Investigation of Precipitation Growth in Aluminium-Silver Alloys via in-situ SAXS

Johanna Akbarzadeh[1], **Herwig Peterlik**[1]
[1] Dynamics of Condensed Systems, *Faculty of Physics, University of Vienna, Austria*

Introduction

Alloys have a big area of applications in engineering where they are mostly exposed to extreme conditions. Therefore, the knowledge about the influence of different parameters (e.g. high temperature exposition, external forces...) on their structure and later on their mechanical properties is of great importance for their production. In-situ small-angle x-ray scattering (SAXS) measurements offer the possibility to study the structural behaviour of materials under various conditions.

The first part of this work deals with the temperature dependence of the growth of the precipitations. Therefore in-situ SAXS measurements were carried out at different temperatures on two alloys: Al2at.%Ag and Al6at.%Ag. Changes in radii and the mean distance between the precipitations were, firstly, determined by "Guinier-fits" and afterwards by using the "Hard-Sphere-Model" [1] [2]. The second part of this work deals with the precipitations' shape modification in dependence on external load [3].

Basic principles

The process of precipitation requires:

- The material is an alloy
- Limited solubility of at least one component in the solid state
- Solubility has to decrease with decreasing temperature

Factors controlling the shapes of precipitates:

- The adopted shape is determined by the balance between interfacial Helmholtz energy and elastic Helmholtz energy
- Hier L is the ratio of elastic to interfacial energy
- When the particles are small, interfacial energy is the dominant factor in setting the particle shape and as L increases, the effect of elastic stress becomes important [4]

$$L = \frac{\delta^2}{C_{44}\sigma_{\alpha\beta}} \frac{3V^{1/3}}{4\pi} \qquad \delta = 2\frac{a_\alpha - a_\beta}{a_\alpha + a_\beta} \qquad a_\alpha, a_\beta \text{ ... latice parameter}$$

δ ... misfit factor $\quad \sigma_{\alpha\beta}$ ...interfacial energy $\quad C_{44}$ ... elastic constant

Sample preparation and Experimental set-up

- Production of Al-2at.%Ag and Al-6at.%Ag alloys in an induction furnace
- Rolling the samples to an x-ray transparent thickness. (45 µm - 60 µm)
- Homogenization at 500°C for several hours
- Quenching in oil instead of water in order to prevent surface oxidation

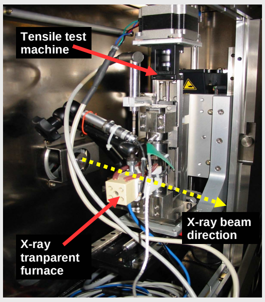

Fig. 1: Experimental setup

Data evaluation

Guinier-Model:

In a first approximation the radii were calculated using a Guinier-plot (q^2 vs. ln(I(q)):

$$I(q) = I_0 \cdot e^{\left(-\frac{q^2 R_g^2}{3}\right)}$$

Therefrom one gets the radius of gyration and can calculate the mean radius of the particles with:

$$R = \sqrt{5/3}\, R_g$$

The mean distance between the precipitates can be roughly calculated with the approximation:

$$d = \frac{2\pi}{q_{max}}$$

Hard-Sphere-Model:

The scattering intensity I(q) of a material consisting of identical particles:

$$I(q) \propto P(q)^2 \cdot S(q)$$

$$P(q) = 3 \cdot \left(\frac{\sin(qR) - (qR)\cos(qR)}{(qR)^3}\right)$$

$$S(q) = \frac{1}{1 + 8\eta\,(1+2\eta)^2 / (1-\eta)^4 P(q)}$$

P(q) ... Formfactor for spherical particles

S(q) ... Structurefactor for particle-particle interaction [1],[2] (Hard-Sphere-Model)

η ... Probability to find neighbouring particles.

Results

Temperature measurements:

- Al-2at.%Ag: 120°C, 150°C, 190°C and 220°C.
- Al-6at.%Ag: 150°C, 165°C and 190°C.

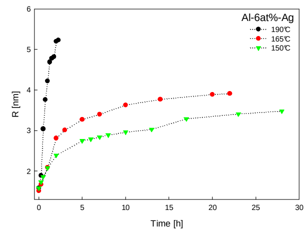

Fig. 2: Time evolution of the radii for an Al-6at.%Ag sample at different temperatures

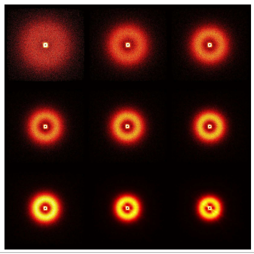

Fig. 3: Time evolution of the scattering pattern for an Al-6at.%Ag sample at 165°C, after quenching (top left) and after 28h (bottom right)

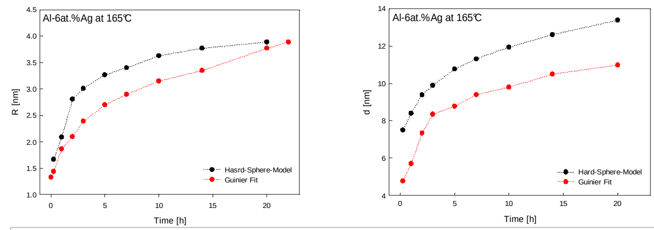

Fig. 4: Comparison of the radii and mean distance evolution of an Al-6at.%Ag sample at 165°C evaluated with „Guinier Fits" and with the „Hard-Sphere-Model".

Investigation of the effekt of external load on the precipitations' shape:

- Al-2at.%Ag alloys were investigated at 190°C for several external loads
- The samples had a thickness of 50 µm and a breadth of 5 mm
- Applied strain ⟶ (20 – 32) MPa

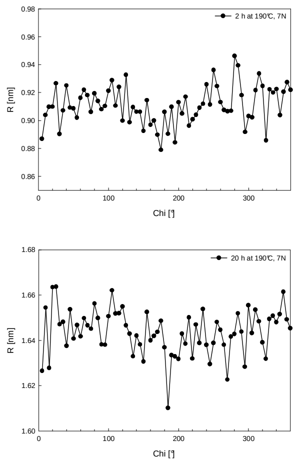

Fig. 6: Calculated radii for an Al-2at.%-Ag sample at 190°C and 7N external load after 2h (top) and after 20h (bottom). No significant dependence on orientation visible.

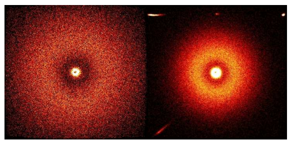

Fig. 5: Scattering pattern of an Al-2at.%-Ag sample at 190°C and 7N external load after 2h (left) and after 20h (right).

In order to proof a shape modification a cake integration with 36 bins in azimuthal direction (opening angle 10°) and 100 bins in radial direction was preformed. For every azimuthal bin the Guinier radius was calculated.

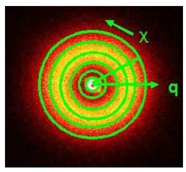

Fig 7: Scheme of a cake integration

Conclusion

- Guinier Fits are applicable while the precipitates have a diameter less then 3 nm
- For precipitates larger then 3 nm the „Hard-Sphere-Model" is a more significant evaluation method
- It was found that there were no measurable changes in the precipates' shape within a resolution limit of 1%. Possible reasons are:
  - Too low temperature
  - Too less strain
  - Too small starting precipitates

References:
[1] J. S. Pedersen, Adv. Colloid Interface Sci. 70, 1997, 171-210
[2] Kinning D. J., Thomas E. L., Macromolecules, Vol. 17, 1984
[3] Gupta et al., Acta Mater. 49 (2001) 53-63
[4] G. Kostorz „Phase Transformations in Materials", 2001, WILEY-VCH

Aknowledgements:

- Prof. B. Sepiol ist acknowledged for sample preparation and helpful discussions
- Dr. M. Leitner is acknowledged for helpful discussions
- D. Gitschthaler and Dr. S. Puchegger are acknowledged for technical support