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Monitoring of Defects Concentration in Deformed Aluminum Using Doppler Broadening Technique

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Abstract

Doppler broadening of the 511 keV positron annihilation line was used to estimate the concentration of defects at different deformation levels of pure aluminum samples. These samples were compressed at room temperature to 15, 22, 28, 38, 40, and 75 % thickness reduction. The two-state positron-trapping model has been employed. The S and W lineshape parameters were measured using high-resolution gamma spectrometer with high pure germanium detector of 2.1 keV resolution at 1.33 MeV of ^{60}Co. The change of defects concentration (C_D) with the deformation level (ε) is found to obey an empirical formula of the form $C_D = A\,\varepsilon^{B}$ where A and B are positive constants that depend mainly on the deformation procedure and the temperature at which the deformation takes place.

Introduction

Positron annihilation techniques have recently been proved to be useful in the investigation of defects in materials [1]. The microstructure properties of these materials can be explained in terms of defects type and their concentration. The positron annihilation spectroscopy techniques can yield two types of information; the electron density and the electron momentum distribution, and this is because of the ability of positrons to form a variety of electronic states in solids. The use of the Doppler broadening of the 511 keV positron annihilation line to obtain the momentum distribution of the annihilating quanta has the advantage over conventional techniques. That is

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because it requires a very weak positron source, essentially no radiation damage is suffered by the samples, the simplicity of the system used to measure the shape of the Doppler-broadened annihilation line [2] and its improved statistics due to the fast data accumulation. The Doppler broadening spectroscopy (DBS) can be explained as follows: The entire energy of an annihilating positron-electron pair includes the energy of their rest masses (2 x 511 keV) and their kinetic energies. The latter causes an energy shift of the annihilation line; the wavelength of the annihilation radiation becomes shorter along the direction of the motion and longer in the opposite direction. The observation of many annihilation events leads to Doppler-broadened annihilation line. In most cases the positron loses its kinetic energy in a very short time due to the slowing down process in matter. Therefore, the Doppler-broadened line corresponds to the momentum distribution of the annihilating electrons. Electrons with high momentum cause a broad Doppler line. In order to estimate the concentration of defect by the two-state trapping model, the lineshape parameters S and W are introduced [3]. The central parameter S is defined as the ratio of the counts in the central region of the annihilation line to the total number of the counts in the line. The wing parameter W is the relative fraction of the counts in the wing regions of the line with respect to the total number of the counts in the line. The relative sensitivity of the different parameters was studied by Campbell [4]. DBS was previously used as a microstructural probe for different materials, including metals [5-10], alloys [11], semiconductors [12,13] ionic crystals [14,15] superconductors [16] and polymers [17]. An up-to-date review is reported elsewhere [18]. In the present work, the concentration of defects in highly deformed Al samples are to be monitored using DBS at different deformation levels. This investigation is carried out in order to examine the potential of DBS as a probe for quantitative analysis of defects.

Experimental and Data Analysis

The ^{22}Na positron source of about 1.2 μCi was prepared by evaporating carrier-free ^{22}NaCl solution between two 3 μm aluminum foils. A sheet of polycrystalline Al of 99.99 % purity was cut into a set of identical $1.6 \times 25 \times 25$ mm^3 samples. All samples were annealed at 350 ^{o}C for 4 hrs. One pair was left as a reference, while the others were defected for different levels (15, 22, 28, 38, 40, and 75 %) by the action of plastic deformation employed by compression. The deformation levels were taken in terms of percentage reduction in thickness. The lineshape measurements were carried out using our Doppler broadening spectrometer (Fig.1), which includes a TENNLEC high pure germanium (HpGe) coaxial detector. Its energy resolution is 2.1 keV at 1332 keV line of ^{60}Co while its relative efficiency is 20%. The preamplifier is built in the detector assembly. The remaining NIMs are ORTEC 572 amplifier, ORTEC 444 gated biased amplifier, Norland 5300 multichannel analyzer (MCA), and an ORTEC 459 high-stability power supply. The measured spectra were analyzed by DBSFIT program (especially written for the present project), which defines the peak count parameters S and W after applying various necessary corrections on the 511 keV lineshape.

Results and Discussion

By employing the two-state trapping model, the S parameter is related to the positron trapping rate (K) through the expression [1]:-

$$K = \frac{S - S_B}{S_D - S} \lambda_B \quad ....(1)$$

where S_B and S_D are the S parameter at the defect-free bulk and at the defect-trapped states respectively. λ_B is the annihilation rate in the bulk state and equals 0.0062 ps^{-1} for Al [19].

The positron trapping rate (K) is defined as the probability per unit time that a positron will be trapped in a defect, and is proportional to the concentration of this defect (C_D) as :

$$K = \mu_D C_D \quad .....(2)$$

where μ_D is the specific positron trapping rate at these defects, which equals 158.5 ps^{-1} [20]. By plotting the S parameter as a function of deformation level (Fig. 2), one can notice that the S parameter increases with the deformation level, reaching almost a saturation value at the end. This is because when deformation occurs, the density of valence electrons is reduced due to generation processes of defects like dislocation. Fig. (3) shows that the W parameter decreases with increasing deformation level. This behavior suggests that a decreasing of positrons overlap with core electrons is occurred. The S_D parameter is determined by plotting the measured S parameter as a function of the measured W parameter, as shown in Fig. (4). The extrapolation of the straight line crosses the S-axis at a certain value which represents S_D. Now, by employing Eqs. 1 and 2, the trapping rate K and thereby the concentration of defects (C_D) was estimated at any deformation level. Fig. (5) illustrates the variation of C_D in ppm with the deformation level (ε). The concentration of defects increases rapidly as the deformation level increases. A significant number of dislocations is formed during deformation. We found that this behavior obeys the following empirical equation: $C_D = 0.0093\,\varepsilon^{2.3465}$

for $0 < \varepsilon < 100$ %, ... (3)

Hence, direct estimation of defect's concentration is possible at any deformation stage.

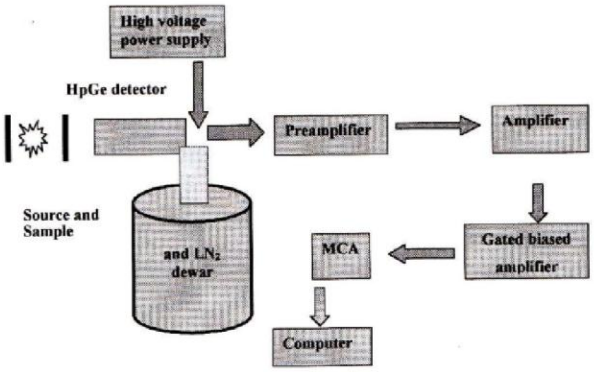

Fig. 1: A block diagram of the high resolution gamma spetrometer used for monitoring the shape of Doppler broadening annihilation line.

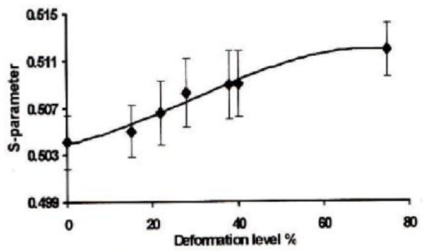

Fig. 2: The change in S- parameter with the deformation level

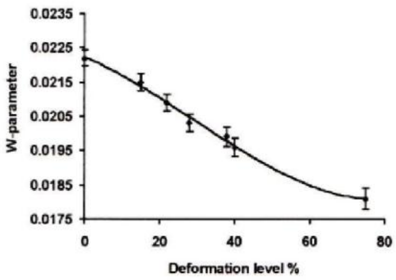

Fig. 3: The change in W- parameter with the deformation level

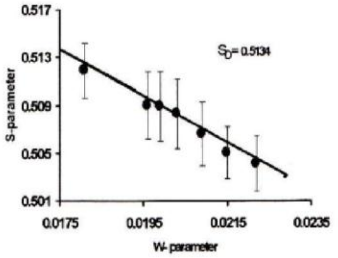

Fig. 4: The S−W plot that used to determine the S- parameter at defective regions

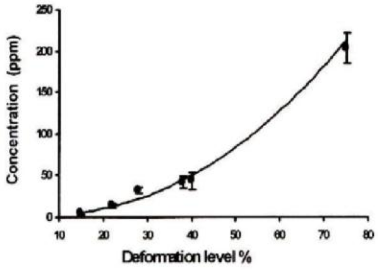

Fig. 5: The change in defects concentration with the deformation level of Al samples.

Conclusion

The present results demonstrate the strong dependence of the Doppler broadening parameters on the deformation level. The concentration of defects (C_D) in Al depends on the deformation level (ε). It follows an empirical formula of the form $C_D = A\ \varepsilon^B$ where A and B are fitting constants having positive values depend on the sample history, the deformation mode and the temperature at which the deformation takes place. The Doppler broadening technique can, besides of its simplicity, be used as a good tool to monitor and estimate the concentration of defects in deformed metals in ppm scale.

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مراقبة تراكيز العيوب في الألمنيوم المشوه بأستخدام تقانة توسع دوبلر

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الخلاصة

يتضمن البحث أستخدام تقانة توسع دوبلر لخط طاقة فناء البوزترون في تحديد تراكيز العيوب المجهرية في عينات من الألمنيوم المشوه وعند مستويات تشويه مختلفة . وأستخدم لقياس معلمات شكل دوبلر كاشف الجرمانيوم عالي النقاوة ضمن منظومة كشف وتحليل أطياف كاما وذلك للحصول على قدرة تمييز عالية للطاقة . أظهرت النتائج ان تغير التركيز C_D مع مستوى التشويه ε يخضع لعلاقة تجريبية بهيئة $C_D = A\,\varepsilon^B$ حيث A و B ثوابت موجبة تعتمد في الأساس على طريقة التشويه ودرجة الحرارة أثناء عملية التشويه .