1990
doi:10.1017/S1431927621007248
Microsc. Microanal. 27 (Suppl 1), 2021

Studying clustering in Al alloys by 4D-STEM

Elisabeth Thronsen[1], Adrian Lervik[2], Ding Peng[1], Calin D. Marioara[3], Jesper Friis[4], Sigmund Andersen[3], Philip Nakashima[5] and Randi Holmestad[6]

[1]Norwegian university of science and technology, United States, [2]NTNU, United States, [3]SINTEF Industry, United States, [4]SINTEF, United States, [5]Monash University, United States, [6]NTNU, Trondheim, Sor-Trondelag, Norway

Small clusters are formed by diffusion of atoms in solid solution during room temperature storage between solution heat treatment and artificial aging of heat-treatable Al alloys. The clusters affect the mechanical properties of the alloy and the its behaviour during the subsequent artificial aging treatment. Understanding the atomic structure and the dispersion of these clusters in the Al matrix is important for alloy design and thermomechanical processing to obtain desired properties. The clusters are typically spherical with a diameter of 1-3 nm [1, 2]. Due to the small size of the clusters compared to a typical Transmission Electron Microscope (TEM) specimen thickness, the cluster to Al signal ratio is low. This calls for more advanced techniques and sophisticated equipment compared to what is commonly required for study larger particles in the Al matrix [3]. In this work, 4D-STEM experiments were performed on Al alloys in conditions containing clusters to assess the applicability of such techniques to study the clustering phenomena.

Both scanning precession electron diffraction (SPED) and scanning convergent beam electron diffraction (SCBED) were performed on an Al-Zn-Mg alloy in a condition containing clusters. Recently, the crystal structure of clusters in the Al-Zn-Mg alloy system, the so-called GPI zones, was elucidated based primarily on high-angle annular dark-field scanning TEM (HAADF-STEM) images [1]. With this technique, the atomic columns of the GPI zones were resolved along its principal axis. The GPI zones were determined to consist of smaller cluster units stacking along certain directions to form larger clusters. Density functional theory calculations indicated the presence of an interstitial column at the center of these cluster units. However, since the atomic columns of the clusters were only resolvable along the principal axis, the interstitial column could not be confirmed based on the STEM images alone.

Figure 1a shows a HAADF-STEM image showing a fine dispersion of GPI zones in the alloy. The virtual dark-field image in b is obtained by placing a virtual aperture in the SPED data stack as shown in the inset. The data stack is taken from a different region than the HAADF-STEM image in a. Due to the low signal from the clusters, individual clusters were identified based on the virtual dark-field image and all PED patterns belonging to one single cluster were summed to enhance the signal. Figure 2 shows the unique PED patterns identified in the SPED data stack. In this talk, it will be shown how SPED can be used to verify or modify atomic models of clusters deduced from HAADF-STEM images. Moreover, SPED was found to give 3D structural information not attainable from the HAADF-STEM images. Recently we have also initiated SCBED experiments on the same alloy, to develop a methodology for performing quantitative CBED to extract information about the symmetries of the clusters with respect to the symmetry of Al. This will also be addressed in this talk.

Acknowledgments:

This work is supported by the Research Council of Norway, Hydro, Benteler and Neuman trough the SumAl (Solute cluster manipulation for optimized properties in Al-Mg-Si based Al alloys) project (NFR 294933), the NORTEM infrastructure (NFR 197405), the 'FICAL' project (NFR: 247598) and by Norwegian university of science and technology via the Digital Transformation initiative 'Alldesign'. The DFT calculations were performed on resources provided by UNINETT Sigma2 - the National Infrastructure for High Performance Computing and Data Storage in Norway (NN8068K)

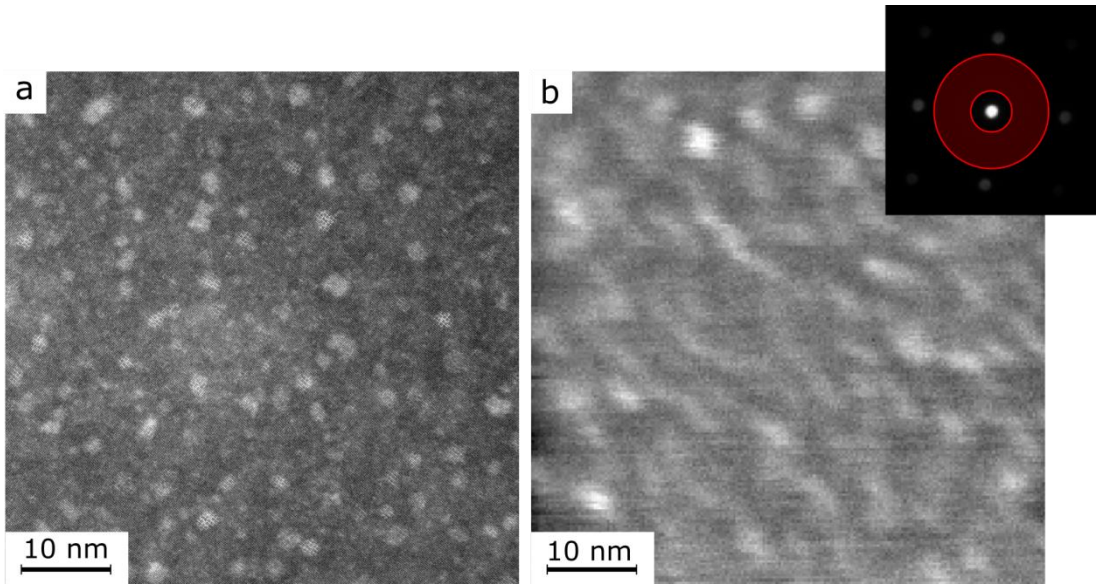

Figure 1. a: HAADF-STEM image showing a fine dispersion of GPI zones in the alloy studied. b: Virtual dark-field image created by placing a virtual aperture in the SPED data stack according to the inset.

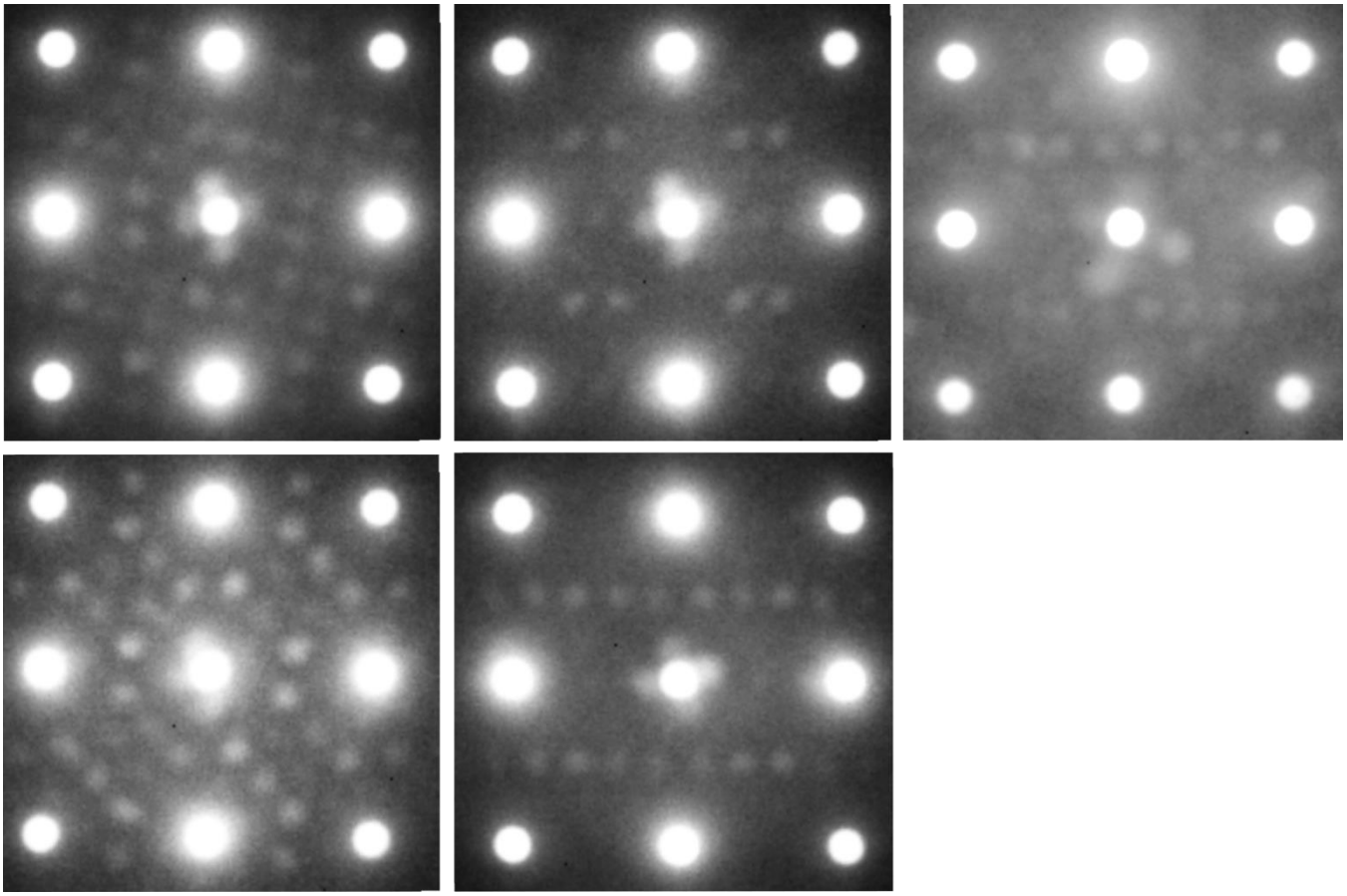

Figure 2. The five unique PED patterns identified in the SPED data stack.

https://doi.org/10.1017/S1431927621007248 Published online by Cambridge University Press

References

[1] A. Lervik, E. Thronsen, J. Friis, C. D. Marioara, S. Wenner, A. Bendo, K. Matsuda, R. Holmestad and S. J. Andersen. Atomic structure of solute clusters in Al-Zn-Mg alloys. Acta Materialia, **205**. 2021 DOI: 10.1016/j.actamat.2020.116574

[2] P. Dumitraschkewitz, S. S. A. Gerstl, L. T. Stephenson, P. J. Uggowitzer and S. Pogatscher. Clustering in age-hardenable aluminum alloys. Advanced Engineering Materials, **20**. 2018. DOI: 10.1002/adem.201800255

[3] S. J. Andersen, C. D. Marioara, J. Friis, S. Wenner and R. Holmestad. Precipitates in aluminium alloys. Advances in physics: X. 2018. DOI: 10.1080/23746149.2018.1479984