EUROPEAN SYNCHROTRON RADIATION FACILITY

ESRF User Office
BP 220, F-38043 GRENOBLE CEDEX, France
Delivery address: 6 rue Jules Horowitz, 38043 GRENOBLE, France
Tel: +33 (0)4 7688 2552; fax: +33 (0)4 7688 2020; email: useroff@esrf.fr; web: http://www.esrf.fr

ESRF

Report on ME-1065

Forewords

This document is a report of the experiments carried out at ESRF from May 11 to May 17 on ID19 within ME-1065. Since it is uploaded on September 6, 2005, it only corresponds to a partial analysis of the data acquired. It is yet proposed to give an overview to the reviewers who will analyze a new proposal submitted on September 1st, 2005.

AMPL Report

The iron content in recycled aluminum alloys present as aluminum-iron intermetallics may lead to damage during the forming operations of automotive alloys. These intermetallics form during the solidification of the alloy and are not affected by subsequent heat treatment operations. One objective of this study is to visualize the structure of an Al-0.6, 1.9 and 8 wt% Fe alloys solidified under conditions that simulate the cooling rates in strip casting. Also to visualize the structure of spray deposited Al-0.6wt%Fe strip.

The images generated at ESRF are viewed using *Amira*, *VolView*, *ImageJ* and *VGExpress* in a 64 bit Opteron PC under Linux operating system. Because of the large size of these files, we were unable to import them completely into the PC. In collaboration with the University computer service group, a script was generated to sample a RAW file for either a portion of the image or for a lower resolution of the entire domain. A 550 μm particle and a strip sample of 0.6 wt% Fe that was atomized in a nitrogen atmosphere was studied.

We have visualized the porosity in the strip (See Figure 1). The fissure is due to the uneven motion of the strip during the deposition process but the other porosity is from solidification shrinkage. Previous efforts to measure and characterize porosity of these strip samples using 2D imaging and Archemedes Principle did not reveal this type of porosity information.

For the 550 μm powder particle, we have identified a single nucleation point for the droplet. Its position is very near the periphery of the surface of the droplet (see Figure 2). Also clearly visible are the primary dendrites growing from the region of nucleation and initial growth (point A in Figure 2). We are presently attempting to image this primary region by itself. In addition, we have observed two regions of high concentration of eutectic in the particle. We have determined that these regions which are evidence of diffusive macrosegregation during solidification are also associated with the last liquid to solidify. This was determined by imaging both these regions of high eutectic concentration and the regions of porosity. The 3D rendering of these images were superimposed as seen in Figure 3 and were observed to be coincident. It was also determined that the prosity in these regions are

interconnected. Further investigation of these samples is ongoing to quantify these observations.

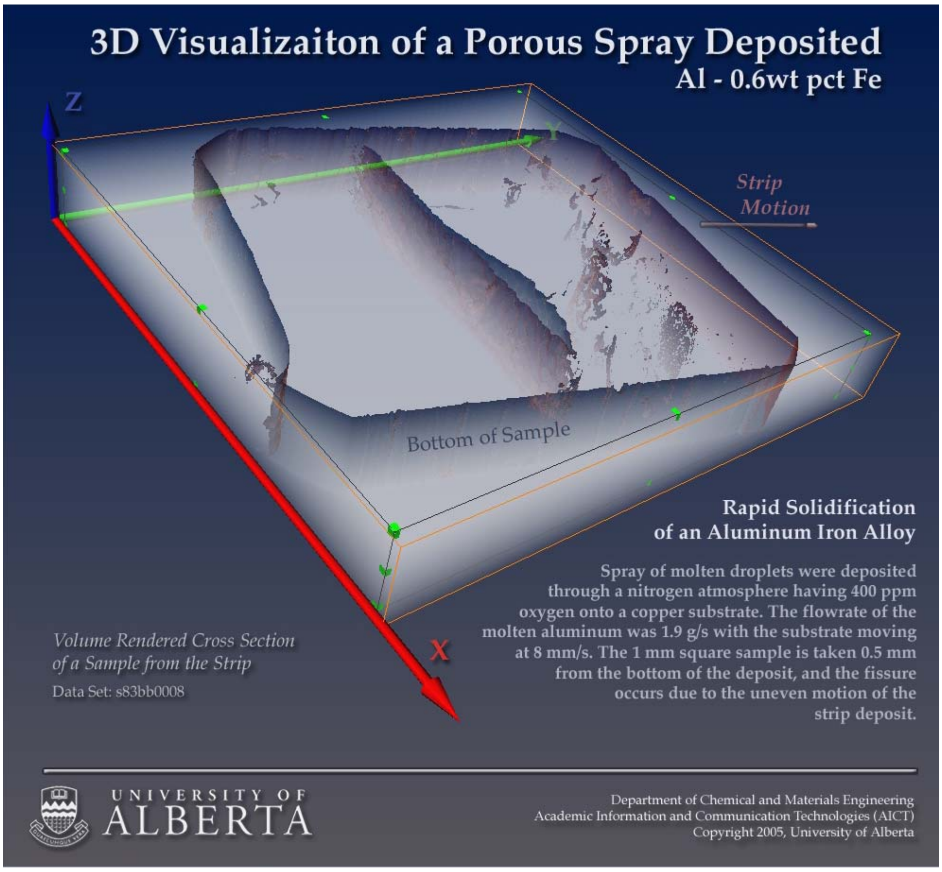

Figure 1: 3D visualization of Al-0.6 wt% Fe strip deposited using Impulse Atomization.

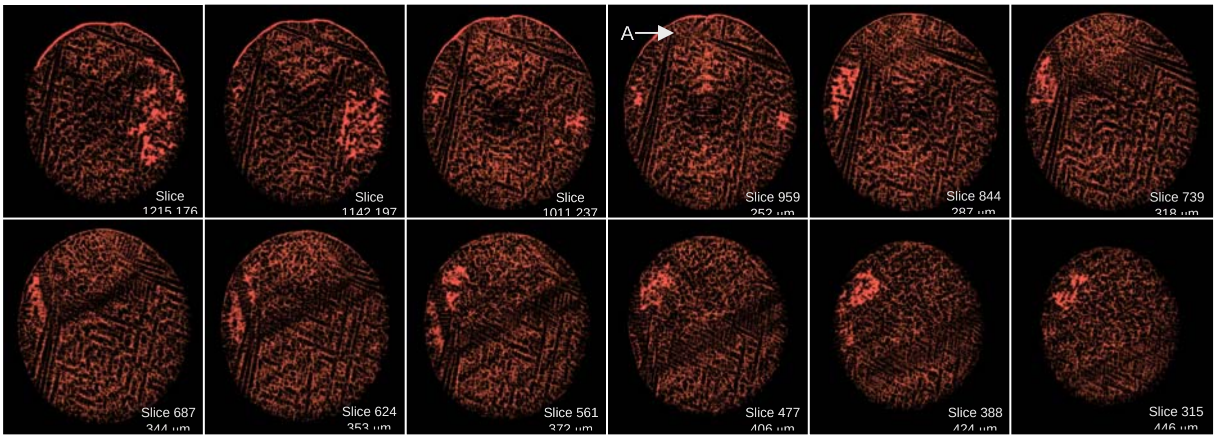

Figure 2: Sequence of images from a 550 µm particle of Al-0.6 wt% Fe showing the nucleation region at point A. The primary growth dendrites are also evident from the nucleation point.

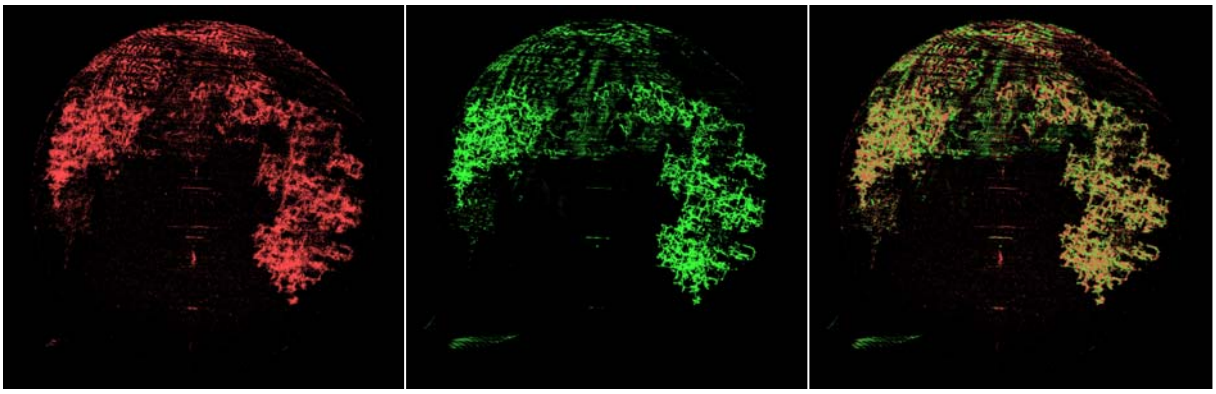

Figure 3: Full volumes of a 550 μm Al-0.6 wt% Fe particle highlighting the highest concentration of eutectic, porosity and a superimposition of eutectic and porosity. This clearly shows that macro-segregation is evident in the particle.

The results of the 550μm Al- 0.6wt% Fe particle was presented at the Conference of Metallurgists in Calgary, Alberta on August 21 to 24, 2005. The poster entitled "Visualization of a Rapidly Solidified Al - 0.6 wt% Fe particle using Micro-Tomography" was co-authored by *C. Watt*, *H. Henein,* of Dept of Chemical and Materials Engineering, University of Alberta, Edmonton, AB, CANADA; *M. Kazakevich2, J. Johansson* of AICT Research Computing Support, University of Alberta, Edmonton, AB, CANADA*; Ch.-A. Gandin* of Ecole de Mines de Paris a Sophia-Antipolis, France and 4. INSA de Lyon, France; *and E. Maire* of INSA de Lyon, Lyon, France. The student C. Watt received the third place award in the poster competition.

CEMEF Report

The purpose of the tomography investigations performed at ESRF was to determine the effect of spontaneous vs. triggered nucleation on the solidification of Al-Cu droplets. This study is in collaboration with DLR (German Aerospace Centre, Köln, Germany) who prepared the samples by electromagnetic levitation.

The three dimensional images obtained at ESRF are loaded and manipulated using the image processing program *Image J*. This program is used to calculate the volume fraction of eutectic averaged over cubic volumes of size 50x50x50 μm^3. Figures 4, 6, and 8 are two dimensional slices at the lower mid-radius, centre and upper mid-radius for the centre piece of the droplet containing 4wt% copper. Figures 5, 7, and 9 display the corresponding distributions of eutectic. Areas of the micrograph with coarse microstructure correspond with regions of lower eutectic fraction. The point of triggered nucleation is apparent in the fine microstructure in Figure 4 compared to the increasingly coarser microstructures in Figures 6 and 8.

The maps of the eutectic volume fraction derived from images obtained at ESRF will be compared to the eutectic volume fraction and copper composition maps acquired from SEM. The distribution maps of eutectic fraction derived from both ESRF and SEM will be compared to the theoretical upper limit determined by Scheil. Furthermore, the copper composition map and both the distribution of eutectic fraction maps will be analyzed in order to verify that regions with high volume fraction of eutectic correspond with regions of high copper content. Indeed, according to the results obtained so far, there is an evident correlation between regions of high copper content and corresponding areas of high fraction of eutectic.

These maps and images provide the ability to visualize the effects of solidification phenomena such as macro segregation, recalescence, and directional vs. equiaxed grain growth, as well as dendrite arm spacing.

Current results obtained from the 4% Al-Cu samples show a distinct difference in both the distribution of copper composition and eutectic volume fraction between the samples that were spontaneously nucleated vs. triggered. The microstructure corresponding to triggered nucleation demonstrates directional growth around the nucleation point. According to the distribution maps, the copper content and fraction of eutectic in this directional growth region are visibly more evenly distributed than in the surrounding areas. This may be due to the high growth rate incited by the significant heat flow from the solidifying droplet to the trigger. In contrast, the microstructure for the spontaneously nucleated sample demonstrates no such directional growth. However, both the triggered and spontaneously nucleated samples show regions containing high copper percent and high eutectic fraction around the periphery indicating macro segregation possibly due to solute diffusion and/or bulk flow.

Finally, further calculation of copper composition and volume fraction of eutectic will be performed using SEM on the samples containing 14 and 24wt% copper. These will be compared to the eutectic fraction maps calculated from images also obtained at ESRF. The maps for 4, 14 and 24wt% copper will be analyzed in order to determine the effect of melt concentration on the previously observed solidification phenomena.

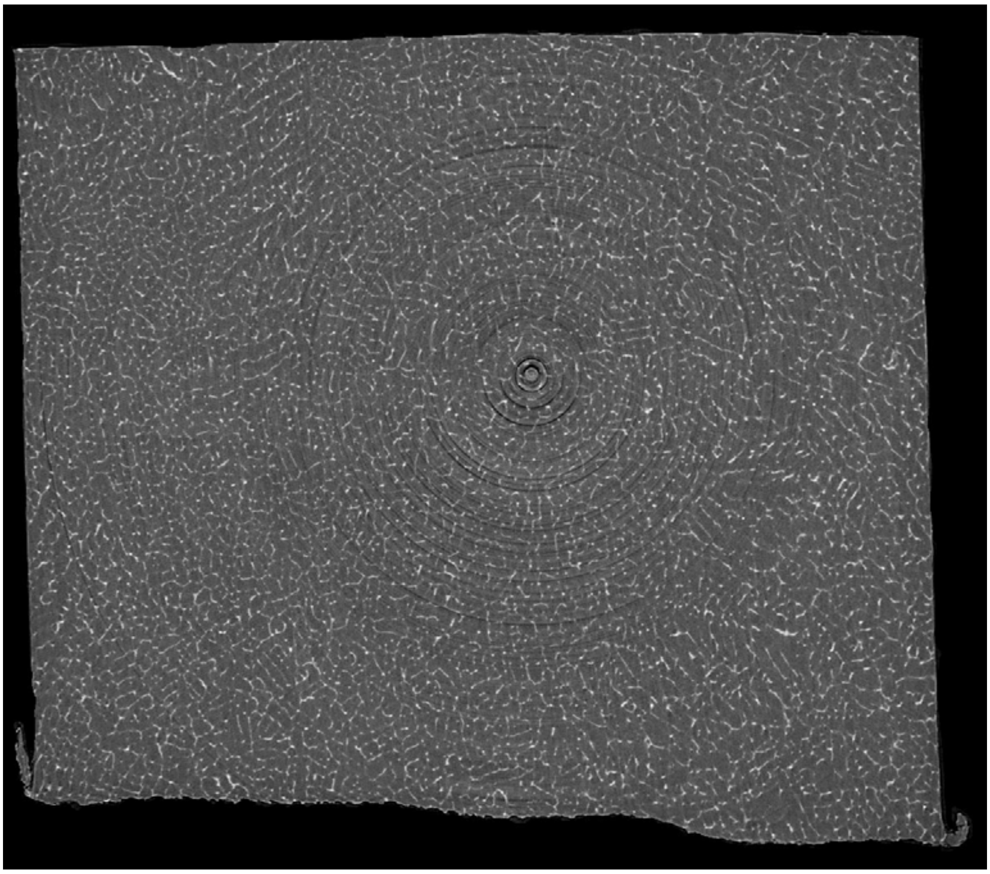

Figure 4. Original image at the lower mid-radius of the middle piece of the 4%Al-Cu droplet.

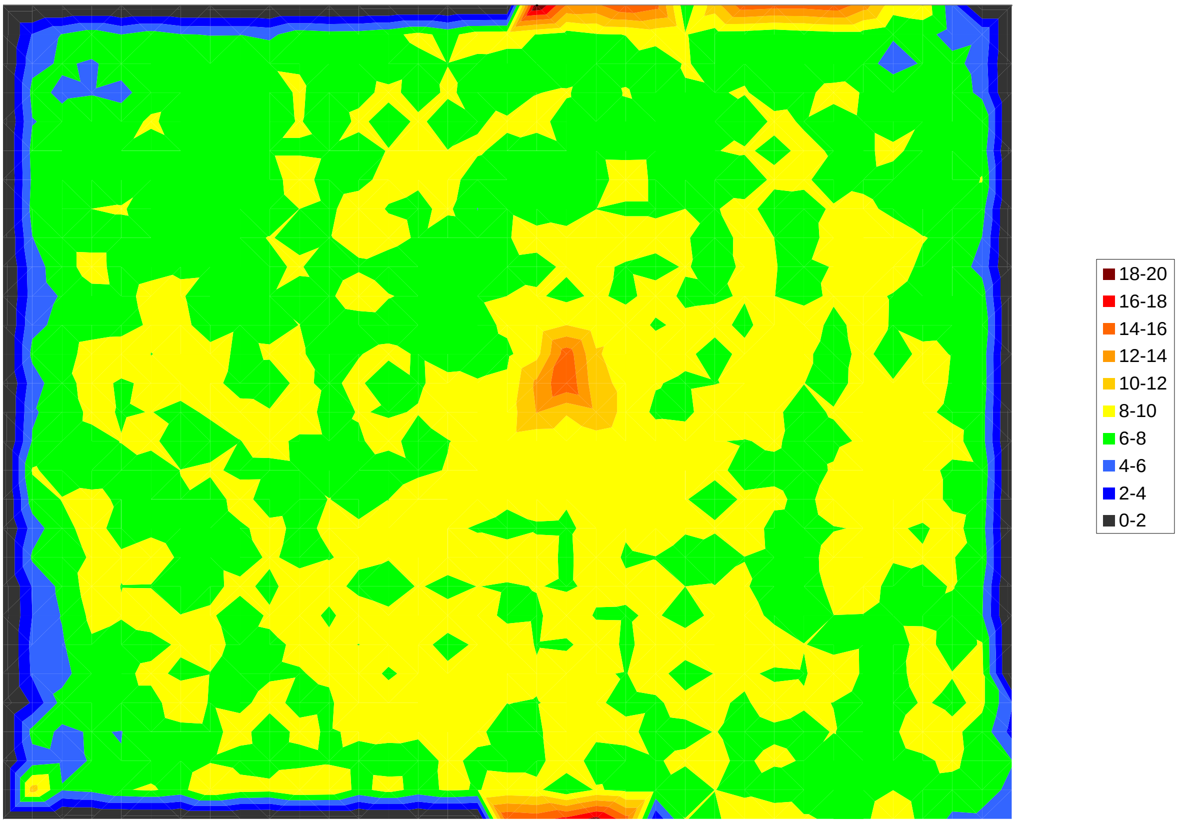

Figure 5. Map of the eutectic volume fraction averaged in 50x50x50μm^3 zones at the lower mid-radius.

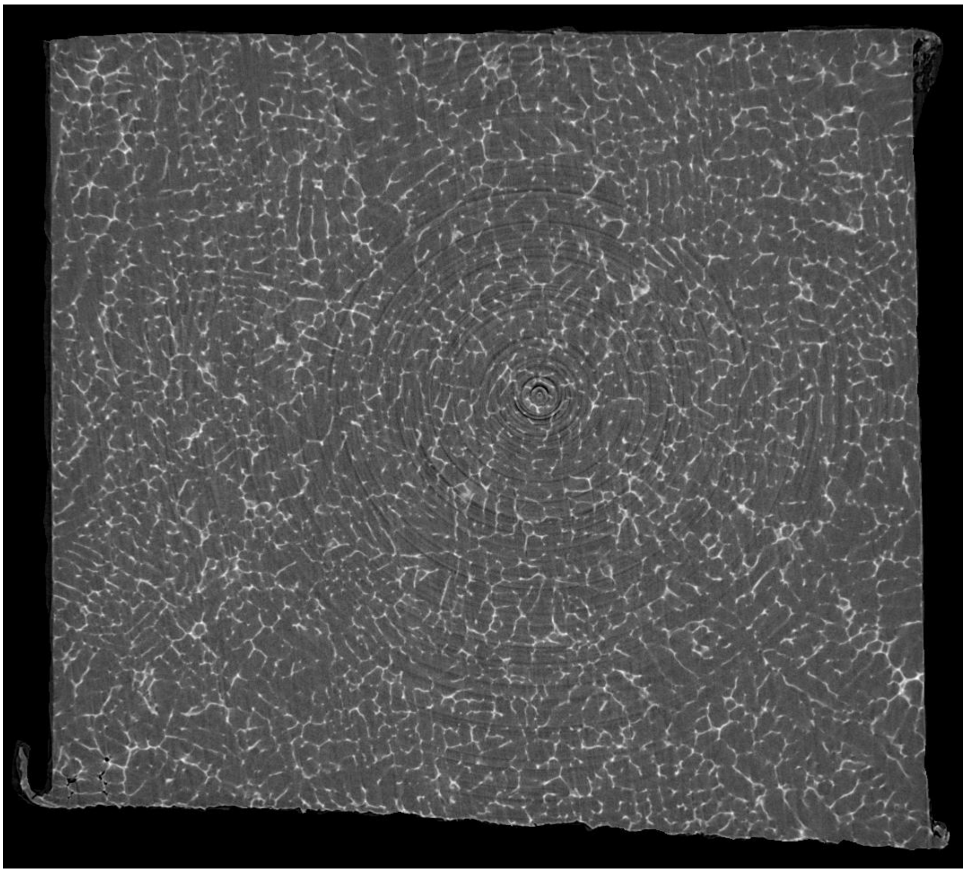

Figure 6. Original image at the centre of the middle piece of the 4%Al-Cu droplet.

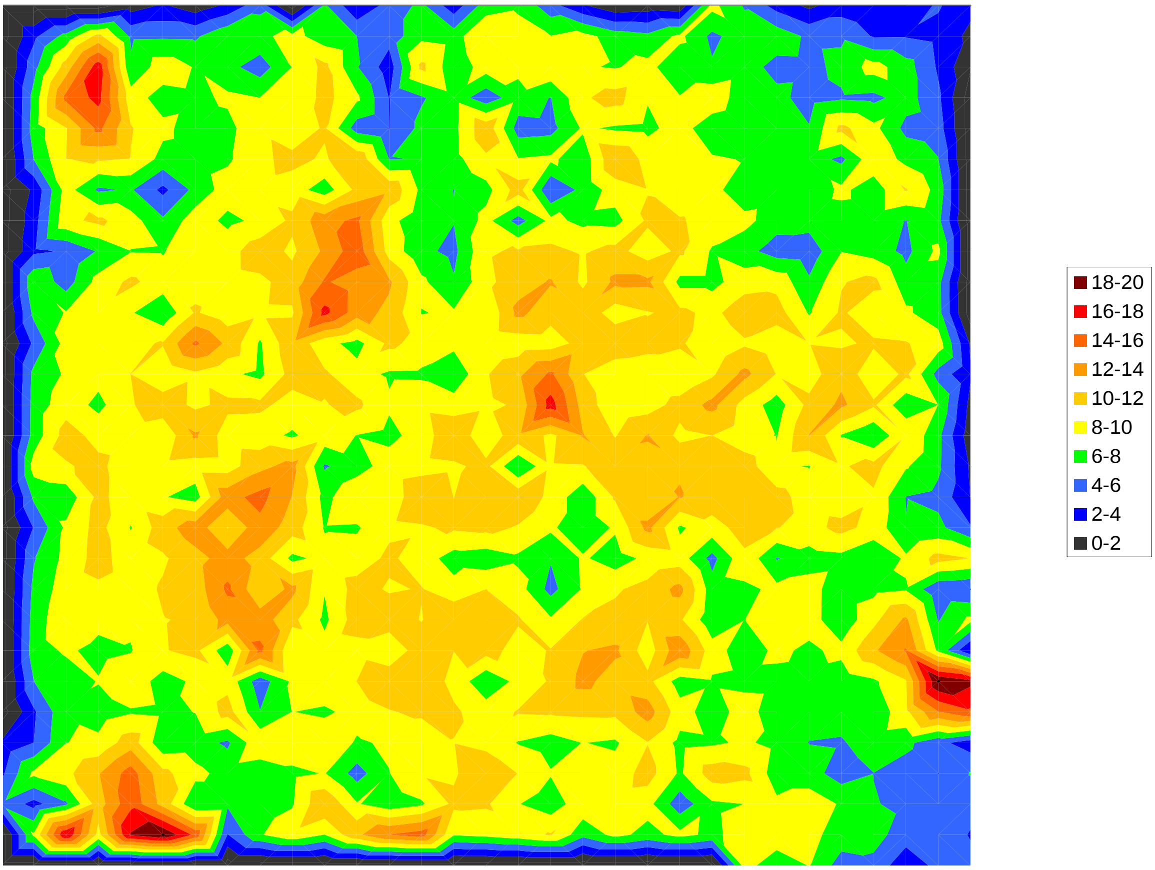

Figure 7. Map of the eutectic volume fraction averaged in 50x50x50µm^3 zones at the centre.

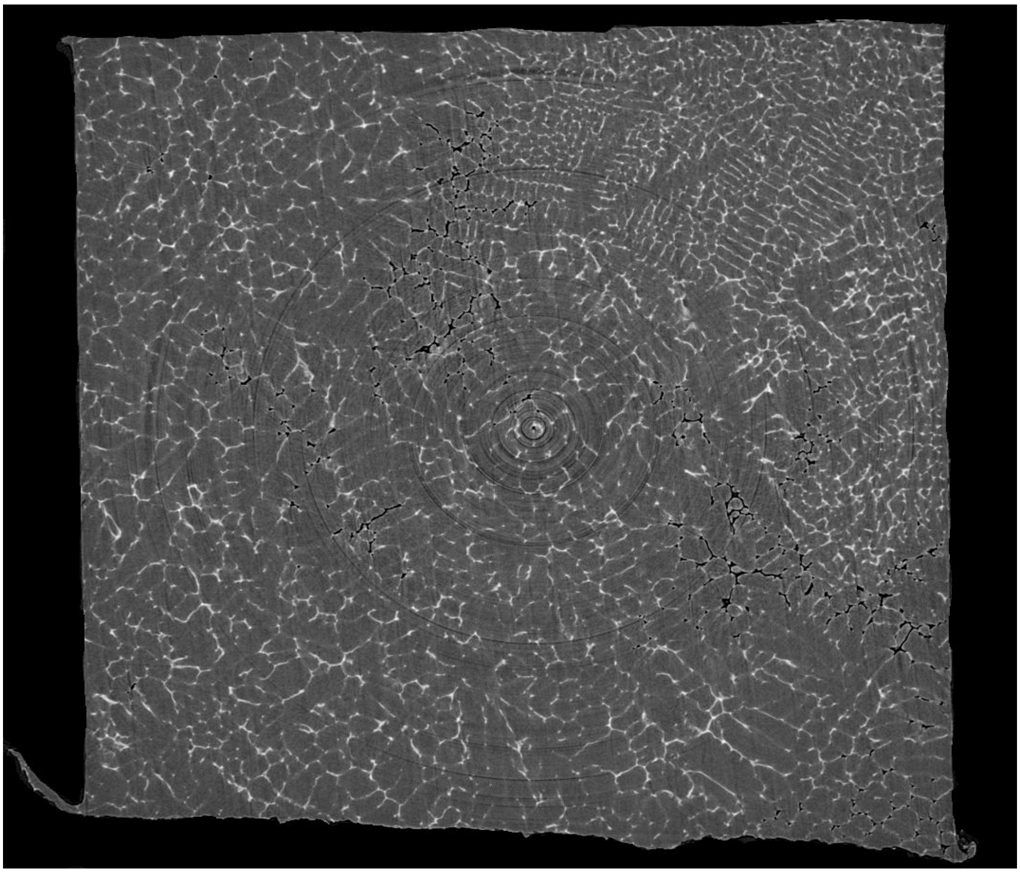

Figure 8. Original image at the upper mid-radius of the middle piece of the 4% Al-Cu droplet.

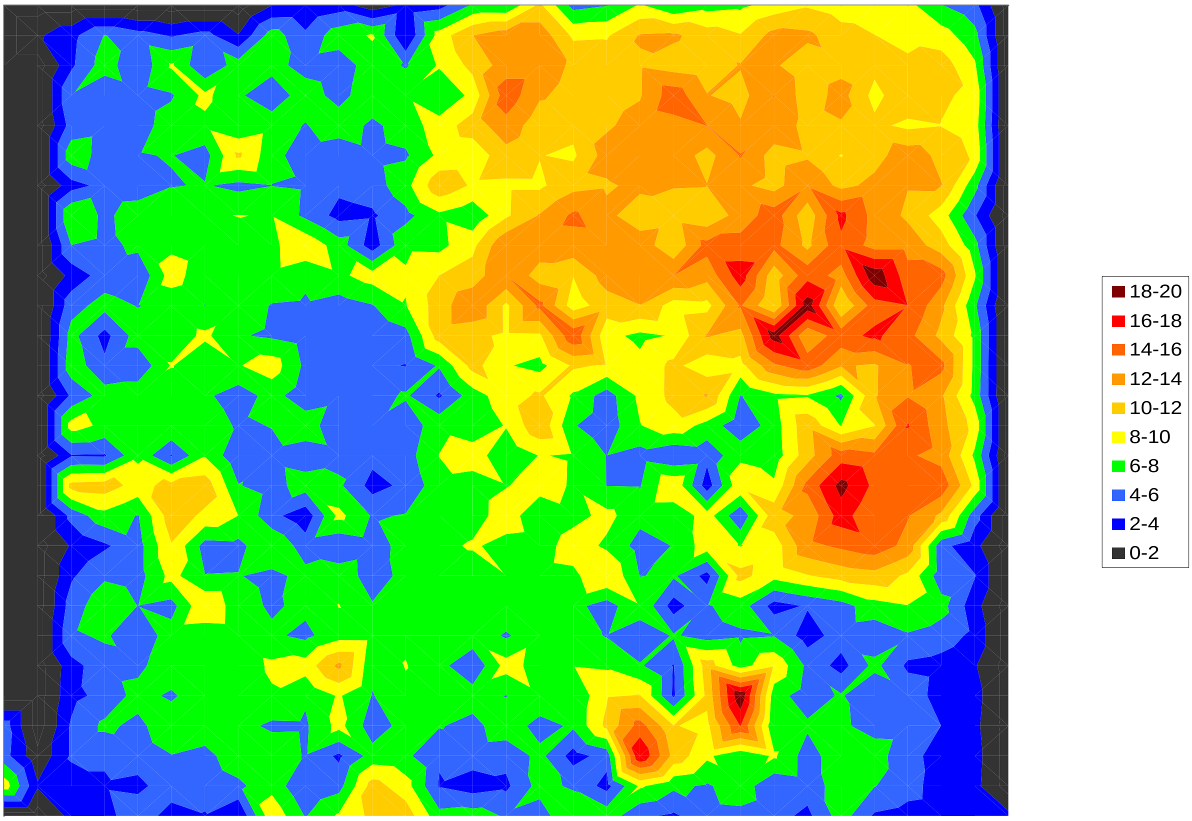

Figure 9. Map of the eutectic volume fraction averaged in 50x50x50μm^3 zones at the upper mid-radius.