| ESRF | **Experiment title:**<br>**Examination of the hardening eta prime phase in Al-Zn-Mg alloys** | **Experiment number**:<br>ME-861 |
|---|---|---|
| **Beamline**:<br>BM01A | **Date of experiment**:<br>from: 03-DEC-04 to: 07-DEC-04 | **Date of report**:<br>28.02.06 |
| **Shifts:**<br>12 | **Local contact(s)**:<br>Mogens Christensen | *Received at ESRF:* |


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**Report:**

The project aimed at investigating structural details associated with an improtant hardening mechanism, so called *precipitation hardening,* in (among others) Aluminum-alloys. The final state is a dispersion of precipiates with characteristic dimensions of some nanometers, coherent to the Aluminium-matrix. These particles (with their associated strain field) then interact with dislocations causing the movements of the latter to be impeded. Percursors for the precipitates are local structures (fluctuations, defects and clusters) in the disorderes solid solution, and partially ordered Guinier-Preston zones, which are recognized as intermediate structures according to the precipitation sequence:

$$\text{Supersaturated solid solution} \rightarrow \text{GP-zones} \rightarrow \eta\text{'-precipitates} \rightarrow \eta\ (\text{eq})$$

The relative amount of phases, forming the above scheme, that is present in a particular grain of the alloy's microstructure, depends on the prior heat treatment. The objective of the present project was to obtain quantitative information about the diffracted intensities of the η'-precipitates in the ternary Al-Mg-Zn system, in order to determine its average structure. Prior to the present investigation, the use of various electron diffraction techniques has been dominating the research on this alloy-system.

Single crystal grains were prepared from a cast polycrystalline sample by using liquid Gallium to wet the grain boundaries. Approximately 15 samples of different size were obtained in this manner. Some samples were tested using the KM6-CCD detector, but owing

to the strong Al-matrix reflections, it was not possible to measure the weak precipitate scattering without introducing serious artifacts in the images. It turned out however that the MAR-image plate was well suited for this type of measurements.
Full datasets were recorded (360° ϕ-rotation, 720 images) on two different samples at $\lambda$ = 0.80 Å, resolution 1.0 Å.

The subsequent analysis yielded a multitude of diffracted intensities best presented in the reconstructed images, and showed the very interesting complementarity between synchrotron radiation and electron diffraction for these kinds of problems. It turned out that the measured samples did contain a significant amount of the equilibrium phase ($\eta$-$MgZn_2$) in addition to the metastable η'. Because of a preferred orientation relationship among both the η' (4 orientations) and the η-precipitates (11 orientations), a symmetry analysis had to be carried out in order to assess internal overlap beteween reflections prior to the extraction of the intesities, cf. Fig. 1. This analysis rests, to a certain extent, on the deduced orientation matrix of the *Aluminum-matrix*. Having this, it is possible (by applying the known orientation relationships) to obtain transformed orientation matrices associated with the different oriented precipitates. In this respect, for future experiments, it will be important to first measure an attenuated sub-dataset containing non-saturating Al-matrix reflections alone, in order to obtain this as accurate as possible.
Taking the overlaps into account, we have been able to extract a reduced three dimensional data set of 80 averaged reflections originating from the η'-precipitates. A further challenge accounted is associated with the intensity distrubutions of equvalent reflections, cf. Fig. 2. It seems evident that the η'-precipitates also are in a state of "continuous transformation", thereby diffracting with different intensities (this point will be subject to further inverstigations). We therefore, in the first approach, only aim for a model representing the average structure.
Several strategies have been devised in order to solve the structre (obtain a structural model). These are related to direct methods, Patterson synthesis, comparison to high resolution transmission electronmicroscopy images (HRTEM) of the precipitates, other electron diffraction techniques etc. This final analysis is not fully concluded *p.t.*, but direct methods indicate a stable solution yielding an R-value of approximately 0.27. A structural model will thus be suggested and information about occupancy and thermal behaviour may be estimated as well.

In conclusion: In addition to obtaining a new structural model for the η'-precipitates, the use of synchrotron radiation has "opened" a very interesting niche, complementary to electron diffraction techniques, in the study of these kinds of problems.

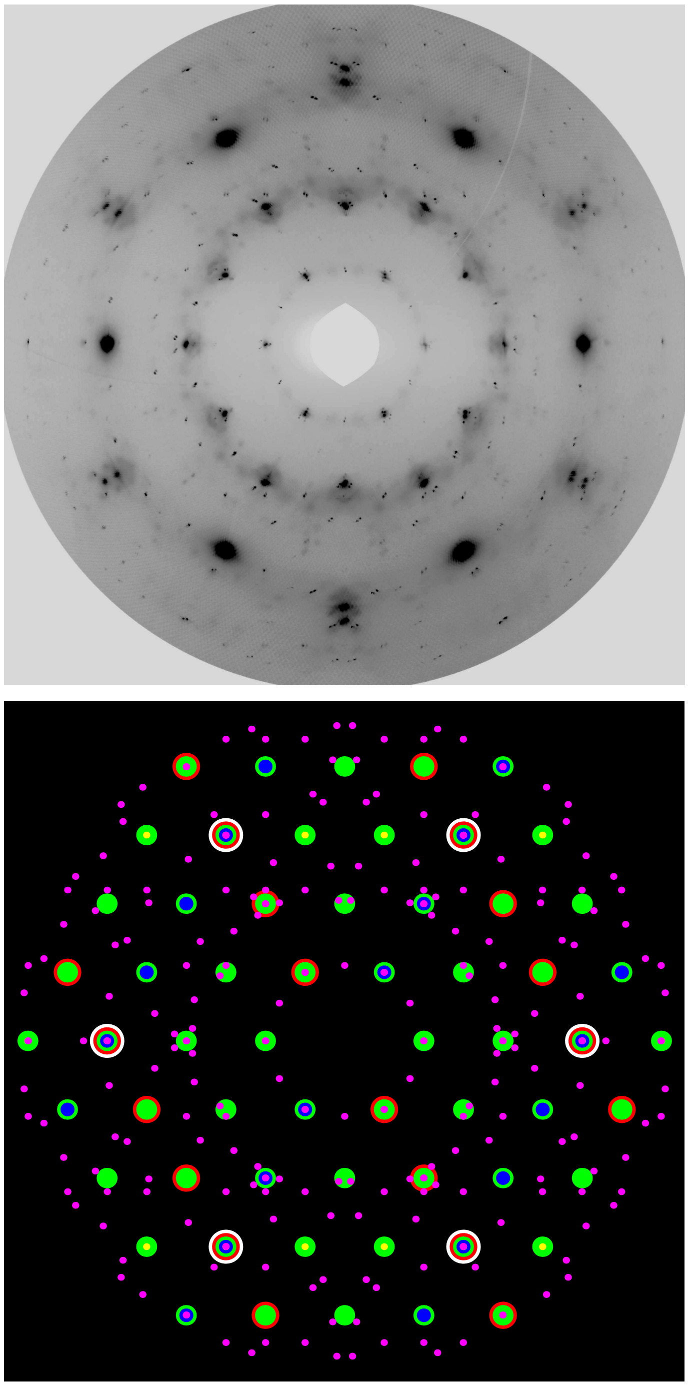

Figure 1: Reconstructed image of the reciprocal lattice plane $hk0$ together with a simulated picture showing the contributions from the aluminum lattice (white) , the $\eta'$ lattices (green, red, yellow and blue) and the $\eta$-lattices (magenta).

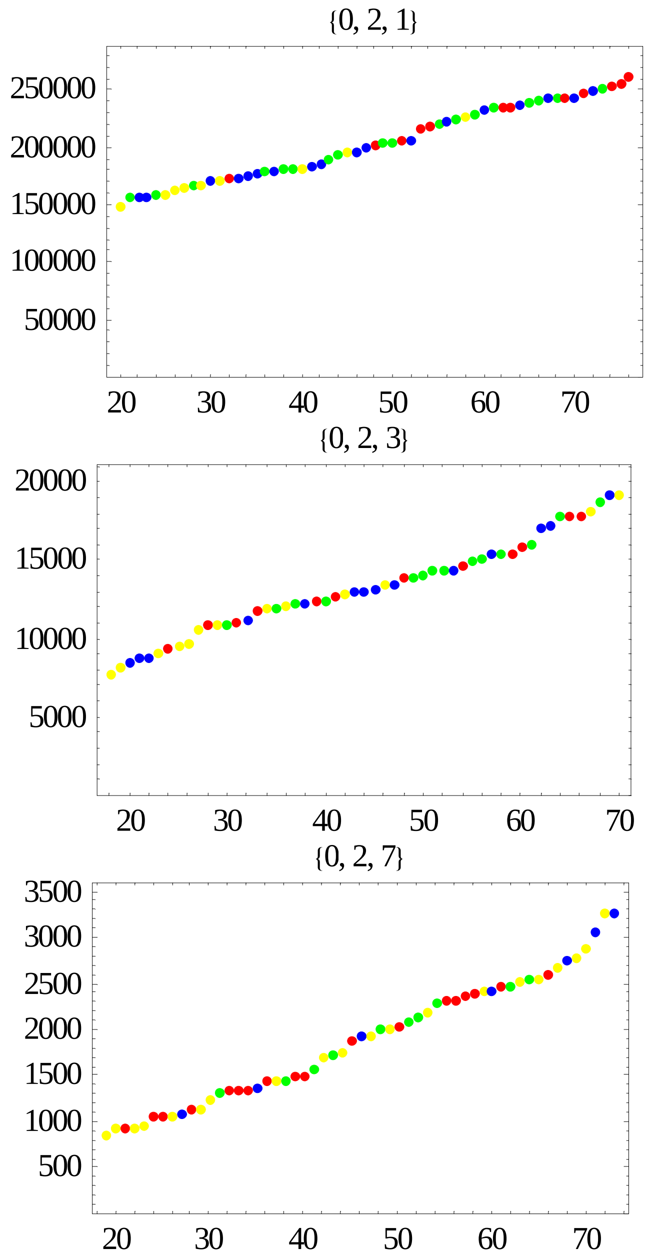


Figure 2. Intensity variations among the symmetry equivalents (Laue class -31m) for three different reflections from the η’-precipitates showing typical range in measured values.