

Solute-vacancy clustering in Al-Mg-Si and Al-Si alloys

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The Al-Mg-Si (6xxx series) aluminum alloys are in high demand as materials for vehicles because of their low weight, excellent formability and age hardenability.^{1,2)} The usual process for heat treatment is solution heat treatment at approximately 820 K followed by quick quenching with water, resulting in a supersaturated solid solution (SSSS). After storage at room temperature (called natural aging, NA), the alloy undergoes artificial aging (AA) at around 420 K, leading to a precipitation sequence:³⁾

SSSS \rightarrow Mg/Si/vacancy cluster \rightarrow Guinier Preston (GP) zone \rightarrow β'' \rightarrow β' \rightarrow β (Mg₂Si).

It is well known that the early stage of solute clustering of Mg and Si proceeds quite quickly and is completed in less than an hour, even at room temperature.^{4,6)} A long-standing problem for industries is that NA treatment often results in a negative effect on the mechanical strength in the following AA.^{3,7)} The microstructures of the precipitations have been studied intensively using transmission electron microscopy (TEM)⁸⁾ and atom probe tomography (APT)⁹⁾ to reveal the age hardening mechanism. These techniques, however, require time consuming sample preparation; thus, it is difficult to directly observe the early stage of clustering. Differential scanning calorimetry (DSC)¹⁰⁾ has been widely used to investigate precipitation processes and cluster formation, but in principle, this method cannot be used for isothermal measurements because the peak positions in a heat flow spectrum depend on the heating rate. From various studies on Al-Mg-Si alloys, it has been found that vacancy behavior is considered to play an important role in the aging process, enhancing the diffusion of solute Mg and Si atoms and nucleation of clusters. Positron annihilation spectroscopy (PAS)^{5,6)} and muon spin relaxation spectroscopy (μ SR)^{11,12)} have been successfully used to investigate the vacancy and clustering behavior in Al-Mg-Si alloys.

New observations of time dependent muon spin relaxation of an Al-1.6%Mg₂Si alloy at the isothermal condition at 280 K, 290 K, and 300K are presented in this report. All the samples underwent heat treatment at 848 K for 1 h and subsequent quenching in ice water (STQ). Approximately 10 min after STQ, the sample was inserted in an ARGUS muon spectrometer, and then, zero-field μ SR measurement was performed at a constant temperature. The observed spin relaxation spectra were fit using a Gaussian function with standard deviation $1/\sigma$ using the

WIMDA program,¹³⁾ where σ is a measure of the depolarization rate of the muon spins. The deduced values of the muon spin depolarization rates at 280 K showed little change in the time range between 20 and 100 min, then gradually decreased with time, and finally decreased linearly in a logarithmic time scale about 400 min after STQ. The depolarization rates at 290 K decreased slowly with time in the early stage till about 30 min, and then decreased linearly in a logarithmic time scale. Those at 300 K decreased in a logarithmic time scale from the early stage, but there are three different time regions. These time variations of the depolarization rates are quite similar to those of the positron annihilation lifetime in Al-Mg-Si alloys,⁵⁾ thus, they can be interpreted in terms of the clustering kinetics of a Mg/Si/vacancy. From an Arrhenius plot of the relaxation rates against the reciprocal measured temperature, the activation energy for clustering of Mg or Si atoms in an Al-1.6%Mg₂Si sample is deduced.

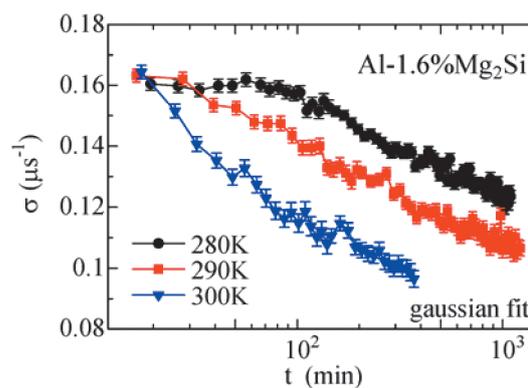


Fig. 1. Aging time dependences of zero-field spin relaxation rate for Al-1.6%Mg₂Si sample at constant temperatures of 280, 290, and 300 K.

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