

s4.m13.o5 **Precession Electron Diffraction, High-Resolution Microscopy and Atom Probe Analysis of Nano-Size Precipitates in Al-Zn-Mg Industrial Alloys.**

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Keywords: Aluminum Alloys; Electron Crystallography; Electron Diffraction

Transmission electron microscopy is an excellent tool for characterization of the semi-coherent, metastable hardening precipitates embedded in aluminum alloys. However, structure solution by conventional electron diffraction is complicated, by double scattering via matrix reflections. This is overcome by the precession technique [1]. Composition is analyzed by the atom probe (APFIM) technique. The semi-coherent, disk-shaped ca 3x5nm-size η' -precipitate on the $\{111\}_{\text{Al}}$ planes, which determines the strength of Al-Zn-Mg alloys is an intermediate stage in a decomposition sequence: super-saturated solid solution \rightarrow GP-zones \rightarrow η' \rightarrow η -MgZn₂ (stable). The η' -lattice is hexagonal: $c_{\eta'} = 2[111]_{\text{Al}} = 1.402\text{nm}$, $a_{\eta'} = (1/2)[211]_{\text{Al}} = 0.496\text{nm}$. High-resolution images revealed that structure proposed earlier from X-ray photographs [2] could not be retained; a model based on a structural unit similar to the stable Laves phase MgZn₂ was proposed instead. The composition of the unit cell Mg₄Zn_{10-x}Al_{4+x} was chosen, which is close to the atom-probe result with $x \sim 3-4$. Electron diffraction intensity data were collected by the precession technique, on film and imaging plates. Three-dimensional data sets obtained by merging 5 or 7 projections recorded on film or imaging plates covered 85% of allowed reflections inside 1Å. From systematic absences the space group P-6c2 (190) was assigned. Patterson and Fourier analysis confirmed basic features of the HRTEM model. Refinement results will be presented; the relation to other structures along the transformation path will be discussed.

- [1] Vincent, R. & Midgley, P.A. (1994). *Ultramicroscopy* **53**, 271-281.
 [2] Auld, J.H. & Cousland, S.McK. (1974). *J Aust. Inst. Metals* **19**, 194-201

s4.m14.o1 **Structure Determination of the Precipitates of AlMgSi-alloys by electron Diffraction and Ab Initio Calculations.** SJ Andersen^a, CD Marioara^a, R Vissers^b, AG Frøseth^{c,d}, P Derlet^e, HW. Zandbergen^b ^aSintef Materials & Chemistry, Appl. Phys, N-7465 Trondheim, Norway, ^bNCHREM, Delft University of Technology, Rotterdamseweg 13, 2628 AL Delft, The Netherlands, ^cNorwegian University of Science and Technology, Dept. of Phys., N-7491 Trondheim, Norway, ^dPSI, Nano- Crystalline Materials Group, CH-5232 Viligen, Switzerland E-mail: sigmund.j.andersen@sintef.no

Keywords: TEM; Electron Diffraction; Ab Initio Calculation

The addition of just 1-2% Mg and Si to aluminium has a profound positive effect on the strength of Al-Mg-Si alloys. The reason is that a high density of very fine coherent, needle-shaped precipitates emerges from solid solution. The needles can have cross-sections below 5 nm². They extend along $\langle 100 \rangle_{\text{Al}}$ directions with lengths ranging from 1-2 nm up to hundreds. High Resolution Electron Microscopy (HREM) and electron diffraction (ED) are methods well suited for structural investigation. By these methods, in some cases supplied by *ab-initio* quantum mechanical (QM) methods, we have determined most precipitate structures of this alloy system: GP-zones [1], β'' -Mg₅Si₆ [2], β' -Mg_{1.8}Si [3], U1-MgAl₂Si₂ [4], and U2-MgAlSi [4]. QM calculations were used to relax coordinates of U1 and U2. The models are either developed on the basis of *exit waves*, HREM images or on the other precipitate structures, and aided by ED. When QM relaxed coordinates cause improvement in the calculated ED patterns, an atomic structure could be confirmed by ED refinements. The structures are related to each other, as well as to pure Si and Al. GP-zones and β'' take monoclinic (C2/m) super-cells in Al. U2, β' and B' (unfinished study) are orderings of Mg and Al on $\{111\}$ type Si-planes parallel with $\{100\}$ type Al-planes. β' (P6₃) and B' show extra reflections along c, which can be explained in terms of a modulation along c of the Si 00z atoms. For example, the average structure of β' (a=715 pm, c=405 pm) was refined to 000 (Si1), 2/3 1/3 1/2 (Si2) and 0.6152 0.6954 0 (Mg) with the occupancy of Si1 being 2/3. A tripling of the unit cell along c (i.e. z=0, 1/3 and 2/3 for both Si2 and Mg) together with the modulation -235-372-235- (pm) of the S1-atom, will explain the super-reflections, account for the occupancy, and prevent too close S1-S1 atoms. The modulation may be given as z=0.07 and 0.2634, but three equally possible, incommensurate strings exists. When a crystal contains similar proportions of the sets, super-reflections are absent [3]. Acknowledgements [5].

- [1] CD Marioara, SJ. Andersen, J Jansen and HW Zandbergen, *Acta Mater.* (2001) **49/2** 321.
 [2] Zandbergen, S.Andersen, H.W and J.Jansen, *Science* 29 Aug (1997) **277** 1221.
 [3] In preparation.
 [4] AG Frøseth, R Hoier, PM Derlet, SJ. Andersen and CD Marioara, *Phys. Rev. B* **67** (2003) 224106.
 [5] SJ Andersen and CD Marioara acknowledge funding by Hydro Al, Elkem Al, Raufoss Al, and the *Norwegian Research Council*.