Phase transformations in Al alloys during heat treatment investigated by *in situ* TEM

SRK Malladi[1,2], Q Xu[2], G Pandraud[3], FD Tichelaar[2], HW Zandbergen[2], K Kulawik[4], B Dubiel[4], A Czyrska-Filemonowicz[4]

1. Materials innovation institute, Mekelweg 2, 2628CD, Delft, The Netherlands
2. Kavli Institute of Nanoscience, National Centre for HREM, Delft University of Technology, Lorentzweg 1, 2628 CJ, Delft, The Netherlands
3. DIMES, Delft University of Technology, Feldmannweg 17, 2628 CT, Delft, The Netherlands
4. International Centre of Electron Microscopy for Materials Science, AGH University of Science and Technology, Al. A. Mickiewicza 30, 30-059 Krakow, Poland

S.R.K.Malladi@tudelft.nl
Keywords: nano-precipitates, TEM *in situ* heat treatment, ChemiSTEM

Most Al alloys undergo special heat treatment processes to obtain their desired properties. For example, mechanical properties of the Al 2024, Al 6005 and Al 7075 alloys are improved by precipitation hardening. In these cases, a distribution of many small precipitates to prevent dislocation motion is introduced by annealing a supersaturated solid solution at a relatively low temperature (artificial ageing). To study possible nucleation sites, growth and/or elemental distribution progress, *in situ* heating in a Transmission Electron Microscope (TEM) can be applied, as has been shown in the past in similar alloys [1,2]. In our study we apply two new techniques to get insight into the elemental distribution during the *in situ* heating of the investigated alloys, namely a low drift *in situ* heating holder (Delft) and a probe Cs corrected FEI TITAN[3] 60-300 (Krakow), operated at 300kV equipped with ChemiSTEM [3]. The combination of these tools make it possible to acquire elemental maps of a region at 512 x 512 pixel resolution (in this investigation), with pixel size in the range of 50 to 0.01 nm (in principle), in a relatively short time (3 minutes in our case). We use a MEMS based resistive heating device which was developed in-house with a specially manufactured low-drift TEM specimen holder to carry out the *in situ* heat treatment experiments [4] of the alloys. The temperature indicated in this holder is ±10%. Specimens for TEM from different alloys of 100 – 150 nm thickness were prepared with a Focused Ion Beam.

In this abstract, we show some results of the *in situ* heat treatments of two Al 2024 alloys, Al 2024-T3 (as received) and Al 2024-Q. The latter is an Al 2024-T3 alloy that was homogenized for 3 hrs at 500 ˚C before sample preparation and subsequently quenched to room temperature (RT) in a brine solution. This alloy was kept at room temperature for a week prior to the TEM experiments, probably leading to some Cu segregation. Only a few results of the 2024-T3 alloy are shown (Si and Fe map not shown) in Fig.1. The moments the series of elemental maps were taken are indicated by "A" and "C" in the T-time graph in Fig. 2 top row. At 100 °C (same structure as at RT), Cu and Mg segregation is visible at precipitates in the matrix and along grain boundaries. During the annealing, Cu, Mg and Mn segregated to the grain boundaries, as shown in Fig.1 for T=300 ˚C. The Si also segregated to the same positions as Mg (not shown). The Mn and Fe maps did not change. When observed carefully, new precipitates can be seen in the matrix at 300 ˚C. In Fig. 2 these precipitates are shown at an earlier moment at 250˚C at a larger magnification, showing rod-like precipitates containing Cu and Mg. These elements segregated into rods after the temperature increased from 200 ˚C to 250 ˚C. Stereo-STEM imaging confirms that the rod-like precipitates formed during heating are nucleated inside the specimen, not on its surface. On comparing these results to *in situ* heating of the Al 2024-Q specimen (Fig.2), we note that in the quenched sample, the precipitates emerge at a lower temperature, i.e. between 120˚C and 130˚C, as expected [5]. These experiments demonstrate that the *in situ* annealing technique shown here can be used to investigate different precipitation behavior resulting from different starting configurations of the materials in a relatively fast manner as compared to post-annealing experiments of bulk materials.

References

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[6] This research was carried out under the project number MC6.05222 in the framework of the Research Program of the Materials innovation institute M2i (www.m2i.nl), the former Netherlands Institute for Metals Research

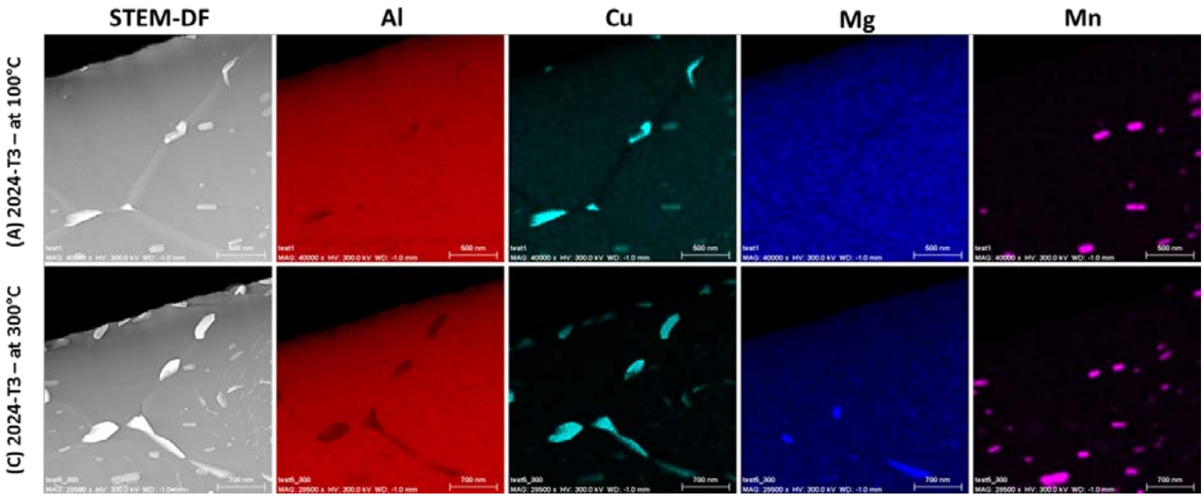

Figure 1*: HAADF STEM images and elemental maps in Al 2024-T3 obtained after in situ annealing at two different temperatures. Total width of each image is 2.4 µm in the top row and 3.4 µm in the bottom row. The moments that elemental maps were obtained are shown in Fig.2 top row (A at 100 ˚C for the top row and C at 300 ˚C for the bottom row).*

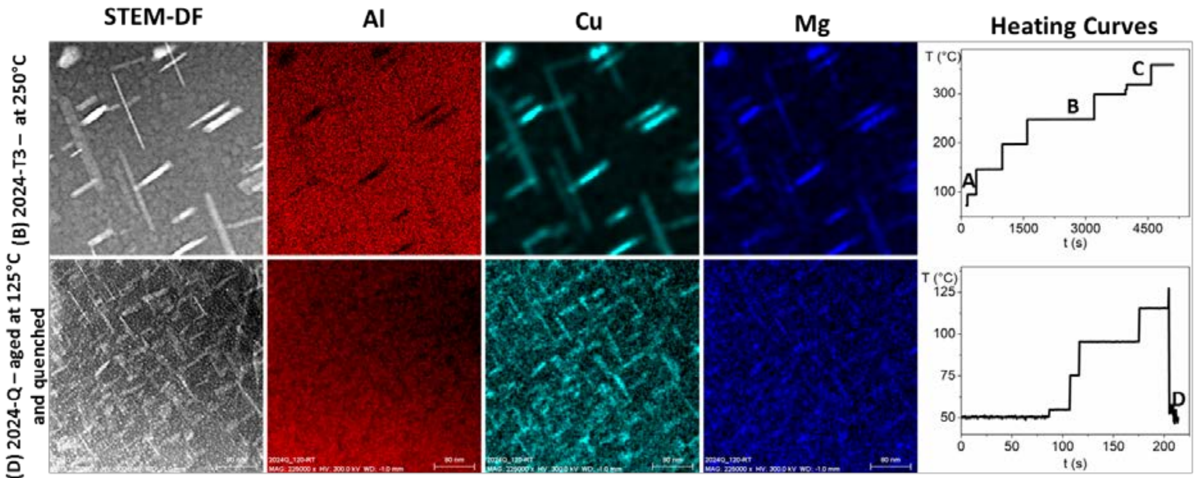

Figure 2*: HAADF STEM images and elemental maps in Al 2024-T3 and Al 2024-Q at the moment Cu-Mg precipitates were clearly formed. Total width of each image is 427 nm. The T-time graph shows the moments that elemental maps were obtained (x) and the ones that are shown here (B and D). In order to obtain the compositional maps at early stage of precipitation, the heating was switched off at 125 °C to arrest the further growth of precipitates.*