DEFECT INVESTIGATION OF PLASTICALLY DEFORMED AL 5454 WROUGHT ALLOY USING PADBS AND ELECTRICAL MEASUREMENTS

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Abstract

Positron Annihilation Doppler Broadening Spectroscopy (PADPS) is a non-destructive technique used in material science. Electrical measurements are one of the oldest techniques used also in material science. This paper aimed to discuss the availability of using both PADPS and electrical measurements as diagnostic techniques to detect the defects in a set of plastically deformed 5454 wrought aluminum alloy. The results of the positron annihilation measurements and the electrical measurements were analyzed in terms of the two-state trapping model. This model can be used to investigate both defect and dislocation densities of the samples under investigation. Results obtained by both nuclear and electrical techniques have been reported.

Key words:
Positron annihilation Doppler broadening spectroscopy; electrical measurements; Al 5454 alloy; defect concentration; dislocation density

1. Introduction

Wrought Al 5454 alloy are used for many applications including building industry, Chemical and food industries (storage tanks and pipes). It is also used as equipment for heating and cooling (heat exchangers, air condition evaporators). In addition it is used for home appliances, Furniture, and for heat insulation. The chemical composition of Al 5454 alloy is shown in Table (1).

Element	Cr	Cu	Ti	Zn	Si	Fe	Mn	Mg	Al
Al 5454	0.05-0.2	0.1	o.2	0.25	0.25	0.4	0.5-1	2.4-3	Balance

Table (1): The chemical composition of Al 5454 wrought alloy.

Al 5454 Alloy have very good resistance to atmospheric corrosion, very good weld ability and good formability by pressing, drawing and roll forming. Series alloys provide a wide range of mechanical properties. In sheet form, the strength/formability characteristic is achieved by application of various degrees of strain hardening and in some cases by intermediate annealing.

The positron annihilation technique is one of the nuclear methods used in material science. This technique now plays an important role in defect spectroscopy and electron band structure and much attention has been devoted to the interaction of positron with defects in solids. Observations showed that positrons can become trapped at imperfect locations in solids and their mean lifetime can be influenced by changes of concentrations of such defects [1-5]. The advantages of this method are in the obtaining of both qualitative and quantitative data on defect behavior in materials. To explain this method several groups [6-8] proposed a phenomenological trapping model. The main assumption of the simple trapping model is that positrons annihilate in solid materials from a free or trapped state.

The Doppler effect is the apparent change in the observed frequency of a wave as a result of the relative motion between the source and the observer. This effect was suggested by Doppler (1883-1955), as an attempt to explain the coloration of stars. The motion of the electron-positron pair causes a Doppler shift on the energy of the annihilation radiation. As a consequence, the line-shape gives the distribution of the longitudinal momentum component of the annihilating pair. The definition of the Doppler broadening line-shape S- and W-parameters is described elsewhere [9-11]. The present paper represents the change of the positron annihilation line-shape parameters and resistivity measurements on a set of plastically deformed Al 5454 alloy at room temperature. The variation of defect and dislocation densities of these samples obtained by both nuclear and electrical measurements has been studied.

2. Experimental Procedures:

2-a) Setup of Positron Annihilation Doppler broadening Technique (PADBT):

The system which has been used in the present work to determine the Doppler broadening line-shape is consists of an Ortec HPGe detector with an energy resolution of 1.95 keV for 1.33 MeV line of ^{60}Co, an Ortec 5 kV bais supply 659, Ortec amplifier 575 and trump 8 k MCA. Figure (1), shows a schematic diagram of the experimental arrangement. Doppler broadening is caused by the distribution of the velocity of the annihilating electrons in the directions of gamma ray emission. The signal coming from the detector enters the input of the preamplifier and the output from the preamplifier is fed to the amplifier. The input signal is a negative signal. The output signal from the amplifier is fed to a computerized MCA.

The positron source of 1mCi free-carrier ^{22}NaCl, was evaporated from an aqueous solution of sodium chloride and deposited on a thin Kapton foil of 7.5 μm in thickness. Radioactive material can be deposited directly on the samples or separation foils can be used to allow using the source repeatedly. The source can also consist of a single radioactive metal foil. The source has to be very thin so that only small fractions of the positron annihilate in the source. A sandwich configuration has been used to guide the positron into the Al 5454 samples. The source-samples configuration was then wrapped in a thin aluminum foil. Each sample spectrum was measured for 1800 seconds.

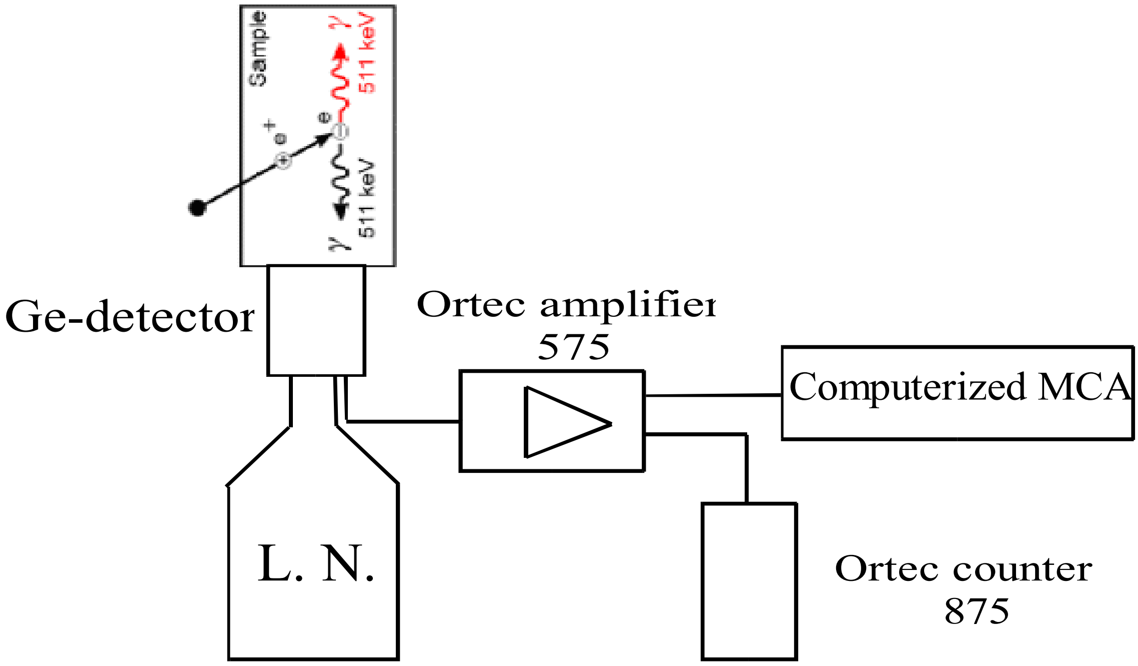


Fig. (1) Experimental setup for Doppler broadening line-shape measurements.

2-b) Electrical measurements:

The electrical resistivity, ρ, of the samples was measured by the two-point probe method. The electrodes are brought into contact with the sample surface. A known current is passed through the electrodes, while the voltage reading is made between the surfaces of the sample. The potential difference, V and current, I, were determined using a digital multimeter. The electrical resistivity, ρ, of the samples was calculated according to [12-14]: where d is the thickness of the sample and R_s is the sheet resistance of the sample, which is given by:

$$\rho = R_s\, d$$

$$R_s = V / I$$

3. Results and Discussions:

The Al 5454 alloy specimens were prepared for both Doppler broadening and electrical measurements to have thickness around 2 mm. These samples under investigation has been electrochemically cleaned then dried at room temperature. The samples were then annealed at 450 °C for 12 hours. These samples are then plastically deformed in the range of 1.3 % to 13.4 % degree of deformation.

The Doppler broadening line shape parameters were measured for deformed and non-deformed samples of Al 5454 alloy. Two spectrums without and with 13.4 % degree of deformation considered samples at 511 keV of annihilation are shown in figure (2). The measured line shape profile of 13.4 % degree of deformation sample reveals a little higher line shape counts than non-deformed sample. The Full Width at Half Maximum (FWHM) is the same for both samples.

The analyses of the line-shape spectra were made using the SP-01 program [15]. The S and W line-shape parameters reflect the characteristics of the defect formation that trap positrons. The dominant change in values of S-parameter as a function of thickness reduction is shown in figure (3). In such figure, the S- parameter increases with increasing of thickness reduction of the sample. For higher thickness reduction (Above approximately 4%), the values of S- parameter is approximately kept constant.

Using the measured value of S-Parameter and the obtained fitted values S_f and S_t from the solid line in figure (3), the trapping rate (K_t) can be calculated from the two state trapping model described in equation (1) [6, 7]. This model assumes that the positron exists in one of only two states in the materials, the free or Bloch state and the defect trapped state.

where S_f ($S_f = 0.4840 \pm 0.00074$) is the S-parameter for an annealed specimen and S_t ($S_t = 0.4875 \pm 0.00073$) is the S-parameter of dislocation saturated sample.

$$S = S_f \left[\frac{1 + K_t S_t}{1 + K_t S_f} \right] \tag{1}$$

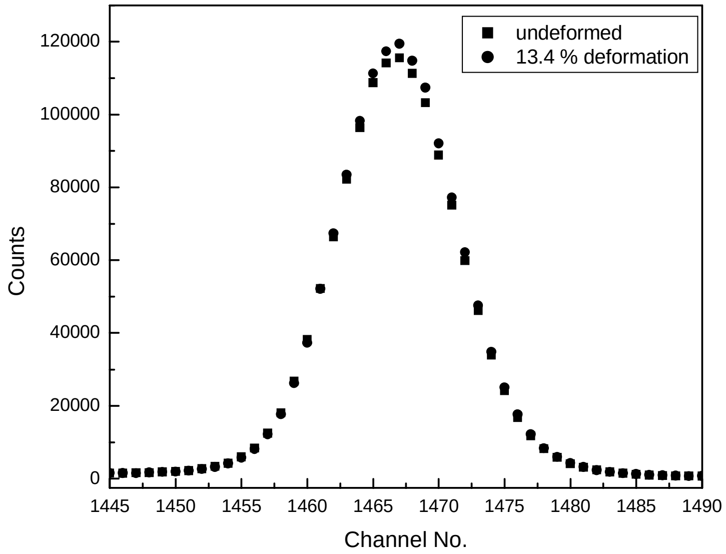


Fig. (2) The Doppler broadening line-shape spectrum for Al 5454 wrought alloy, without and with 13.4 % degree of deformation.

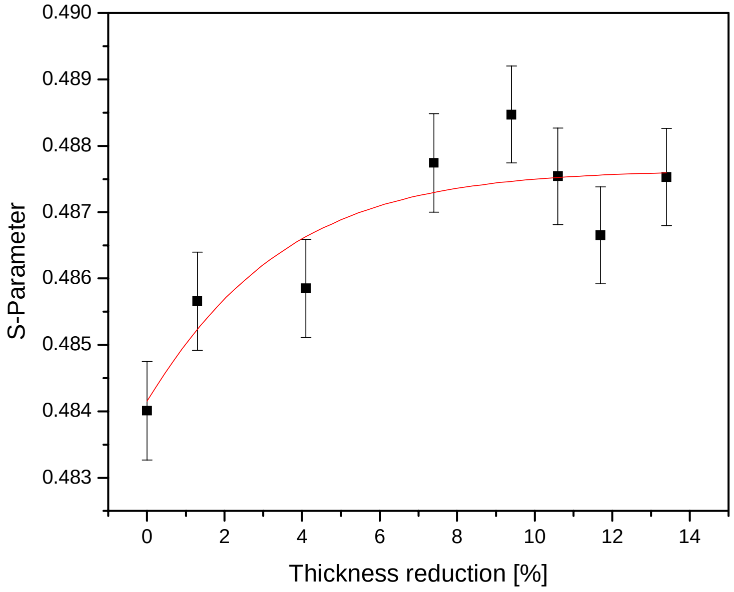


Fig. (3) S-parameter as a function of thickness reduction for 5454 Al-alloy.

The trapping rate is related to the trapping efficiency υ as follows:

$$K_t = (1.248)\times 10^{-3}\left[Log\,(1-r)\right]^2 \frac{\upsilon}{b^3} \qquad (2)$$

where r is the fractional thickness reduction and b is Burger vector of 5454 alloy that is estimated to be equal to 2.8598 Å.

The Trapping Efficiency (υ) is related to the trapping cross-section (σ) according to following equation:

where V is the mean thermal velocity of the thermalized positron and from the Maxwellian

$$\upsilon = \sigma V \qquad (3)$$

$$V = \sqrt{\frac{8K_B T}{\pi m}} \qquad (4)$$

distribution one obtain:

where K_B, T and m are Boltzman constant, temperature and mass of the positron respectively. The trapping Rate (K_t) is proportional to defect density $\rho^{/}$ (cm^{-3}) as follows:

In this equation, we have defined a defect density as the number of trapping sites per unit

$$K_t = \nu\,\rho^{/} \qquad (5)$$

$$\rho^{/} = \frac{\rho}{b} \qquad (6)$$

volume. Dislocation density is generally expressed as length per unit volume, ρ (cm^{-2}) [4, 16]. The defects density $\rho^{/}$ (cm^{-3}) is related to dislocation density ρ (cm^{-2}) according to the following equation:
where b is Burger vector in cm.

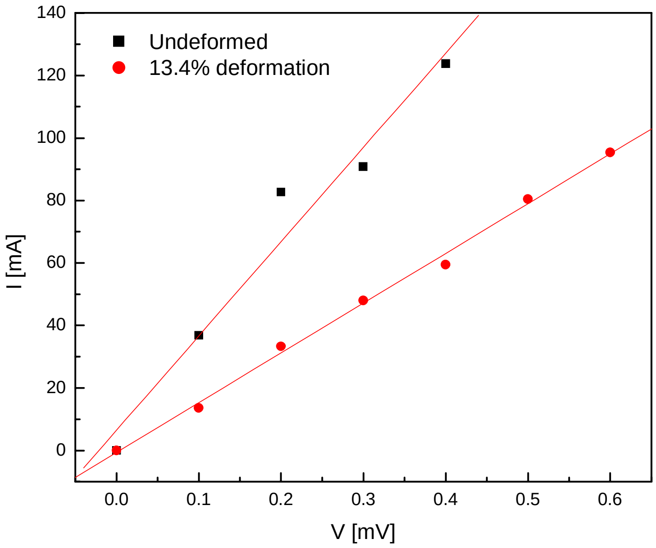
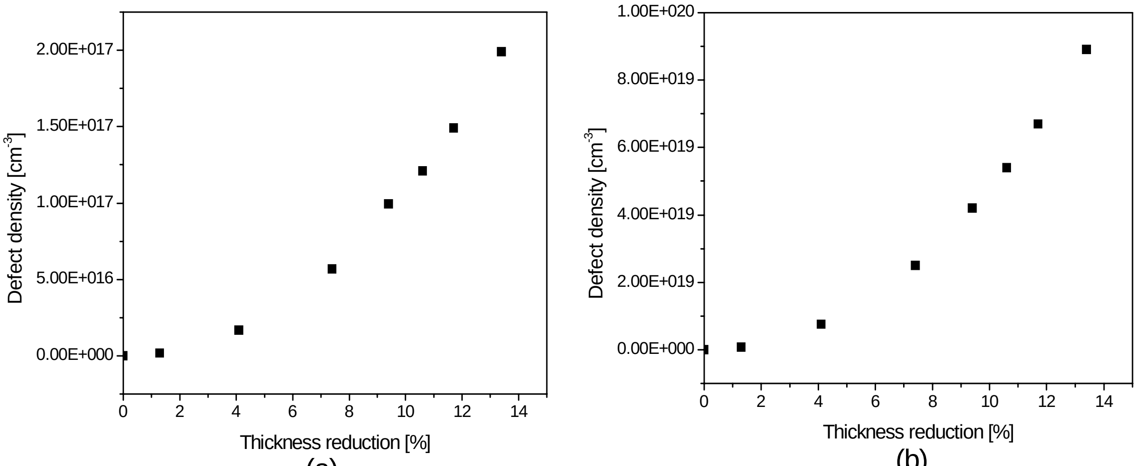


Fig. (4) I-V curve for Al 5454 wrought alloy,
without and with 13.4 % degree of deformation.

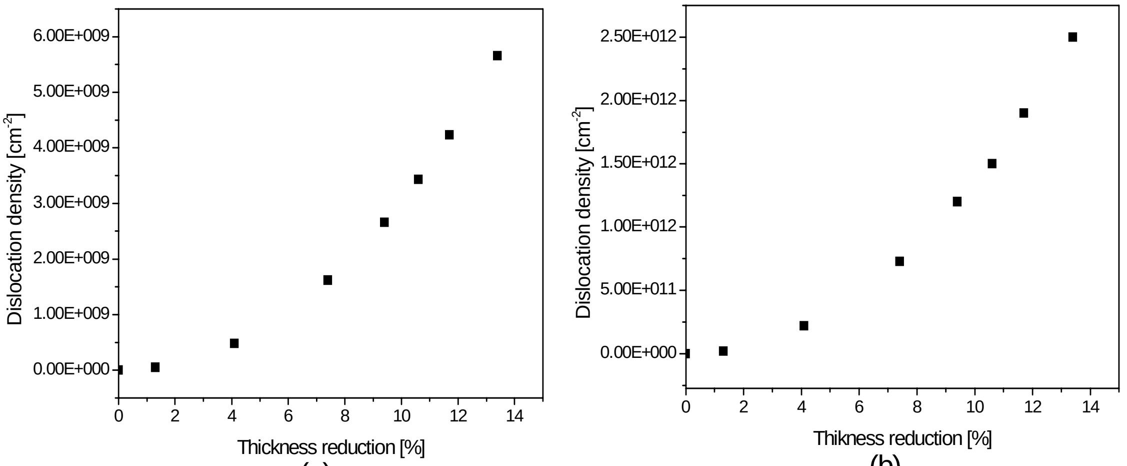
The results of the PADBS have been compared with results obtained from electrical measurements. In the electrical measurements the I-V values for all samples has been measured. Figure (4) reveals the I-V curves for undeformed and deformed sample at 13.4 % degrees of deformation. The resistivity has been calculated from the slope of the lines shown in such figure. The resistivity of all samples has been calculated also by the same way.

Using the measured value of resistivity (ρ) and the obtained fitted values ρ_f (the resistivity of the annealed sample) and ρ_t (the resistivity of the dislocation saturated sample) instead of S, S_f and S_t, the trapping rate (K_t) calculated from electrical measurements can be determined from equation (1). Both defect and dislocation densities can be calculated as previously described in equations (5) and (6) using the trapping rate calculated from electrical measurements.



(a) (b)

Fig. (5) Defect density as a function of thickness reduction using PADBS (a) and resistively (b).



(a) (b)

Fig. (6) Dislocation density using PADPS (a) and resistively (b).

The concentration of the defect (defect density) as a function of thickness reduction for both results obtained by PADBS and electrical measurements are shown in figures 5 (a) and 5 (b) respectively. It is clear that both results obtained by PADBS and electrical measurements reveal the same behavior. The defect density is increasing exponentially with increasing of thickness reduction. A small increase in defect density is obtained with increasing the degree of deformation up to approximately 4 % degree of deformation is reached, while a fast increase of defect density is obtained afterwards. At maximum thickness reduction (13.4 %), a higher defect density value of about $9x10^{19}$ cm^{-3} is obtained by electrical measurements, while value of about $2x10^{17}$ cm^{-3} is reached for PADBS measurements.

The dislocation density as a function of thickness reduction for both results obtained by PADBS and electrical measurements are drawn in figures 6 (a) and 6 (b) respectively. Results of both PADBS and electrical measurements behave the same way as defect densities shown in figures 5 (a) and 5 (b). At maximum thickness reduction (13.4 %), maximum dislocation density of about $2.5x10^{12}$ cm^{-2} is obtained by electrical measurements, while value of about $6x10^{9}$ cm^{-2} is reached for PADBS measurements. The difference in results of defect and dislocation densities obtained by PADBS and electrical measurements could be related to the experimental errors during measuring VI values of samples under investigation. Results of the positron annihilation have proven that this technique is a powerful tool for detecting defects in Al-alloys [16-21].

4. Conclusions:

Nuclear and Electrical techniques were used to study the defects in Al 5454 wrought alloy. The Doppler broadening line-shape S- parameter increases with increasing of thickness reduction of 5454 wrought alloy. Above 4% of thickness reduction, the S-parameter values kept approximately constant. Results of both defect and dislocation densities obtained by PADBS and electrical measurements reveal the same behavior. A higher defect and dislocation densities values are obtained by electrical measurements, while smaller values are reached for PADBS measurements at maximum thickness reduction (13.4 %). Due to the expected experimental error during measuring the I-V values, the calculated values of both defect and dislocation densities are much accurate using the Doppler broadening line-shape measurements.

References

[1] I K Mackenzie, T L Khoo, A B McDonald and B T A McKee, Phys. Rev. Lett. 19 (1967) 946.
[2] P Hautojarvi, "positron in solids" Topics in current physics, Springer-verlag, Berlin, Germany 12 (1979).
[3] E A Badawi, M A Abdel-Rahman and S A Mahmoud, Appl. Surf. Sci. 149 (1999) 211.
[4] C Dauwe, M Dorikens, L Dorikens-Vanpraet and D Segers, Appl. phys. 5 (1974) 117.
[5] M A Abdel-Rahman and E A Badawi, jpn. J. Appl. Phys. 35 (1996) 4827.
[6] B Bergersen and M J Stott, solid state Commun. 7 (1969) 1203.
[7] D C Connors and R N West, phys. Letters A 30 (1969) 24.
[8] W Brandt, "Proc. International Conference on positron annihilation" Eds. A T Steewart and L O Roelling, Academic Press, New York, USA (1976) 80.
[9] I K Mackenzie, J A Eady and R R Gingerich, Phys. Lett. 33 A (1970) 279.
[10] M A Abdel-Rahman, M Abdel-Rahman, M Abo-Elsoud, M F Eissa, Y A Lotfy and E A Badawi, Progress in Physics 3 (2006) 91.
[11] M Abdel-Rahman, M Abo-Elsoud, M F Eissa, N A Kamel, Y A Lotfy and E A Badawi E A, Progress in Physics 3 (2006) 66.

[12] L J Van der Pauw, Philips Res. Rep. 13 (1958) 1.
[13] A A Ramadan, R D Gould and A Ashour, Thin Solid Films 239 (1994) 272.
[14] A Ashour, E Badawi and N Z El-Sayed, Surface Review and letters 13 (1) (2006) 69.
[15] http://www.ifj.edu.pl/~mdryzek
[16] J Baram and M Rosen, phys. Stat. sol. (a) 16 (1973) 263.
[17] G Dlubek, H Krause, R Unger and W Heyroth, phys. stat. sol. (a) 169 (1998) R11.
[18] E A Badawi, Surface Review and Letters 11 (2) (2004) 167.
[19] G Dlubek, O Brummer and E Hensel, phys. Stat. Sol. (a) 34 (1976) 737.
[20] M A Abdel-Rahman, Emad A Badawi and M S Abdallah, Review Letters 11 (4) 2004.
[21] R Ferragut and A Somoza, Phys. Stat. Sol. (a) 175 (1999) R1.