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Published in:
MC 2011 Kiel

Publication date:
2011

[Link back to DTU Orbit](#)

Citation (APA):

Wollgarten, M., Chang, C. S. T., Duchstein, L. D. L., Schumacher, G., & Banhart, J. (2011). Quantitative investigation of precipitate growth during ageing of Al-(Mg,Si) alloys by energy-filtered electron diffraction. In MC 2011 Kiel: Microscopy Conference 2011 (pp. M5-P600). DGE – German Society for Electron Microscopy.

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Quantitative investigation of precipitate growth during ageing of Al-(Mg,Si) alloys by energy-filtered electron diffraction

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Keywords: Aluminum alloys, electron diffraction, precipitate growth

Besides other application fields, light-weight Al-(Mg, Si) (6XXX series) alloys are of substantial importance in automotive industries where they are used for the production of car body panels. The material gains its strength by precipitation of metastable Mg-Si-based phases. Though the general precipitation sequence of these phases is well studied [1,2], there remains an effect which is not fully understood up to now. Strengthening upon annealing, e.g. during paint baking of car body sheets, strongly depends on the storage duration at room temperature of the semi-finished parts [3,4].

It is commonly accepted that the early stages of precipitate growth are important for the understanding of this peculiar behaviour. During these stages, electron diffraction patterns of Al-(Mg, Si) alloys show diffuse features (Figure 1 (a) and (b)) which can be traced back to originate from β'' Mg₅Si₆ precipitates [5-7].

In this paper, we use energy-filtered electron diffraction to determine dimensions of the β'' Mg₅Si₆ precipitates along their a, b and c-axes as a function of ageing time and alloy composition.

In our contribution, we first derive that there is an optimal zone axis - $\langle 013 \rangle$ - from the view point of practicability. We show that diffraction patterns along this direction allow us to determine precipitate sizes along the b-direction independent of the extension along the a- and c-axes.

Sample material is obtained from Al-0.6wt%Mg-0.8wt%Si (type F) and Al-0.4wt%Mg-0.4wt%Si (type H) alloys made by Hydro Aluminium (Bonn, Germany) that have been solution heat treated for one hour at 540°C, ice water quenched and subsequently artificially aged for various durations at 180°C. Samples for transmission electron microscopy have been prepared by electropolishing of thinly cut sections. Experiments were carried out in a Zeiss LIBRA 200 operated at 200 kV using the in-column omega filter for zero-loss filtered electron diffraction. Recording was on imaging plates (made by Fuji company) which were read out in a scanner of type Ditabis Micron. Scanning resolution was about $7 \cdot 10^{-3}$ nm⁻¹/pixel.

Suitable parts of the recorded patterns are fitted by use of the kinematic diffraction theory (Figure 2). Assuming independent log-normal distributions for each precipitate dimension, the development of expectation value and its standard deviation is studied as function of ageing time (Figure 3).

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8. We thank C. Förster for preparation of the TEM samples.
9. We also thank Prof. J. Hirsch of Hydro Aluminium, Bonn, for providing the sample material.

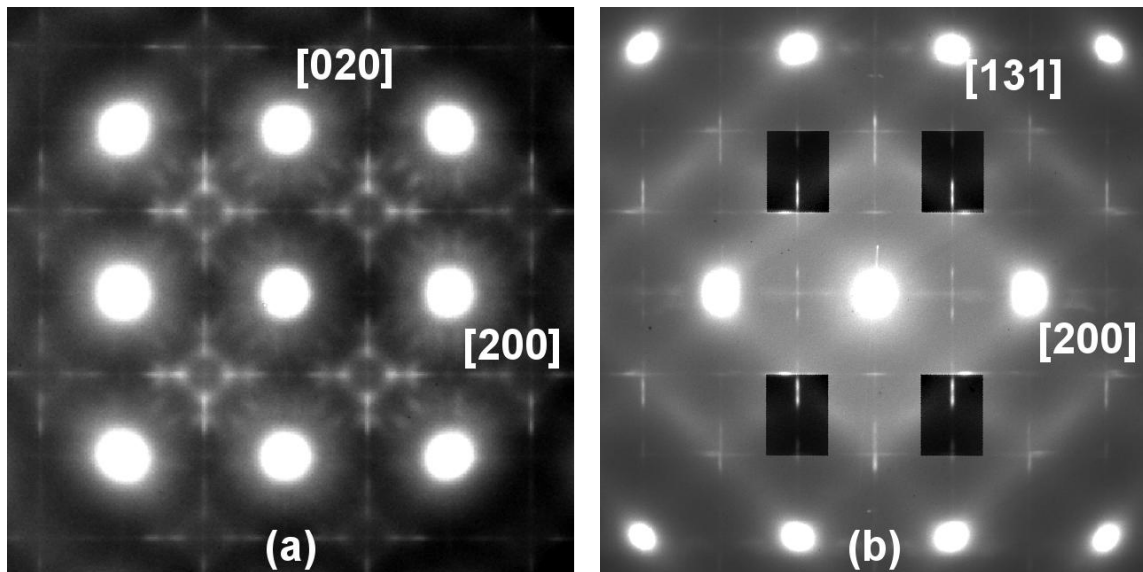


Figure 1. Energy-filtered electron diffraction patterns along a (a) $\langle 001 \rangle$ (alloy F, 18 h of artificial ageing) and a (b) $\langle 013 \rangle$ (alloy F, 8 h of artificial ageing) direction. The indices refer to the Al matrix phase. The background-subtracted areas in (b) are used for the fitting of intensity profiles (see also Figure 2).

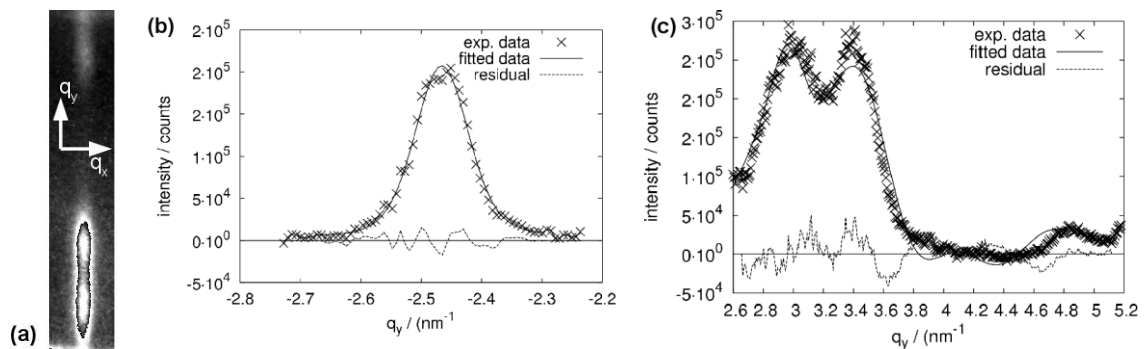


Figure 2. (a): Section of a diffraction pattern along a $\langle 013 \rangle$ direction which is used for fitting. Intensity profiles along the q_x -direction are used for the determination of the precipitates' b-dimension. Profiles along the q_y -direction are taken to determine the dimensions along a and c-axes. (b),(c): typical profiles along q_x - and q_y -direction, respectively. Experimental data is shown by crosses while the fitted data is drawn as continuous line. The bottom line represents the residual error.

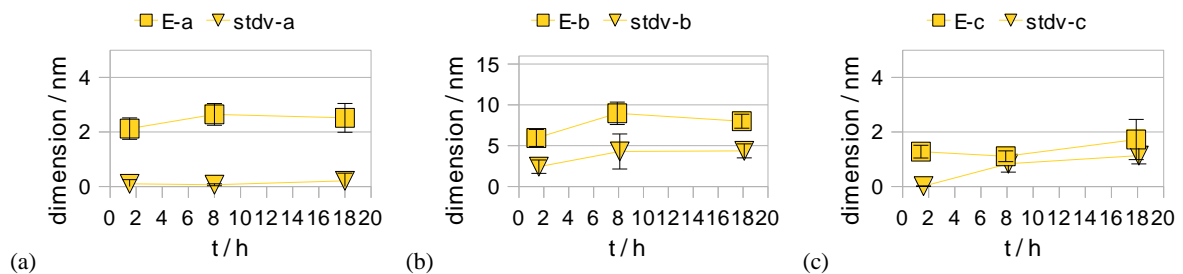


Figure 3. Plots showing the measured development of precipitate dimensions in alloy F along the a-, b- and c-axes as function of ageing time.