| ESRF | **Experiment title:** Characterisation of the microstructure evolution in Al-Zn-Mg welds using small angle scattering | **Experiment number**: ME 6 |
|---|---|---|
| **Beamline**: BM02 | **Date of experiment**: from: 08.06.2000 to: 13.06.2000 | **Date of report**: |
| **Shifts:** 12 | **Local contact(s)**: Dr. Frédéric Livet | *Received at ESRF:* |

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## Report:

The aim of the experiment ME-6 on the D2AM beamline (BM02) was to use small angle X-ray scattering in order to give some insights on the precipitation microstructure resulting from a welding process in terms of volume fraction and size on an Al-Zn-Mg alloy used in automotive applications. Welding treatment results in coarsening and dissolution of precipitates in the heat-affected zone (HAZ) which induces a severe loss of strength. A good knowledge of the precipitation processes and of their kinetics will be very useful to control the mechanical behaviour of welded joints.

### Experimental results

The strengthening of Al-Zn-Mg alloys is due to a complex sequence of precipitation:

$$\text{Solid solution} \rightarrow \text{GP} \rightarrow \eta' \rightarrow \eta,$$

where GP zones and η' are metastable phases and η is the stable $MgZn_2$ phase. Consequently two types of experiments have been used to cover a wide range of scattering angles corresponding to the expected precipitate sizes (from a few Å for GP zones to about 1000 Å for the stable coarse phases present in the HAZ). The first one was a usual set-up with a range of scattering vectors between $2.10^{-2}$ and 0.5 $Å^{-1}$ and the second one was a very small angle set-up where scattering vectors down to $10^{-3}$ $Å^{-1}$ have been recorded.

Three initial states of the material were investigated: T4 (containing GP zones), T6 (containing η') and T7 (containing η).

- Scaning of the HAZ of several welds prepared by instrumented welding from T4, T6 and T7 initial states of the material. The high spatial resolution of the ESRF beam allowed us to determine quantitatively the precipitate size and the integrated intensity directly associated to the volume fraction in each point of the HAZ (up to 80mm from the weld) with a sub-millimetre precision. More than 100 points per weld have been recorded and interpreted.
- In-situ heat treatments of two types were performed using a furnace specially designed for this experiment: isothermal treatments and continuous heating ramps with heating rates up to 200°C/min. Thanks to the high flux available at ESRF, good counting statistics with a few seconds acquisitions allowed us to study the high temperature non-stationary precipitation and dissolution kinetics in these alloys, which is a necessary step in better understanding the results obtained from the actual welds through the development of physical metallurgy models.

## Results

Fruitful results about the precipitation state in welds were obtained from scans of the T4, T6 and T7 initial materials. As it can be seen on figure 1, the width of the HAZ in the T7 material (also observed in the T6 one) is well marked (here about 30mm) and corresponds to hardness measurements. The material is characterised by an unaffected zone far from the weld and a completely dissolved one separated by a transition zone where coarsening occurred. In the completely dissolved zone, precipitation of GP zones with a Guinier radius of about 4 Å occurred during holding at room temperature.

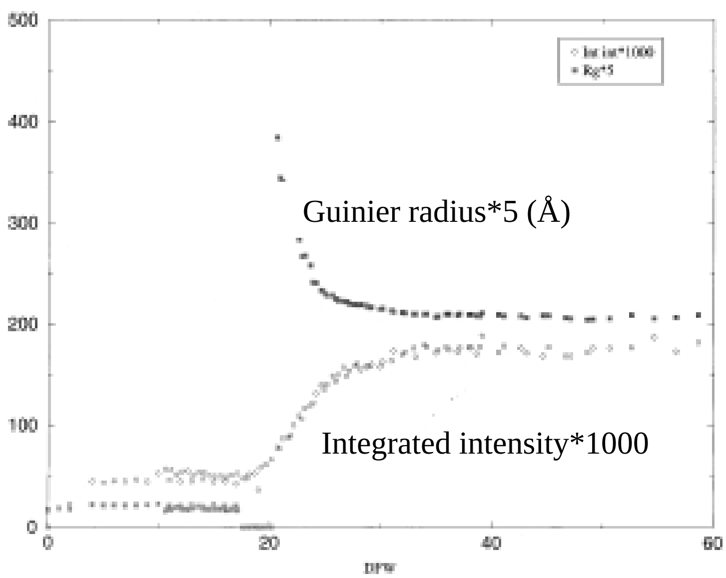


Figure 1: Integrated intensity and Guinier radius (in Å) versus distance from the weld (DFW in mm) obtained by scanning a T7 initial material.

At this time, only the integrated intensity and not the volume fraction is presented because of the uncertainty about the composition of unstable phases, in particular the transition phase η'.

In-situ heat treatments gave also interesting results about the kinetics of precipitation. On figure 2, the isothermal treatment at 200 °C on a T6 material is presented, which is characterised first by a decrease of the integrated intensity, due to be the dissolution of η' present in the initial state and then by the growth of a second phase, assumed to be the equilibrium η-phase. Work is under progress to determine the nature of the phases by transmission electron microscopy.

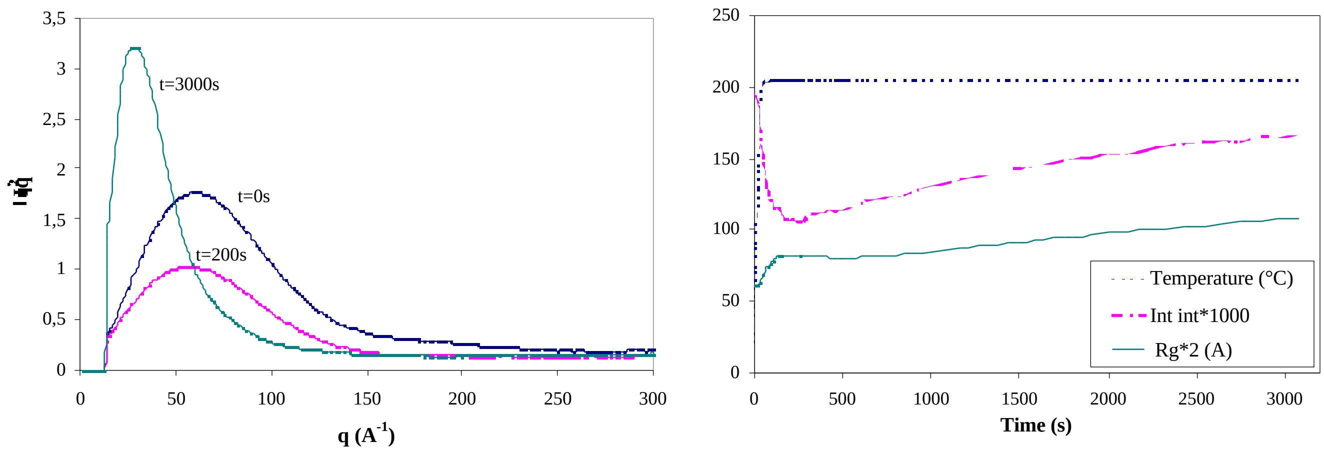


Figure 2: a. Evolution of $Iq^2=f(q)$ with time at 200°C of an initial T6 material.
b. Integrated intensity and Guinier radius versus time at 200°C of an initial T6 material.

## Conclusions

This experiment gives us fruitful information about the precipitation state in the weld materials and about the precipitation kinetics involved during rapid heat treatments. This microstructural information is of fundamental importance for the modelling of microstructure evolution in welded structures.

We present here a summary of the experiments:

| SCANS | | | |
|---|---|---|---|
| T4 – 80 mm from the weld | | | |
| T6 – 60 mm from the weld | | | |
| T7 – 60 mm from the weld | | | |
| **IN-SITU, Isothermal Treatments** | | | |
| T4 – 100°C | T4 – 150°C | | |
| T6 – 200°C | T6 – 250°C | T6 – 300°C | T6 – 350°C |
| T7 – 200°C | T7 – 250°C | T7 – 300°C | T7 – 350°C |
| **IN-SITU, Continuous Heating Ramps** | | | |
| T4 – 5°C/min | T4 – 20°C/min | T4 – 200°C/min | |
| T6 – 5°C/min | T6 – 20°C/min | T6 – 200°C/min | T6 – 200°C/min + cooling |
| T7 – 5°C/min | T7 – 20°C/min | T7 – 200°C/min | T7 – 200°C/min + cooling |

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|---|---|
| | Experiments using small angle scattering |
| | Experiments using both small angle scattering and very small angle scattering |
| | Experiments using very small angle scattering |