	Experiment title: Morphology and crystalline texture of magnetic arborescences grown by Electrodeposition under magnetic	Experiment number: HE 1066
Beamline:ID22	Date of experiment: from: 18 April 01 to: 22 April 01 and 20 June 01 to: 22 June 01	Date of report: 03 March 02
Shifts: 18	Local contact(s): Dr. Timm Weitkamp and Dr. Michael Drakopoulos	*Received at ESRF:*

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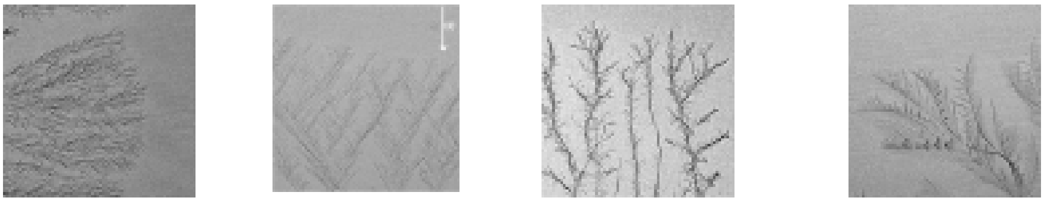
Report:

The aim of the experiment: on ID22 was to study by micro-imaging and micro-diffraction the morphology and the crystal texture of magnetic arborescences grown by electrodeposition.

We are interested in pattern formation in the electrochemical deposition of magnetic metals, Fe and Co, from thin layers of $Fe(SO_4)$ and $Co(SO_4)$ aqueous solutions under an applied magnetic field. Arborescent aggregates are obtained, and the magnetic field may have a strong effect on the morphology. For instance a spectacular in-plane magnetic field effect is observed on the growth of Fe aggregates: the macroscopic morphology changes from circular in zero field to rectangular in finite field, one edge of the rectangle being parallel to the field. The growth is dendritic and in zero field there is no correlation in branch orientations at long distance, while under in-plane field the branches grow along two well defined directions with respect to either the field direction or its perpendicular. Ascribed to magnetic dipolar effects, this selection of branch orientations explains the observed field-induced macroscopic morphology symmetry breaking: branches growing along two well defined directions at the same speed give a straight front. As suggested by the growth in zero field the angle itself between two branches appears however to be of crystalline origin. TEM experiments revealed pure Fe and Co single crystalline dendrites, as long as 2µm with side branches as small as 50 nm, emphasising the fractal character of the arborescences since similar dendrites are observed from the µm to the mm scale range. As to which scale the single crystalline state extend is unknown and an answer was expected from x-ray microdiffraction with resolution down to 2µm.

The μ**imaging experiment** was performed using the FReLoN2000 Fast-Readout Low-Noise CCD camera, allowing spatial resolution down to the µm scale with a field of view up to 1mm^2 . Fig. 1 shows examples of the obtained images. Compared to optical images, owing to the fact that X-rays images result from a combination of absorption and phase contrast, much more accurate details of the morphology are observed. A better insight of the growth process was provided thanks to the quality of the images and to the temporal resolution (about one image per second) which allowed useful dynamic in-situ observations.



a **b** **c** **d**

Figure 1. Growths of Fe 0.2m/l obtained without magnetic field (a), under in plane magnetic field (b) and under perpendicular magnetic field (b), growth of Zn 0.1 m/l under perpendicular magnetic field (d).

The μ**diffraction experiment** was performed using a monochromatic beam of 25 KeV (λ=0.49Å). The sample situated at 89mm from the camera was set perpendicular to the beam.

In the case of Fe arborescences, no diffraction pattern could be observed. It is not clear if that is due to a failure to find the right crystal orientation, a poor crystallisation or some unknown reasons.

In the case of Zn, large dendrites (>100 µm) may be obtained depending on external parameters. Such dendrite was studied, using a monochromatic beam of spatial extension about 4µmx10µm. Examples of the diffraction patterns recorded are shown in fig 2. Several spots at a same Bragg angle are observed indicating that each branch is probably made of several elongated crystals. On scanning the dendrite, according to the rectangle shown fig.2 (size: 100µm x 40µm), structural relationships on large spatial scale are evidenced between the branches. A number of structural defects are found in the central nerve.

In conclusion the results obtain on Zn dendrites are encouraging, but microdiffraction with a monochromatic beam seems not to be the right tool for such a study. Since large dendrites are not simple single crystals, and no signal was observed on Fe dendrites, Laüe experiment using a white beam, combined with topography in monochromatic beam, seems to be more appropriate. Beam time is requested on ID19 to perform such an experiment.

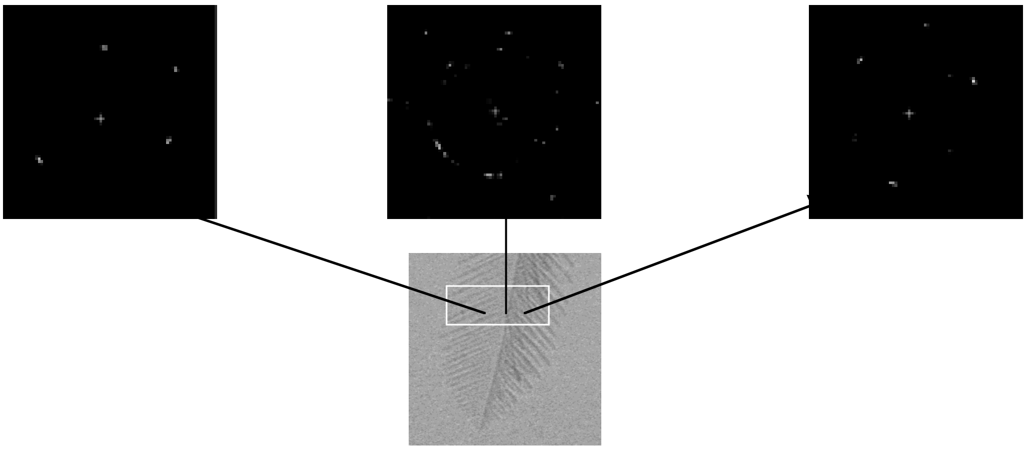


Figure 2: examples of diffraction patterns obtained on different parts of a Zn dendrite