In situ observation of nano-precipitate growth

Application Note

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Dispersion strengthening is a common phenomenon used to enhance the strength of engineering materials. In commonly used structural alloys for automobile and aerospace applications like aluminum and nickel based alloys, this is achieved by formation of precipitates upon heattreatment, called precipitation hardening. The difference in solid solubilities of alloying elements makes it possible to precipitate fine particles of a secondary phase by heat-treatment, called aging.

DENSsolutions' Single Tilt (70 degrees) Sample Heating System (DENS-H-SH70) provides the opportunity to optimize this process by directly imaging the growth of nano-precipitates during the heat treatment. Fig. 1 gives an example for the application of the system in the study of precipitate growth in Al2024 (image by courtesy of Sairam Malladi [1]).

DENS-H-SH70 can heat specimens from room temperature to 1200 °C in a (S)TEM. Either fixed temperatures or a change of temperature with time (heating profile) can be easily set via computer control. The system has a temperature setting time of just seconds and a temperature stability better than 0.03 °C. The low spatial drift of under 1 nm/min at 800 °C keeps the area of interest in the field of view and enables atomic resolution imaging during heating.

DENS-H-SH70 large tilt range of ±70° enables the acquisition of tomography tilt series of the growing precipitates. Depending on the recorded image intensities, it might be possible to reconstruct and visualize the three dimensional density distribution of the sample. Fig. 2b shows a 3D network of nano-precipitates in Al2024 reconstructed from a series of HAADF-STEM images.

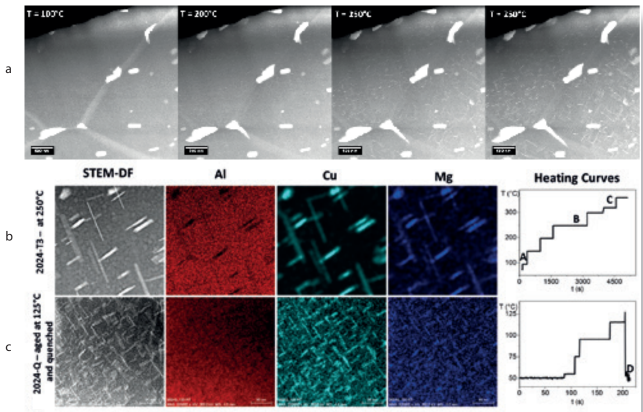

Fig. 1 (a) HAADF-STEM images of Al2024-T3 during heat-treatment from 100 to 250 °C. From 180 °C onwards, redistribution of copper is initiated and from 240 to 270 °C the nucleation of pillar-like nano-precipitates.
(b) Corresponding EDX elemental maps and heating profile. (c) EDX maps of a sample that was subject to a different heat treatment show differences in precipitate concentration and size.

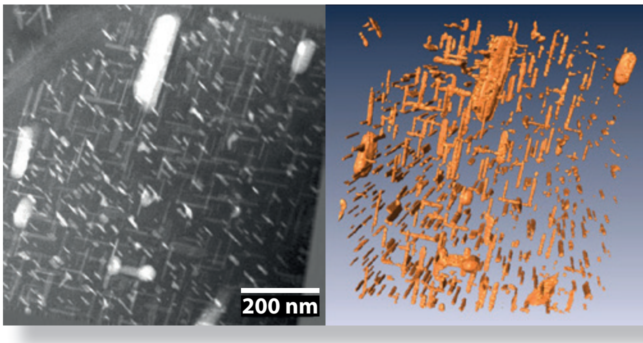

Fig. 2 (a) STEM-HAADF image from a series of tomography tilt images recorded during heat treatment of Al2024 [1]. While the nano-precipitates were visible in a single projection image, the interpretation of their 3D distribution, network, orientation and shape were facilitated by the inspection of a 3D density map calculated from the entire data set (b, surface rendering of 3D map).

[1] Images by courtesy of Sairam Malladi, National Center for HREM, TU Delft, The Netherlands, with thanks to Prof. Aleksandra. Czyrska and DSc Beata Dubiel, AGH University of Science and Technology, Krakow.

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