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Influence of Heat Treatment on the Microstructure and Mechanical Properties of Hypoeutectic Al-5wt% Si Alloy

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Abstract

The present study aimed to investigate the influence of heat treatment on the microstructure and mechanical properties of Al-5wt% Si alloy by positron annihilation spectroscopy and Vickers microhardness measurements. Samples of Al-5wt% Si alloy were treated with a T6 heat treatment, that is, solution treatment at 550°C for 2 h, quenching in cold water at 0°C, followed by aging at different temperatures for various periods of time. All of the samples were then characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD) analysis as well as by hardness tests. The results revealed that the average life time, τ_{av}, and hardness, H_v, continuously increased with increasing aging temperature at lower aging times (15 and 30 min). At higher aging times (60 and 120 min), both τ_{av} and H_v decreased with increasing aging temperature. A positive correlation was found between the positron annihilation parameters and the macroscopic mechanical properties through the measurements of Vickers microhardness for the samples. The lattice strain, η, average crystallite size, d, and dislocation density, δ, parameters deduced from the analysis of X-rays diffraction patterns were found to be consistent with the calculated mechanical data and positron annihilation parameters. The variations in τ_{av} and H_v with increasing aging time and aging temperature have been explained in terms of the formation and/or dissolution of Si precipitates of different number and size.

Keywords

Al-Si Alloys, Positron Annihilation Life Time, Vickers Microhardness

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1. Introduction

Positron annihilation spectroscopy (PAS) is a unique tool of microstructure investigations of metals and alloys. It is well recognized as a unique probe to detect the micro-defects of materials due to its unique salient features such as non destructiveness, depth-sensitivity, and ease of use. Positron annihilation is very sensitive to open volume defects (like vacancies, vacancy clusters and dislocation loops) introduced by plastic deformation or misfit dislocations at the interface between semi-coherent or non-coherent precipitates and the surrounding matrix [1-4].

The binary aluminium–silicon (Al–Si) alloys can be classified into three main categories; hypoeutectic ones having less than 11wt% Si, eutectic alloys containing approximately 11–13wt% Si and hypereutectic alloys having more than 13% Si [5]. These alloys are the most widely used aluminum foundry alloys due to their low density, high

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specific strength, high wear resistance, low thermal expansion coefficient, good workability and excellent cast ability [6-8]. The excellent mechanical properties of these alloys can be referred to hard Si particles embedded in an Al-matrix. It is well known that the size and shape of Si particles in Al–Si alloy determine the mechanical properties of these alloys [9].

Several investigations have been performed using positron annihilation lifetime (PAL) for examining defects in Al-Si alloys [10-14]. The isochronal annealing of quenched commercially pure Al and Al-0.96wt% Si between room temperature (RT) and 550°C has been investigated [12]. The annealing of defects was investigated using Positron Lifetime Spectroscopy (PLS) and Doppler Broadening Spectroscopy (DBS). The retardation in the annealing of defects, in the two alloys was interpreted in terms of precipitation of impurities and Si atoms. Badawi et al. [15] studied the vacancy formation and migration in A365 (Al-7wt% Si-0.4wt% Mg) cast alloys, after quenching, using PLS and DBS from RT to 600°C. In their work also the vacancy formation enthalpy was calculated by different methods. Microstructural features and mechanical properties of Al-Si alloys have been studied by many authors [16-18]. The effect of silicon content on the performance of Al-Si cast alloys was studied in detail by many researchers [7, 8, 19, 20]. In some of those studies, it was shown that silicon content up to eutectic composition improved the tensile strength and wear behavior of the Al-Si alloys, but above this level it made an adverse influence on the properties [7, 8]. Therefore, the hypoeutectic alloys had higher wear resistance than hypereutectic alloys. Some other researchers showed that the wear resistance was improved with silicon content up to 16–20% [19, 20]. Torabian et al. [19] have studied a series of Al-Si alloys, ranging in silicon content from 2 to 20 wt%. They found that the increase in the silicon content in the alloy increased the wear resistance of alloy, with the effect being more pronounced up to 15 wt% silicon content. The stress-strain behavior Al-3wt% Si and Al-3wt% Si-1wt% Sn (or Ag) alloys have been investigated in the temperature range (140–220°C) [21]. It was observed that Sn addition improved the mechanical properties of the samples but this was not achieved with Ag adding which improved softening and ductility under the same test conditions.

In the present work, the effect of heat treatment on the mechanical properties and microstructure of Al-5wt% Si alloy was investigated using positron annihilation lifetime (PAL) and Vickers micro-hardness (H_v). An attempt was done to establish a correlation between the positron annihilation parameters and the macroscopic mechanical characteristics through the measurements of Vickers micro-hardness for the specimens under investigation.

2. Experimental Procedures

2.1. Samples Preparation

The commercially Al–5wt% Si alloy used in the present investigation was received from AlcoTec Wire company-USA. The alloy was spectroscopically analyzed at AlcoTec Wire company using the OES-3500 optical emission spectrometer. The analysis data are given in Table 1. After annealing for homogenization at 550°C for 60 h, the samples were cut into squares of 1cm side and 1.25 mm thickness. After solution heat treatment at 550°C for 2 h, to produce the α- solid solution phase, the specimens were quenched into cold water at 0°C and immediately aged at different aging temperatures, T_a (200, 250, 300 and 350°C) for various periods of time, t_a (15, 30, 60 and 120 min) followed by water quenching at 0°C. The accuracy of the temperature measurements is of the order of ± 1°C.

Table 1. Chemical composition (wt%) of the alloy used in the present study.

Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
5	0.15	0.001	0.03	0.003	0.003	0.006	Remainder

2.2. Positron Annihilation Lifetime Measurements

The positron source was prepared by depositing $^{22}NaCl$ aqueous solution (about 20μCi) on a thin kapton foil (7μm thick). $^{22}NaCl$ spots were dried and covered with another identical foil glued together by epoxy glue and evacuated for a long time (more than 24 h) to be convinced that no air between the two foils. A conventional positron lifetime spectrometer was used for PAL measurements [22]. In this technique, two detectors are placed very close to the source-sample sandwich to observe the emission (signaled by 1.28 MeV γ- quantum) and the annihilation (signaled by a 0.511 MeV γ- quantum) of the positrons, thus making it possible to record their lifetime, i.e. the time difference between the two signals. To calculate the time resolution of the system, the positron annihilation lifetime spectrum of the kapton sample was measured. The kapton seems to be the only polymer with no positron yield, i.e. has no long-lived components. The time resolution was determined using RESOLUTION program [23] and is found to be 220 ps (full width at half maximum, FWHM). The sample/positron source/sample sandwich was put in a glass tube to perform the PAL measurements in vacuum at room temperature (about 27°C). The accumulation time ((3 hours) provided excellent counting statistics namely 10^6 counts.

In the present study, all obtained spectra were analyzed using the LT 9 code [24] to extract the following positron annihilation parameters: the lifetime components of positrons in free and bound states (τ_1& τ_2) and their intensities (I_1& I_2). The third component τ_3 represents the formation of positronium at the surface layer of the sample; it could be neglected for alloy samples. Meanwhile, positron average lifetime, τ_{av}, can be calculated using the formula; $\tau_{av} = I_1\,\tau_1 + I_2\,\tau_2$.

2.3. Hardness Measurements

All Vickers microhardness measurements were taken at room temperature by using a Shimadzu 2 series microhardness tester with a pyramid shaped diamond indenter. Indentations were done in the central area of the sample such that they lie away from the edges. Care was taken to keep the sample surface perpendicular to microscope axis. The average of at least ten indentations was reported. The hardness values (H_v) were obtained using 4.903 N load and 10s dwell time.

2.4. Microstructure Examinations

Standard scanning electron microscopy (SEM) and X-ray diffraction (XRD) were carried out on selected samples for different experimental conditions. For the microstructure investigations, specimens were ground with silicon carbide papers of successive grades size 120, 320, 800 and 1200 and polished with one-micron diamond paste. A mixture of 60 ml ethylene glycol, 20 ml acetic acid, 1 ml concentrated HNO_3 and 19 ml water was used for etching samples. The microstructure of the samples was studied by SEM and XRD.

X-ray diffraction patterns were recorded at room temperature on a Philips PW3710 X-ray diffractometer by utilizing Cu Kα radiation with a wavelength λ of 1.5406 Å operated at 40 kV and 40 mA settings. For phase identification, measurements were scanned for a wide range of diffraction angles (2*θ*) ranging from 20 to 100° with a scanning rate of 2°/min.

3. Results and Discussion

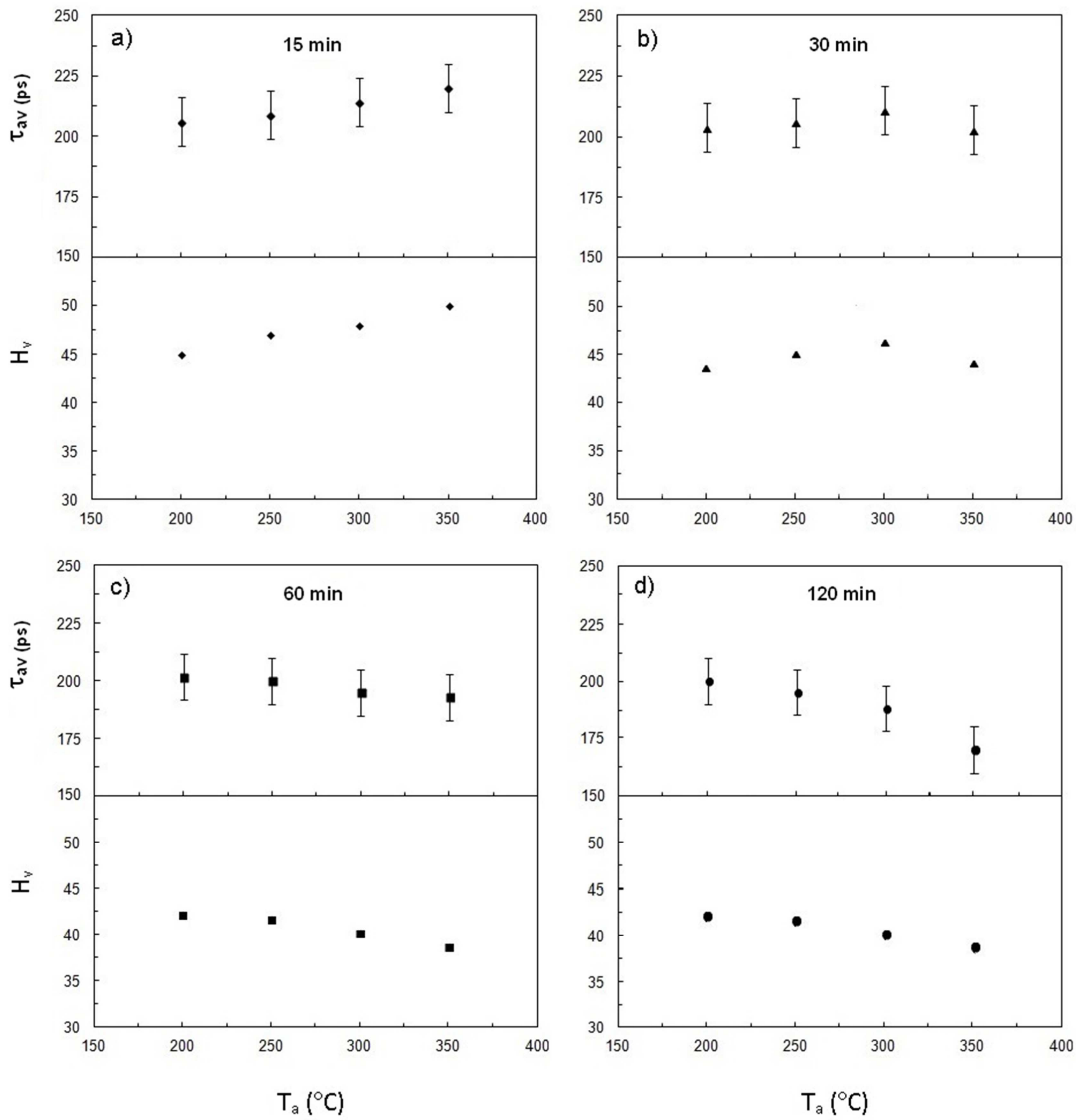


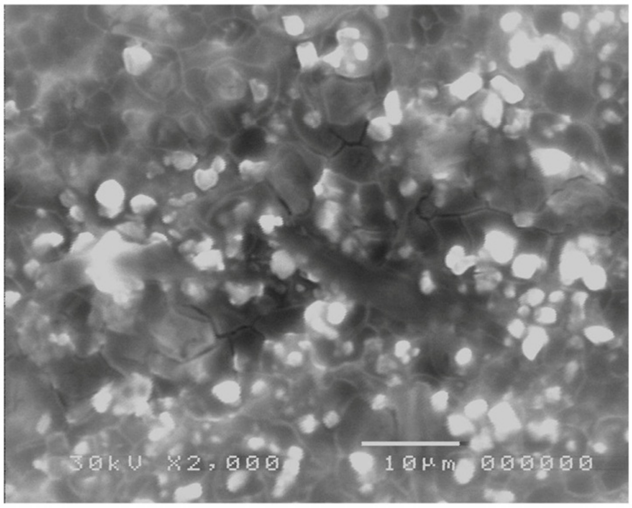
Fig. 1. τ_{av} and H_v as a function of aging temperature, T_a, for Al-5wt% Si alloy aged for a) 15 min, b) 30 min, c) 60 min, and d) 120 min.

The variation in average life time, τ_{av}, and hardness, H_v, values is mainly influenced by the precipitation and/or dissolution of solute atoms. The degree of strengthening obtained is highly dependent upon the volume fraction and size of the particles, as well as the nature of the interaction of the particles with the dislocations [25]. Figure 1 (a-d) shows τ_{av} and H_v as a function of aging temperature, T_a, for samples of Al-5wt% Si alloy pre-aged for different periods of time ranging from 15 to 120 min. From Fig. 1a, it is clear that for samples aged at lower aging time (15 min), the values of average life time, τ_{av}, and hardness, H_v, are continuously increased with increasing aging temperature. This behavior could be explained in view of the following mechanism. As the Al-Si alloy has been solid-solution heat treated and subsequently quenched, the quenched-in defects (small vacancy clusters) disappear by the formation of dislocation loops or voids [26]. These clusters act preferential nucleation sites for Si precipitates during aging. Simultaneously, some of the Si atoms precipitate on the loops stabilising them. The number of precipitated Si atoms increases with increasing aging temperature (Fig. 2 a and b) leading to an increasing number of stabilised loops. This process explains the initial quick increase in the values of τ_{av} and H_v (Fig. 1a). This behavior is in agreement with the reported results by Nakagawa et al. [27] who concluded that vacancy clusters would act as centres for nucleation of Si precipitates on thermal aging.

The above interpretation based on formation of Si particles is strongly supported by X-ray diffraction pattern. Figure 3 shows a typical X-ray diffraction pattern of samples aged for 15 min at different aging temperatures. As it is clearly shown, no lines corresponding to Si could be observed at aging temperature of 200°C. This means that the lower aging time and aging temperature were not enough to precipitate Si particles in the Al matrix. With increasing the aging temperature relative strong diffraction lines corresponding to Si particles has been observed for samples aged at higher aging temperature (250-350°C).

For samples at aging time of 30 min, the values of τ_{av} and H_v increased as aging temperature increased up to 300°C and then slightly decreased at 350°C (Fig. 1b). This behavior can be attributed to the formation large number of Si precipitates in the aging temperature range 200-300°C (Fig. 4a). With increasing aging temperature from 300 to 350°C, some of Si precipitates start to coarsen (Fig.4b) and tend to lose their coherence with Al-matrix. Since the incoherent precipitates make limited contribution to the hardness of the alloy [28], a reduction in τ_{av} and H_v values should be expected in the corresponding aging temperature 300-350°C. For longer aging times (60 to 120 min), the decrease in τ_{av} and H_v values could be attributed to the coarsening and/or dissolution of Si precipitates. Thus the probability of positron trapping at the precipitates is decreased since the volume fraction of Si precipitates increased.

a)



b)

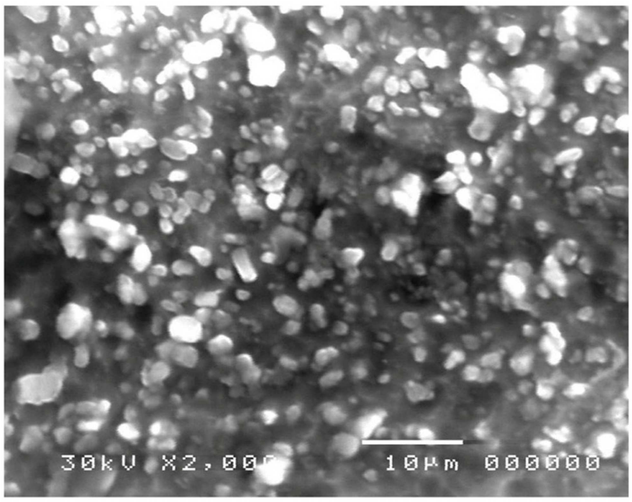


Fig. 2. SEM micrographs of Al-5wt% Si alloy aged for 15 min at a) 250°C and b) 350°C showing Si precipitates. More number of dispersed Si precipitates is observed at higher aging temperature (350°C) compared to lower aging temperature (250°C).

It is well known that, the broadening of XRD peaks of an alloy can be caused not only by the lattice micro-deformations but also by the small grain size of the alloy [29]. The average crystallite size, d, and the lattice strain, η, can be evaluated by XRD peaks analysis according to the Williamson–Hall formula [30]:

$$\beta \cos \theta = k\lambda/d + 2\eta \sin \theta \quad (1)$$

where β is the peak width at half maximum intensity (FWHM) of the main peaks, θ is the Bragg angle, k is the Scherrer constant (k=0.9) and λ is the X-ray wavelength (λ = 0.15406 nm). By plotted $\beta \cos \theta$ was against $\sin \theta$, straight lines are obtained with the slope of 2η and the intercept as ($k\lambda/d$). The dislocation density, δ, can be defined as the length of dislocation lines per unit volume of the crystal and calculated by using the formula [31]:

$$\delta = 1/\eta^2 \tag{2}$$

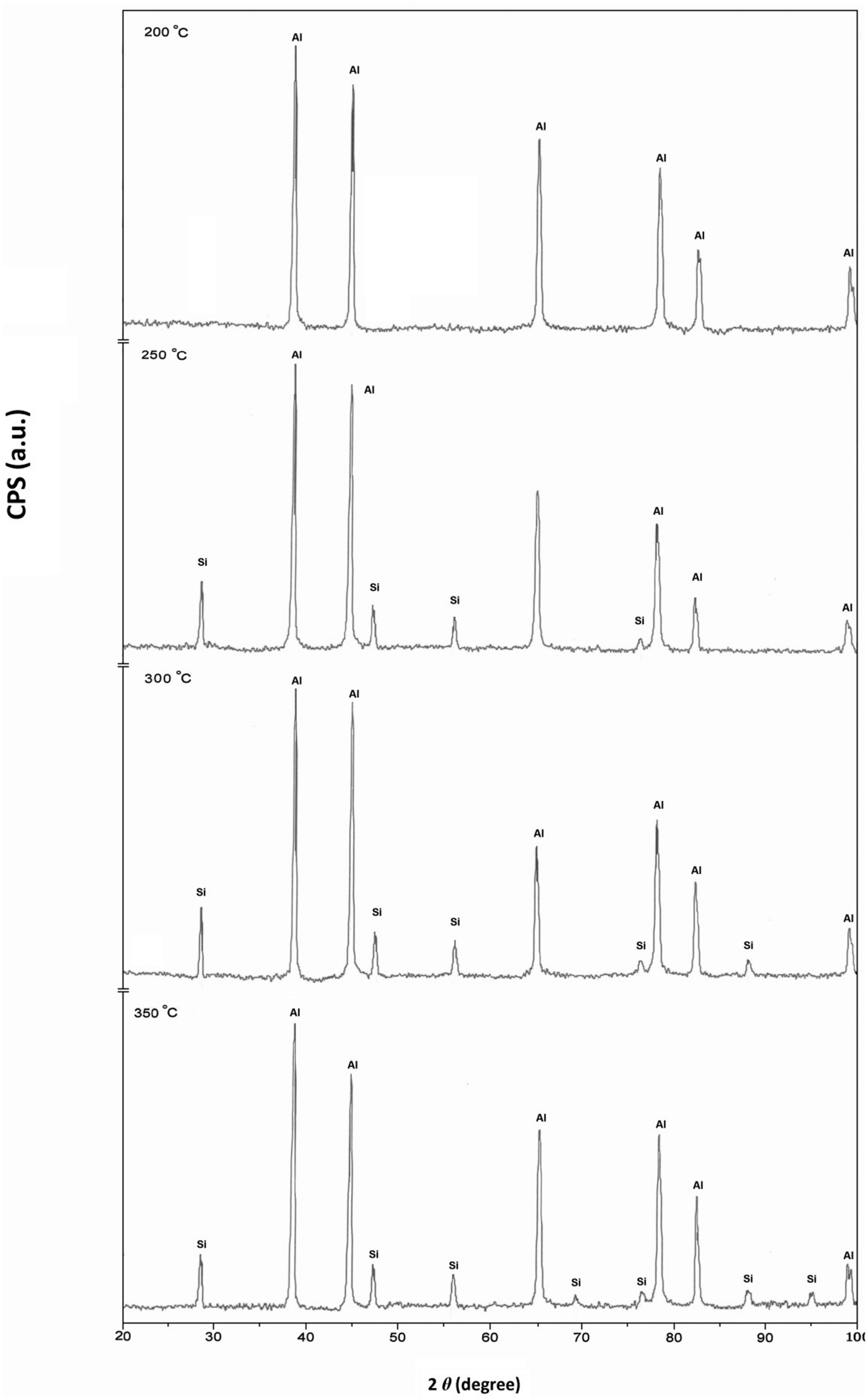


Fig. 3. XRD pattern of Al-5wt% Si alloy aged for 15 min at different aging temperatures.

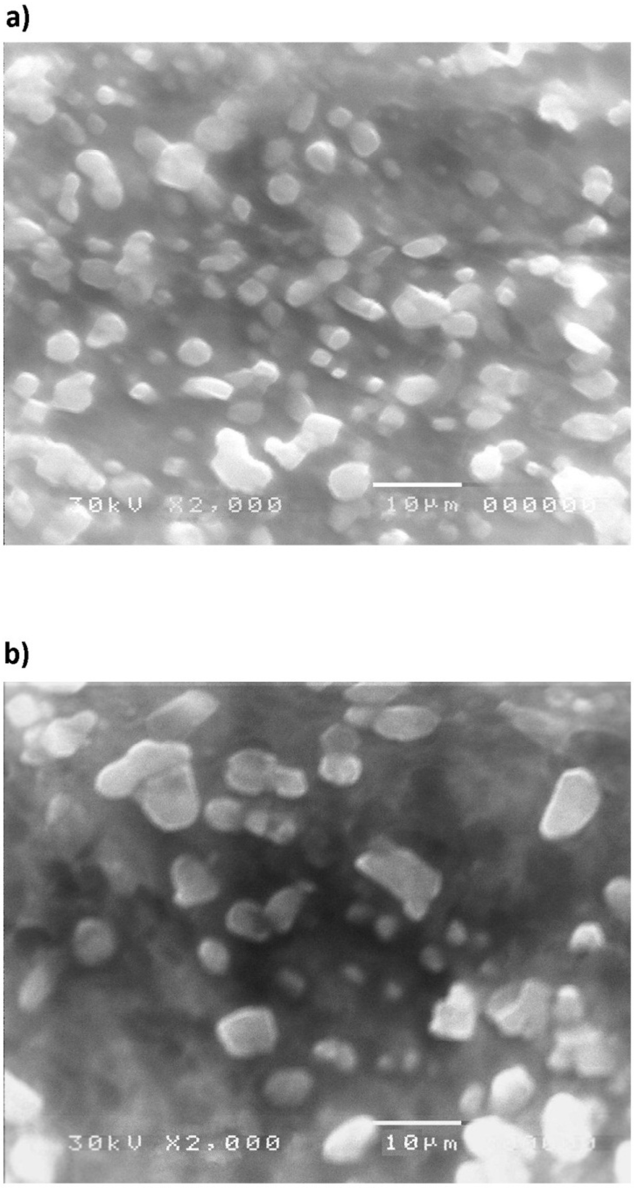


Fig. 4. SEM micrographs of Al-5wt% Si alloy aged for 30 min at a) 250°C and b) 350°C showing the coarsening of Si precipitates at higher aging temperature (350°C).

The aging temperature dependence of the lattice strain, η, the mean crystallite size, d, and the dislocation density, δ is depicted in Fig. 5. It can be observed that the aging temperature dependence of both lattice strain η, and dislocation density δ are in contrary to the behavior of the crystallite size d. The increase in both lattice strain, η, and dislocation density δ values with increasing aging temperature for lower aging times (15 and 30 min) could be attributed to the precipitation of Si-rich phase in the Al–rich phase matrix. For longer aging times (60 and 120 min), the dissolution of the second phase (Si–rich phase) in the Al-rich phase matrix with increasing aging temperature resulted in the increase in the homogeneity of the α–phase matrix and the homogeneity of the distribution of Si atoms in the Al–rich phase matrix leading to the observed decrease in both η and δ.

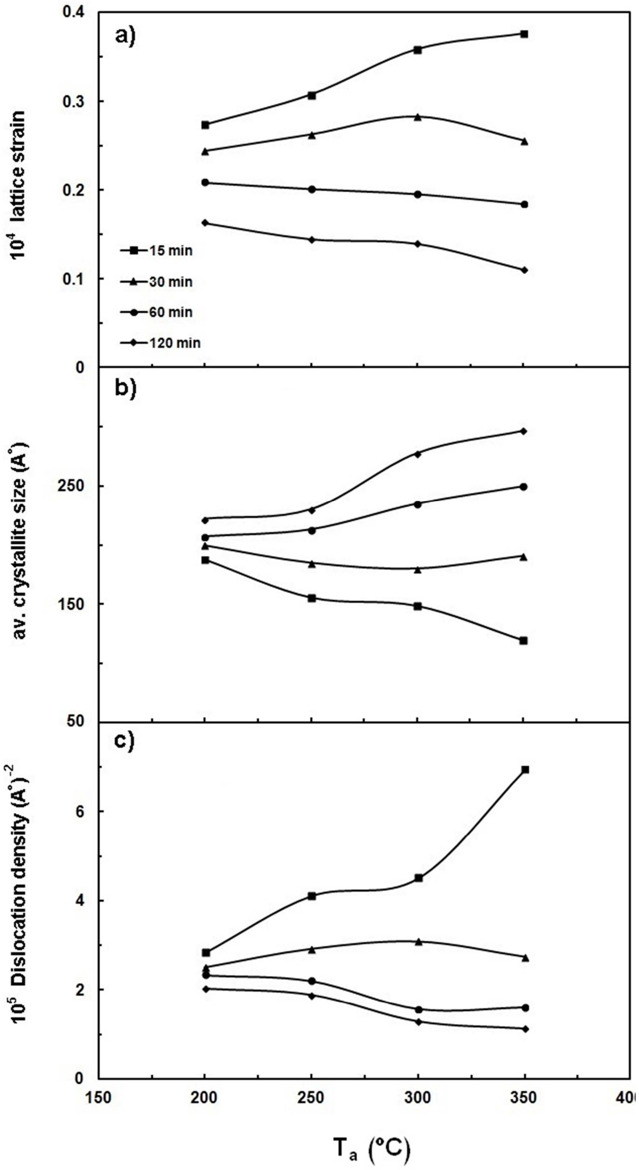


Fig. 5. The aging temperature, T_a, dependence of (a) lattice strain, η, (b) average crystallite size, d and (c) dislocation density, δ at different aging times.

4. Conclusions

Positron annihilation lifetime (PAL) spectroscopy and Vickers microhardness measurements (H_v) were performed to study the effect of heat treatment on the microstructure and mechanical properties of Al-5wt% Si alloy. The variation in the average life time, τ_{av}, and hardness, H_v, values with increasing aging temperatures and aging times was explained on the basis of formation and/or dissolution of Si precipitates in Al-Si system. A positive correlation was found between the positron annihilation parameters and the macroscopic mechanical properties through the measurements of Vickers microhardness for the samples.

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