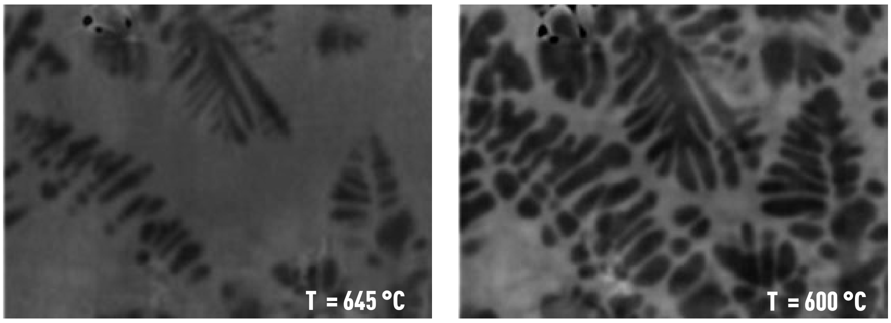


Fig. 1 : 2D cross-sectional views extracted from volumes illustrating dendritic growth evolution in the course of solidification. Primary and secondary branches can be distinguished, surrounded by solute enriched liquid. The dendrite arms grow as solidification progresses.

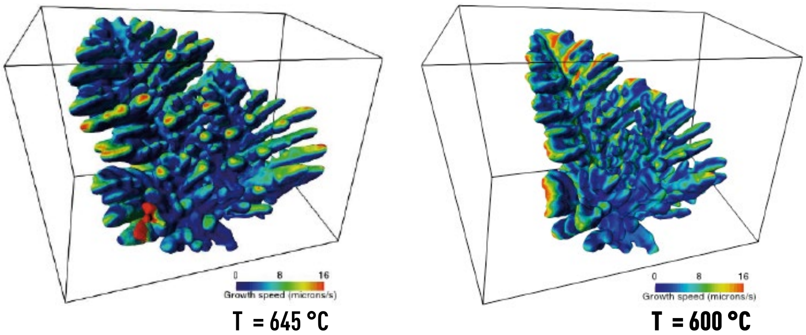


Fig. 2a : representation of the local speed determined from 3D surface meshes of solid-liquid interface in the course of solidification (local Euclidean distance between surfaces calculated and divided by the elapsed time between scans).

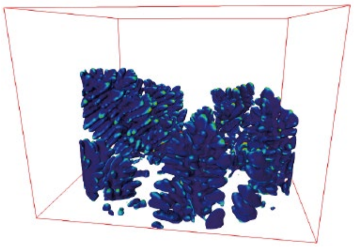
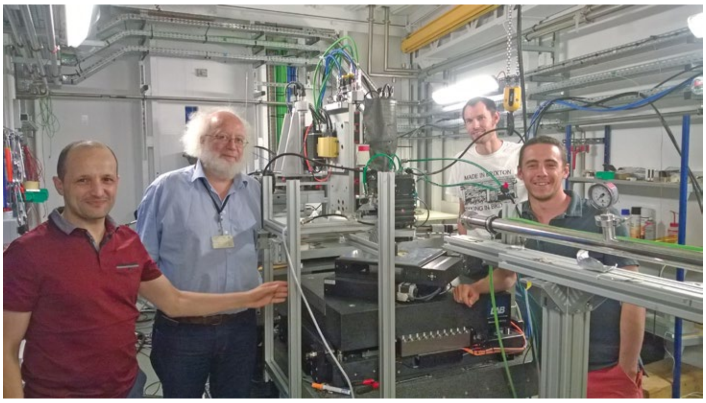


Fig.2b : product of local speed by local curvature at the tip of dendrites.

References: 1. Limodin et al., Acta Mat, 57:2300-2310 (2009) - 2. Gibbs et al., Sci Rep, 5 :11824 (2015) - 3. Daudin et al., Acta Mat (2016, Accepted)



Loïc COURTOIS and Kamel MADI (3Dmagination) with Jean Paul ITIE and Andrew KING (SOLEIL's PSICHE beamline)

3Dmagination, a young start-up located on the Harwell Oxford Campus, has built upon leading-edge developments in 3D imaging, with the purpose of training the engineers of the future and developing innovative solutions for its clients from a variety of sectors (e.g. biomedical, energy, automotive).

"Thanks to our industrial and academic partnerships and to more than twelve years of experience in imaging, material mechanics and processes, we have developed a true know-how in algorithm development and in the extraction of reliable and relevant scientific information from tomographic data, while constantly striving to optimize the entire data acquisition chain and to enrich the dialogue between experiments and simulations. Currently, our clients are really eager to study the evolution of a material structure in real time under extreme conditions (e.g. mechanical, thermal). Such information is essential to understand—and therefore better control—the manufacture and operational behavior of industrial products. Spatial and temporal resolutions prove to be decisive, and our laboratory equipments do not yet allow us to capture all the dynamics of microstructure formation in real time.

3Dmagination
"Imaging beyond imagination"

The PSICHE beamline at SOLEIL offers ultimate performances (coherence, very high X-ray flux) and an energy range that is intermediate between Diamond and ESRF—other synchrotrons with which we also collaborate. The duration of the experiments is significantly reduced (~a few seconds with a spatial resolution of the order of a few microns), which opens up vast application perspectives for our clients, for example for the study of phase transitions in thermal barriers, damage and rupture mechanisms in steels, and crystallization in building materials."

"A strategic location"

"Thanks to its strategic geographical location, our close relationship with SOLEIL constitutes a major asset for our company, as our customers are located on the Franco-British axis. Furthermore, a team of experts is constantly attentive to our expectations in order to meet our clients' demands. They are also always eager to develop new experiments by means of an evolving and adaptable beamline offering great flexibility for the integration of new equipments. Our privileged relationship with SOLEIL and the Centre des Matériaux of MINES Paris Tech will lead us to push back the boundaries of traditional 3D imaging in favor of a new kind of multimodal imaging in which the PSICHE beamline achieves particularly high performances, and in which we will combine high resolution tomography and X-ray diffraction in situ."