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Procedure for preparing Al specimens in [100] zone axis orientation for in-situ heating with a DENS chip

Protocols and softwares

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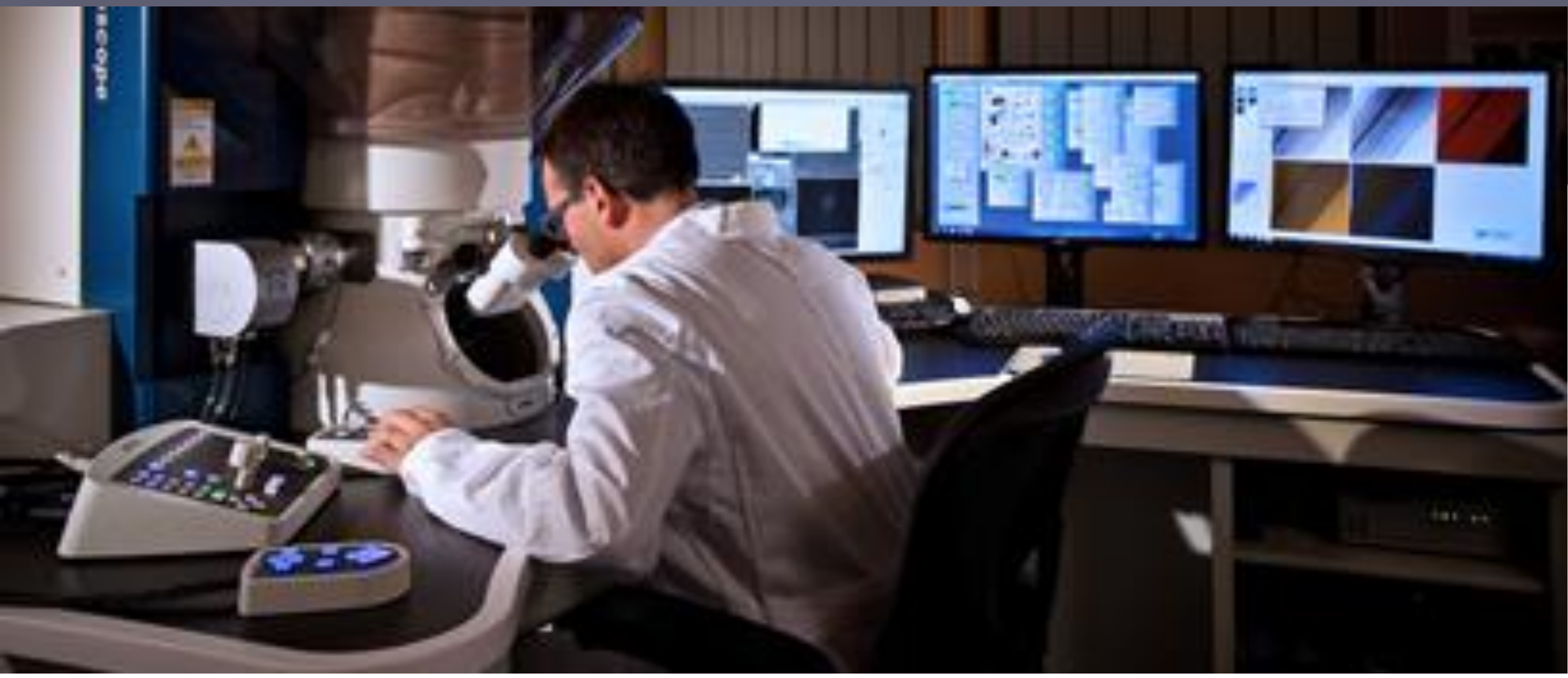

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Age-hardenable aluminium alloys (Al-Mg-Si, Al-Mg-Zn, Al-Cu..) gain their strength through nanosized precipitates formed in the aluminium matrix through heat treatments (see [1] and [2] and references therein). Transmission electron microscopy (TEM) specimens are typically made by electropolishing of thin foils, using an electrolyte consisting of 2/3 methanol and 1/3 nitric acid. The aluminium samples studied are multi-crystalline with grain sizes 1-100 um. Often the specimens should be observed in the [100] zone axis. Usually, the electropolished TEM specimens contain many electron transparent grains, so that it is possible to find grains in the [100] orientation within the tilt range of the specimen holder.

With in-situ heating experiments it is possible to observe precipitate evolution directly as a function of heat treatment. However, in order to perform in-situ heating using a WildFire DENSsolutions holder [3], it is necessary to attach the specimen to a chip while making sure to obtain the right orientation. Furthermore, aluminium specimens prepared with the FIB do not have as good quality as specimens prepared by electropolishing. The following procedure was therefore developed for preparing TEM lamellae of aluminium alloys to be studied in-situ in the [100] (or another given) orientation in a DENSsolutions holder.

The procedure is as follows (correlated with the numbers in Fig. 1):

1) Make an electropolished TEM specimen using standard procedure (grind down to foils of ~100 um thickness, punch out 3 mm disks, use a Struers Tenupol-5 electropolisher, with electrolyte 2/3 methanol and 1/3 nitric acid, cooled down to -20°C)
2) Perform TEM of the electropolished specimen, using a standard specimen holder. Identify an adequately thin grain oriented close to the [100] zone axis and save images of the grain and the surrounding area, so that the grain easily can be recognized in the FIB-SEM afterwards.
3) For FIB, attach the edges of the TEM specimen to a stub with conductive tape. Identify the [100] grain found in 2) by performing SEM. Mill out the [100] grain without exposing the region of interest (ROI) to the ion beam. Perform all milling and welding more than ~20 µm from the ROI. Attach the lift-out needle to the [100] lamella by ion beam deposition of carbon and transfer it to the DENS chip. Cut off the needle from the lamella by milling.
4) The DENS chip with the [100] grain can now be inserted into the TEM and the precipitate evolution can be observed in the right orientation during in-situ heat treatment.

Equipment used:

1) Struers Tenupol-5 electropolishing unit
2) FEI Helios G4 DualBeam FIB with lift-out needle
3) DENSsolution WildFire TEM holder with chips
4) TEM (JEOL 2100F used here)

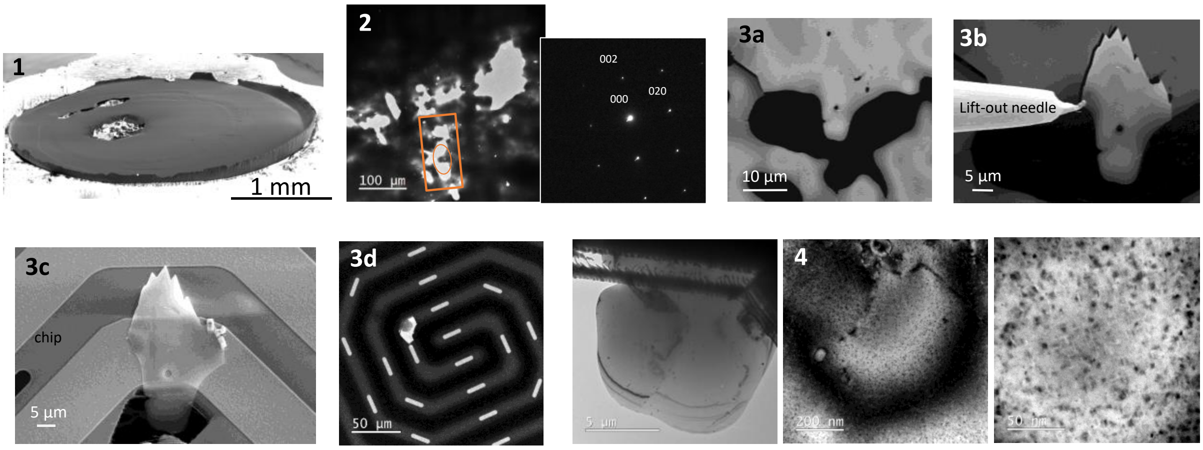

Fig. 1. Steps in the preparation of an aluminium TEM specimen in [100] orientation for in-situ heat treatment with a DENSsolutions holder. **1.** SEM image of an electropolished Al specimen. **2.** TEM is used to find a thin [100] grain. The inset shows a [100] zone axis diffraction pattern. **3.** FIB is used to mill out the grain and attach it to the chips. **3a.** Mill out the grain. **3b.** Attach the lift-out needle. **3c.** Attach the lamella to the DENS chip. **3d.** Cut off the lift-out needle. **4.** Study the sample in-situ in the TEM.

References:

[1] Andersen, S.J., Marioara, C.D., Friis, J., Wenner, S. & Holmestad, R. Precipitates in aluminium alloys, Adv. in Phys. X, 3:1, 1479984 (2018), https://doi.org/10.1080/23746149.2018.1479984;

[2] Saito, T., Mørtsell, E.A., Wenner, S., Marioara, C.D., Andersen, S.J., Friis, J., Matsuda, K. & Holmestad, R. Atomic structures of precipitates in Al-Mg-Si alloys with small additions of other elements Adv. Eng. Mater. 20, 1800125 (2018). https://doi.org/10.1002/adem.201800125.

[3] Sunde, J.K., Wenner, S. & R. Holmestad, In situ heating TEM observations of evolving nanoscale Al–Mg–Si–Cu precipitates, Journ. of Microscopy, 279, pp. 143–147 (2020). https://doi.org/10.1111/jmi.12845