

# Quantifying the Role of Vacancies in Precipitation-Hardened Aluminium Alloys

B.M. Gable<sup>1</sup>, P. Loo<sup>1,2</sup>, N. Ciccossillo<sup>1</sup>, S.N. Smith<sup>1</sup>, A.J. Hill<sup>2</sup>, T.J. Bastow<sup>2</sup> & B.C. Muddle<sup>1</sup>

<sup>1</sup> Department of Materials Engineering, Monash University, Australia  
<sup>2</sup> CSIRO Manufacturing & Infrastructure Technology, Clayton, Australia

## Abstract

This work investigated the role of quenched-in vacancies on the microstructural evolution and age-hardening response of a lean Al-Cu alloy. Through positron annihilation lifetime spectroscopy (PALS), transmission electron microscopy (TEM) and hardness testing it has been shown that there is a dramatic difference in  $\theta'$  particle size and distribution, resulting in ~10-15% difference in hardness, arising from varying the solution heat treatment temperature. These results indicate that the initial vacancy concentration influences the nucleation density and uniformity of both dislocation loops and  $\theta'$  particles. Coincidentally, this approach may provide useful insight into the study of autocatalytic nucleation.

## Introduction & Background

Improvements in mechanical properties arising from a finer and more uniform distribution of precipitates have been reported for some time, and these relationships are now quantifiable. Less well known is the precise role of vacancies in the nucleation of secondary phases, although it is clear that they influence solute diffusivity and thus the nucleation rate;

$$\frac{dN}{dt} \propto D_{Cu} \cdot e^{-\frac{Q}{kT}} \propto D_0 \cdot \text{Vacancy Conc.}$$

Positron annihilation lifetime spectroscopy (PALS) is a technique used for the analysis of vacancy-type defects. The vacancy concentration may be monitored over an extended period of time, making it useful for the study of ageing dynamics in alloys, including the early stages of phase evolution. PALS data reflects changes in the number of vacancies within the material, and in the nature/identity of the atoms surrounding those vacancies.

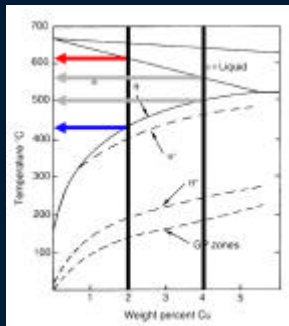


Figure 1 Al-Cu phase diagram

The Al-Cu binary phase diagram (Figure 1 [Porter & Easterling]) indicates that an Al-2.0Cu (wt%) alloy has an accessible single phase field (a) between ~430-610°C, which offers a much wider distribution than the richer Al-4.0Cu (wt%) alloy, while still offering reasonable age-hardenability [Hardy & Silcock].

## Experimental Procedure

An Al-2.0Cu (wt%) alloy was cast, warm rolled, homogenised, cold rolled and finally sectioned into ~20x10x0.5mm coupons. Two solution heat treatment (SHT) temperatures were chosen for comparison; 450°C and 600°C. Specimens were rapidly water quenched from the SHT, dried and immediately introduced to oil baths ranging from 150-200°C and later hardness tested at various ageing times. PALS data on naturally aged specimens were also collected in order to monitor the relative vacancy concentrations arising from the two different SHT temperatures. Conventional TEM was used to characterize the microstructural evolution. Calculations to estimate equilibrium vacancy concentrations were completed using the equation (expression given used for pure Al);

$$X \cong Ne^{-\frac{E_{Al}^v}{kT}}$$

Where  $X$  is the equilibrium vacancy concentration,  $N$  is the number of lattice sites,  $E_{Al}^v$  is the vacancy formation energy in Al,  $k$  is Boltzmann's constant and  $T$  is the absolute temperature.

## Results & Discussion

The natural ageing PALS results are illustrated in Fig.2a and demonstrate that there is a longer positron lifetime associated with the higher SHT, presumably from a greater quenched-in vacancy concentration.

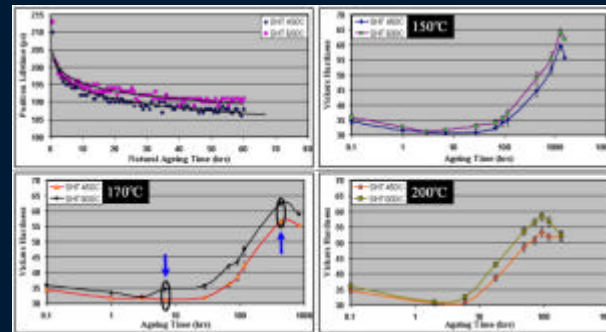


Figure 2 – Summary of PALS and age-hardening data

A collection of age hardening curves collected as a function of SHT temperature are also summarised on Fig.2b-d. Each curve demonstrates gains ~10-15% for the majority of ageing times between varying SHT's. It is also important to note that the ageing kinetics remain quite similar throughout the age hardening of the alloy.

Table I – Estimations of Relative Vacancy Concentrations

Temperature Ratio	Relative Vacancy Concentration
600°C / 450°C	~ 6
600°C / 170°C	~ 5400
450°C / 170°C	~ 900

Representative microstructures from the peak-aged conditions (500hr) for the 170°C treatment are given in Fig.3. These micrographs demonstrate that the SHT temperature dramatically affects the resulting  $\theta'$  particle distribution. The lower (450°C) SHT temperature resulted in a significant



portion of non-uniform  $\theta'$  particle 'groups', while the distribution of the  $\theta'$  from 600°C treatment is very uniform. A summary of the quantitative TEM analysis is given in Table II, which confirms that both the average nucleation density and plate dimensions are influenced by the initial vacancy concentration.

Figure 3 – TEM micrographs for the peak-aged condition at 170°C.

Table II – TEM  $\theta'$  Particle Analysis for 170°C Artificial Ageing

SHT (°C)	AA Time (hrs)	Secondary Feature	Diameter (nm)	Thickness (nm)	Number Density (# / $\mu\text{m}^2$ )
450	0	Dislocation Loops	67.3	—	27.4
	7	$\theta'$ plates	113.0	2.5	40.8
	500	$\theta'$ plates	368.4	4.7	38.4
600	0	Dislocation Loops	75.0	—	70.6
	7	$\theta'$ plates	108.4	2.2	33.7
	500	$\theta'$ plates	248.4	3.4	108.6

## Summary & Conclusions

- A high initial vacancy concentration led to a denser and more uniform distribution of  $\theta'$  plates when artificially aged, a microstructure that was responsible for ~10% gains in hardness
- Qualitative PALS results do confirm that the quenched-in vacancy concentration differs for the two SHT's employed
- The similarity in age-hardening kinetics suggests that the  $\theta'$  plate-lengthening kinetics are not markedly different between the two SHT's.
- This work may provide some insight into the impetus for autocatalytic nucleation in that initial vacancy population seems to be a key factor