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# Positron Annihilation and XRD Studies on Deformed Al-Alloys with low concentration of Mg

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### Abstract

Positron behaviour at grain boundaries is characterized by their bulk diffusibility D, their bulk free lifetime $\tau_f$ and their trapped lifetime $\tau_t$ ($\tau_t > \tau_f$). Al-Mgx samples (x = 0-1.4 at %) have been homogenized at 573 K for 12 hours. The mean lifetime, as (indirectly) measured at various concentrations, show a good fitting with Sigmodel giving $\tau_t$ = 180.664 ps and $\tau_f$ = 163.9 ps. The mean lifetime was found to vary exponentially with the degree of deformation. The XRD study showed that preferred orientation occurred at degrees of deformation > 32 %, whereas the peak intensity then increases with sample deformation. Heat treatment (recrystallization) is also followed by XRD study. The peak intensities decrease with increasing annealing time (attributed to recrystallization), but it does not completely reverse to the condition origin. For all cases, no peaks of Mg were revealed indicating the positron annihilation as a sensitive technique for detecting low concentrations in alloys.

## 1. Introduction

Information on the concentration, configuration and various microstructure characteristics of solids (and their lattice defects) can be obtained by positron annihilation technique. The positron lifetime, $2\gamma$-angular correlation and Doppler broadening of the annihilation line was successfully used in defect studies, especially in metals [1,2]. The clustering of vacancies in quenched metals was studied largely by resistivity measurements and electron microscopy [3]. Electron microscopy can provide information about defect clusters such as their concentration, size and spatial distribution. However, because of the limit in resolution of the conventional electron microscope, vacancy clusters smaller than about 10 Å in diameter cannot normally be detected. Positron annihilation spectroscopy has proved a useful alternative tool [4]. With the introduction of a sufficient concentration of open volume defects (e.g. about 10 ppm vacancies) a large fraction of thermalised positrons can interact with electrons and become trapped in defects. A positron is strongly localized in a single vacancy and even more strongly in a vacancy cluster [5]. Grain boundaries in materials posses a more open structure than the perfect crystal as shown by Seeger and Schottky [6]. The potential energy of positrons will be lower in grain boundaries than in the bulk of the metal. Since in one dimension an attractive potential well gives rise to at least one bound state, no matter how shallow and narrow the well, $e^+$ can be localized in the grain boundaries. At sufficiently low temperatures, grain boundaries in metals will, therefore, act as traps for thermalized $e^+$. Owing to the reduced electron density, the lifetime of positrons in grain boundaries $\tau_b$ is larger than the free lifetime $\tau_f$ of $e^+$ annihilating in a perfect environment. The mean lifetime should increase with decreasing mean grain size $l$ "Signature Euro" . This increase depends on the fraction of implanted $e^+$ that are capable of reaching the grain boundaries during their lifetimes. Therefore, there is at least the possibility at least in principle, to deduce the $e^+$ diffusivity $L_d$, from the observed $\tau$ vs. $\ell$ relationship. The first systematic data on the dependence of the $e^+$ lifetime on the mean grain size has been introduced by Lynn et al. [7] on polycrystalline Cu, which interpreted the results in terms of $e^+$ trapping at grain boundaries as did Leighly [8]. More detailed measurements of $e^+$ annihilation on fine-grained ZnAl alloys as a function of the mean grain size were also reported by [9,10]. For large grain sizes, the mean lifetime $\tau$ varies linearly with the inverse grain size $\ell^{-1}$, in agreement with most of the available experimental data [10]. Some measurements had been pervious performed on alloys by MacKee et al. [9], Xianyi et al., [11], Weiming et al. [12], Abdelrahman [13] and Dupasquier et al. [14]. The present paper shows use of the lifetime technique to study the change in grain size with Mg concentration and degree of deformation in Al-Mg$_x$ alloys. The microstructural parameters of the samples have been evaluated using X-ray diffraction (XRD) and the results have been correlated with those obtained by positron annihilation technique.

## 2. Experimental

Five samples of Al-Mg$_x$ alloy (x = 0, 0.23, 0.85, 1.27 and 1.4 at %) were homogenized at 573 K for 12 hours then slowly cooled to room temperature. As shown schematically in Fig. (1), the positron source was sandwiched between two identical samples. The source was $^{22}$Na, deposited from aqueous (sodium chloride) solution on a thin Kapton foil. The source - sample configuration was then wrapped in a thin Al foil. The block diagram of the system used in measuring lifetimes is shown in Fig. (2) and described elsewhere [15,16]. The positron lifetimes were recorded by a time spectrometer using a fast/fast coincidence method. The time resolution of the system using $^{60}$Co source was approximately 280 ps (FWHM). The coincidence counts rate were accumulated in each spectra for all samples during a period of 1 h. The lifetime spectra were analyzed with computer program PATFIT [17]. The X-ray diffraction analysis was used to obtain information on the preferred orientation, crystallite size and recrystalization, using a JEOL diffractometer Model (60 PA)with Ni filter and Cu-K$_\alpha$ radiation $\lambda = 1.5418$ Å.

The diffractograms were recorded from $2\theta$=30° to 100° at low scanning speed.

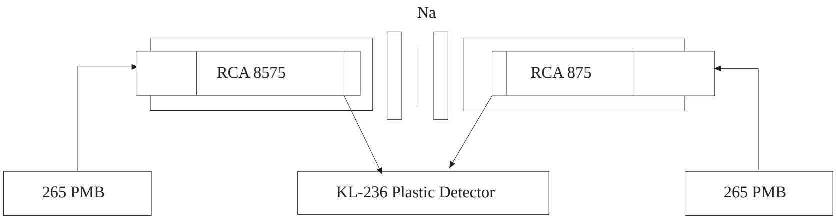


**Figure 1.** Schematic diagram of the experimental arrangement.

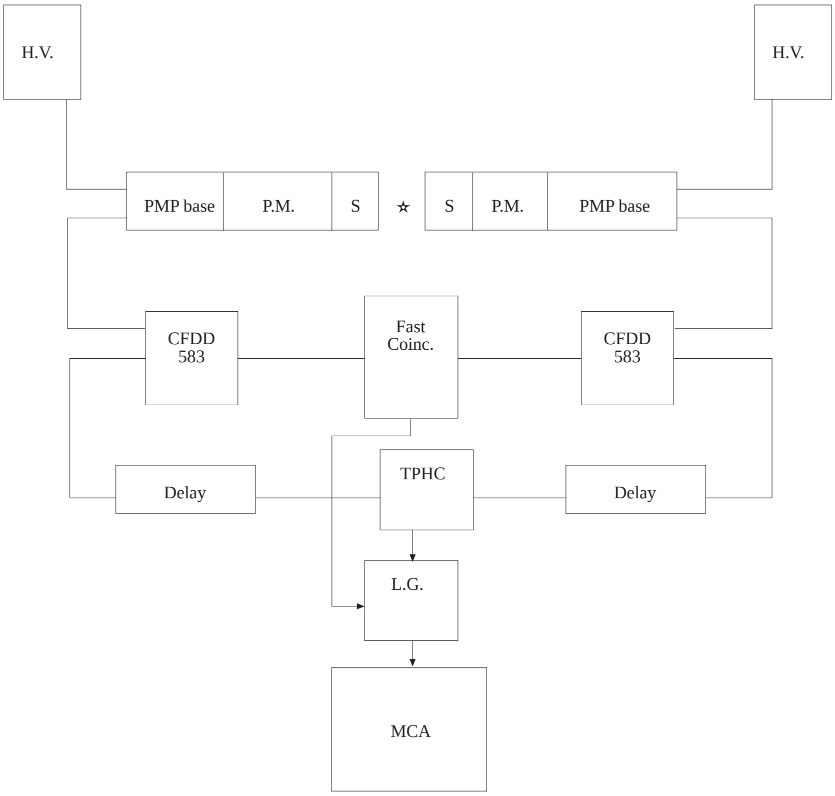


**Figure 2.** Block diagram of the position lifetime measurement system.

## 3. Results and Discussion

### 3.1. Positron Annihilation Studied

The mean lifetime $\tau$ is given by $(\tau_f I_f + \tau_t I_t)$, where $\tau_f$ and $\tau_t$ are the free and trapped lifetime, and $I_f$ and $I_t$ are their respective intensities. The mean lifetime values (from the average of three measurements) as obtained experimentally is plotted in Fig. (3) as a function of the Mg concentration. The experimental data showed a good fitting with Sigmodel (solid line) from which the trapped lifetime $\tau_t$ and free lifetime $\tau_f$ were determined as 163.900 ps and 180.664 ps, respectively. Such values, obtained for AlMg$_x$, are in a good agreement with Boileau et al. [18] and support the assumption that a positron does not more favorably become localized in the vicinity of a subsitutional Mg-atom than of an Al-atom.

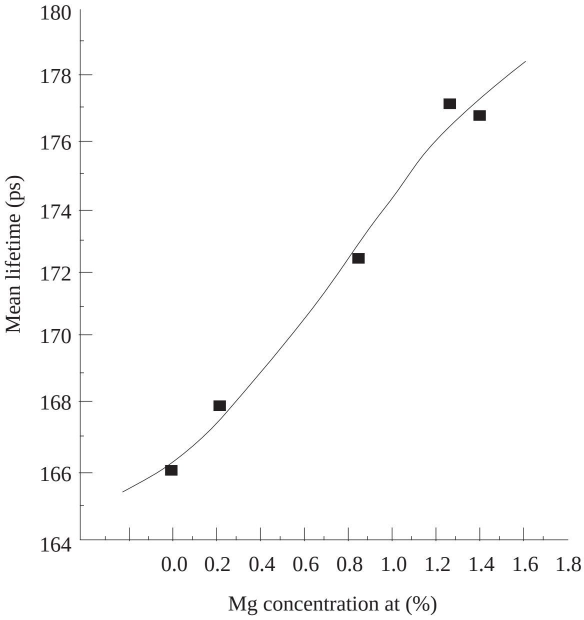


**Figure 3.** Positron annihilation mean lifetime as a function of Mg concentration

It is due to Hidalgo and de Diego [19] that the grain size $\ell$ and the mean lifetime $\tau$ is related to $\tau_f$ and $\tau_t$ by a simplified model which leads to the relationship.

$$\tau = \tau_f + [(\tau_t - \tau_f)\frac{L_d}{\ell}] \tag{3.1}$$

where L$_d$ is the mean diffusion length of positron in metal (Al) : $L_d$=1500 $\mathring{A}$ . By substituting for L$_d$ , $\tau_t$ and $\tau_f$ in the above equation, the grain size was calculated.

Figure (4) represents the relation of the grain size as a function of Mg concentration. A decrease of the grain size from 1.2 $\mu$m to 0.2 $\mu$m is observed as the Mg-concentration increases up to x=1.4 at%.

Figure (5) represents the value of the mean lifetime as a function of degree of deformation for pure Al and its alloy samples of Mg impurities at 1.4 at%. It shows that the mean lifetime increase as indication of the decrease of grain size. The dependence of the grain size on the degree of deformation is depicted in Fig. (6), which shows also an exponential decrease.

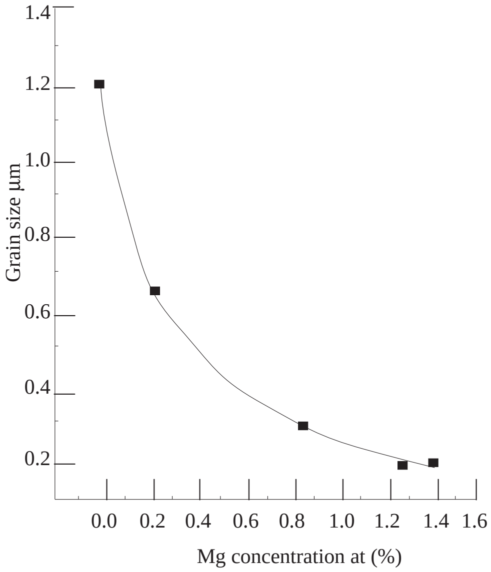


**Figure 4.** Grain size as a function of Mg concentration

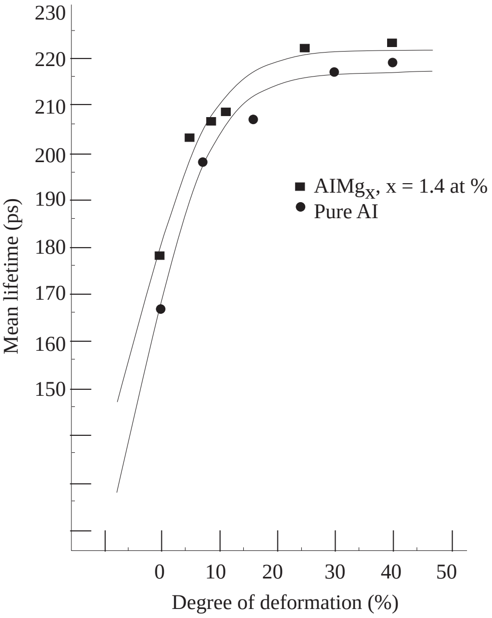


**Figure 5.** Positron annihilation mean lifetime as a function of degree of deformation (%)

### 3.2. X-Ray Diffraction Studies

X-ray diffractograms of AlMg$_x$ sample (x=0.7) at different degree of plastic deformation (0- 40 %) are shown in Fig. (7). The main features of the diffraction patterns are the same, but only a considerable variation of the peak intensity is observed. On the other hand , no peaks of free Mg was revealed. The diffraction peaks at $2\theta = 34.7, 38.5, 44.8, 65.2, 69.5, 78.1, 82.3, 112$ and $116.5°$ respectively, correspond to (110), (111), (200), (220), (300), (311), (222), (331) and (420) planes of face center cubic aluminum as confirmed by JCPDS X-ray powder file data (4-787). The average value $a_o$ of the calculated lattice parameters equals 4.05 $\AA$ in agreement with the references. However, in all cases the intensities of (111) and (200) were extremely high in comparison with the other reflections, indicating they are the preferential orientation of the microcrystalites. The results showed that the preferred orientation became observable at degree of deformation $> 32$ %, after which the peak intensity increases with increasing deformation. The degree of such a preferred orientation was found to decrease with increasing the deformation upto 32 %, then increases with deformation as proved by the variation of the intensity ratio $I_{(200)}$ / $I_{(111)}$, illustrated in Fig. (8).

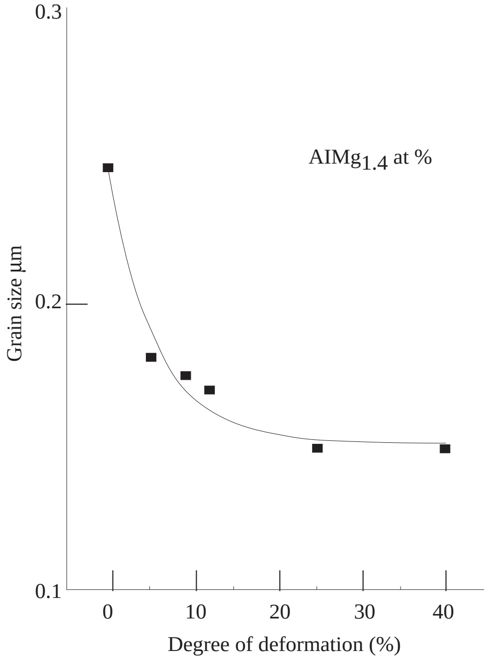


**Figure 6.** Grain size as a function of degree of deformation (%)

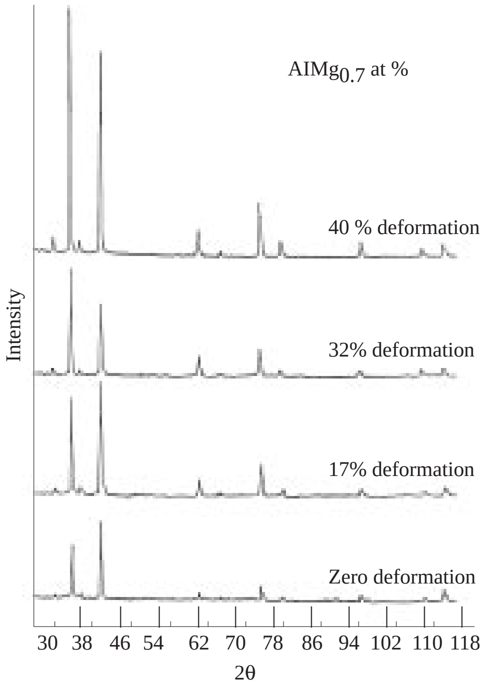
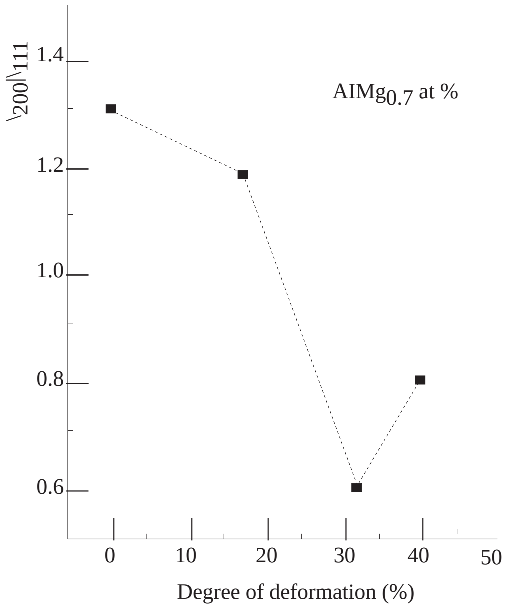


**Figure 7.** XRD chart as a function of degree of deformation (%)

Figure (9). Show the diffractograms of the $AlMg_x$ (x = 0.7 at %) for different annealing time at constant temperature of 773 K. Increasing the annealing time did not create new phases. The overall intensity of the reflections decreases with increasing the annealing time.



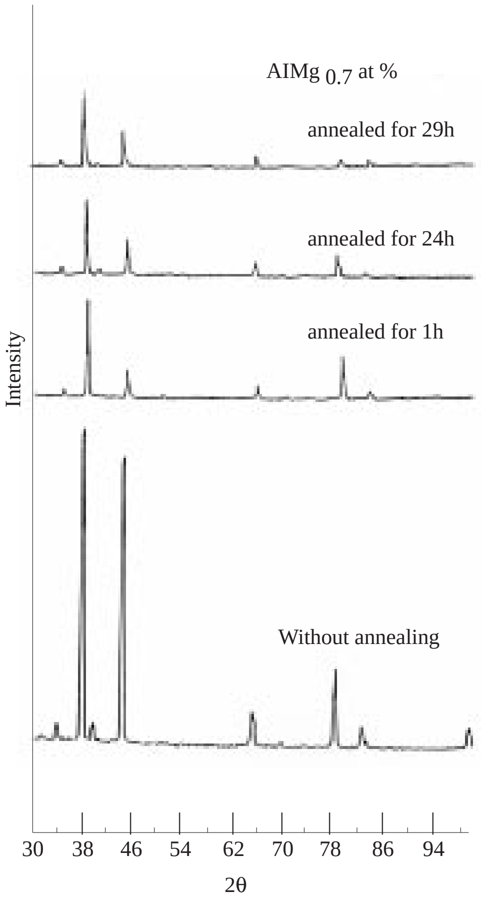


**Figure 8.** Relative intensity as a function of degree of deformation (%)

**Figure 9.** XRD chart as a function of annealing time

## 4. Conclusion

The positron annihilation parameters are found to be a sensitive function of grain size, indicating positron annihilation as a powerful technique for detecting low concentrations in metal alloys. Recrystallization does not always return the sample to its original condition in one respect. The material may differ in the way its grains lie in the material. On the other hand, if the deformation is continued until the reduction is 95 % or more, the grains in the recrystallized sample will not be oriented in the random manner that existed before deformation.

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