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Quantitative Atom Probe Tomography Analysis of Solute Clusters formed During Early Rapid Hardening in Al-Cu-Mg Alloys

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INTRODUCTION

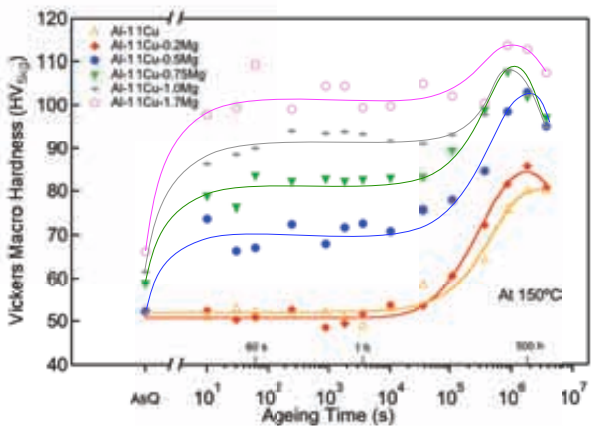
Al-Cu-Mg alloys which lie in the α+S phase field and have a low Cu:Mg ratio exhibit a remarkably rapid hardness increase during ageing at elevated temperatures [1]. This rapid hardening phenomenon (RHP) occurs in Al-1.1Cu-xMg alloys that contain at least 0.5 Mg (at.%), and may provide > 50% of the total hardness increment within the first 60 seconds of ageing.

Proposals to explain this phenomenon include the concept of solute cluster strengthening [2], but due to the difficulty to differentiate clusters from random compositional fluctuations in the solid solution, there has been a lack of work on the characterisation of solute clusters.

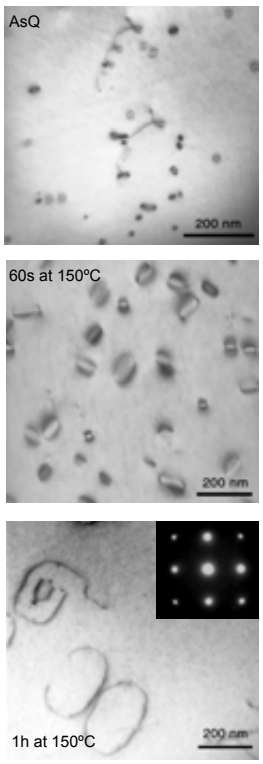
Atom probe tomography (APT) using the Local Electrode Atom Probe (LEAP) instrument together with statistical techniques and new cluster-finding algorithms has enabled identification, reconstruction and analysis of three-dimensional atomic scale clusters of solute atoms.

[1] J.T. Vietz and I.J. Polmear, J. Inst. Metals **94** (1966), p. 410-419.
[2] S.P. Ringer, T. Sakurai and I.J. Polmear: Acta Materialia **45** (1997), p. 3731-3744.

AGE HARDENING



TEM - Al-1.1Cu-0.5Mg

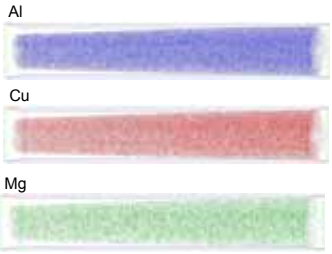


APT 3D MAPS - RAW DATA

Al-1.1Cu-0.5Mg
Aged 1 h at 150°C

322 x 58 x 55 nm
22.8 million atoms

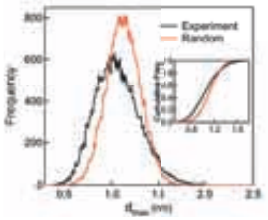
Need to perform data mining to extract useful information.



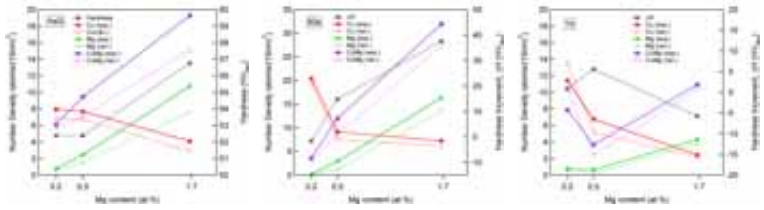
CORE-LINKAGE CLUSTERING ALGORITHM

Based on the principle that atoms involved in a cluster are closer together than atoms which form part of the matrix. Parameters can be chosen heuristically (see plot).

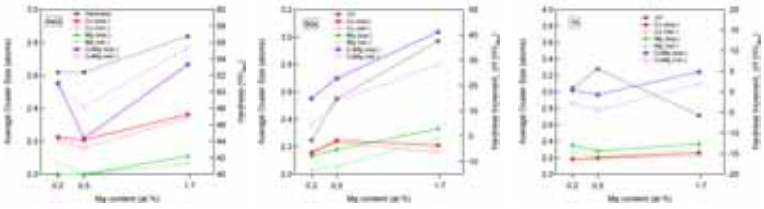
1. Atoms within d_{max} (nm) distance apart from each other are labelled "core" atoms
2. Atoms within d_{link} (nm) distance from core atoms become part of a cluster
3. Define a minimum number of atoms, N_{min}, per cluster



NUMBER DENSITY OF CLUSTERS



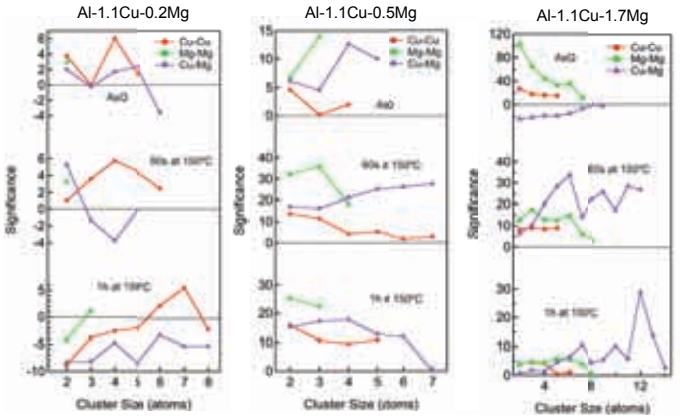
AVERAGE SIZE OF CLUSTERS



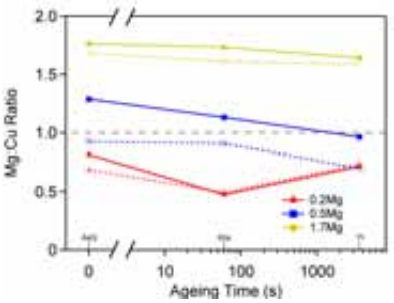
SIGNIFICANCE OF NUMBER DENSITY CLUSTERS

Statistical Significance $= \frac{N_V^{exp,i} - \sigma N_V^{ran,i}}{\sigma_{N_V(ran,i)}}$ $\quad \sigma_{N_V(ran,i)} = \sqrt{\frac{N_V^{ran,i}}{iV}}$

N_V = number density clusters
σ = standard deviation
V = volume
i = cluster size



CHEMISTRY OF CO-CLUSTERS



CONCLUSIONS

- Significant clusters have been observed at an atomic level using APT and a new clustering algorithm.
- Average cluster size is small - not yet mainstream thinking.
- 0.5 at.% Mg is critical composition threshold for rapid hardening (RH).
- Comparison of experimental and random cluster characteristics (number density, average cluster size) show differences between non-RH 0.2Mg alloy and RH alloys (0.5Mg, 1.7Mg).
- Number densities of Mg-Mg clusters and Cu-Mg co-clusters are more significant than Cu-Cu clusters
- These clusters correlate to the rapid hardening phenomenon.

ACKNOWLEDGEMENTS

RKWM would like to thank Mr Leigh Stephenson, Ms Anna Ceguerra, Mr Daniel Haley, Dr Michael Moody and Dr Tomoyuki Honma (all from The University of Sydney) for their help and support.

RKWM acknowledges scholarship funding support from the Australian Research Council and CSIRO.

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