JMEPEG (2003) 12:190-195

A Study on Commercial Pure Al (1050) After Cold Rolling at Room Temperature With Various Deformations Using Positron Annihilation

E. Gomaa, N. Mostafa, M. Mohsen, and A.S. Taha

(Submitted 20 November 2002)

Positron annihilation lifetime (PAL), Doppler broadening (DB), and Vickers microhardness measurements (Hv) were performed to study the isochronal annealing of commercial pure Al (1050) in the temperature range from room temperature (RT) to 823 K after cold rolling at RT with various deformations of 16%, 37%, and 54%. PAL measurements show a decrease in the average lifetime of positron (τ_{av}) followed by saturation at 190 ps as a function of annealing temperature. The results of Doppler broadening of annihilation radiation (*S* and *W* parameters) are found to be consistent with PAL measurements. The correlation between the characteristics of positron annihilation and Hv shows three annealing stages of microstructure in commercial Al, which are attributed to recovery, partial recrystallization, and complete recrystallization.

Keywords Al (1050), cold rolling, Doppler broadening, positron annihilation lifetime, recovery, recrystallization, Vickers microhardness

1. Introduction

Deformation and annealing are important processing methods for producing desired properties of materials by controlling their microstructure.[1] Deformation generates some other defects such as grain boundaries, vacancies, and small vacancy clusters in addition to many types of dislocations.

The sensitivity of positron annihilation spectroscopy to dislocations, vacancies, and vacancy clusters has been used in many experiments to investigate the nature of the annealing stages in Al.[2,3] On the other hand, positron lifetime measurements have been conducted to study how the positron trapping efficiency of dislocations in Al 99.999% purity changes during isochronal annealing after deformation at room temperature (RT).[4] The combination of positron annihilation lifetime (PAL) and Doppler broadening of annihilation radiation (DBAR) provides information on the momentum distribution of the annihilation electrons.[5] The two techniques have been applied to study the recovery stages of commercial pure Al and Al-0.9 wt.% Si alloys.[6] Further DBAR measurements were performed on commercial pure Al (99.5%) to evaluate the microhardness variations during isochronal annealing.[7]

The aim of the present work is to study the annealing stages during isochronal annealing of commercial pure Al (1050), (99.5% purity) and various amounts of cold rolling leading to 16%, 37%, and 54% thickness reduction over the temperature interval 300-823 K using the PAL technique, DBAR, and Vickers microhardness (Hv). A correlation is undertaken between the microstructure defect aspects given by PAL, DBAR, and the characterization of the material through the measurements of Hv.

E. Gomaa, N. Mostafa, and **M. Mohsen,** Physics Department, Faculty of Education, Ain Shams University, Cairo, Egypt; and **A.S. Taha,** Metallurgy Department, Atomic Energy Authority, Cairo, Egypt. Contact e-mail: ehsannemat@yahoo.com.

Table 1 Chemical Composition (wt.%) of Commercial Pure Al (1050)

Material	Mn	Mg	Fe	Si	Ti	Cu	Ni	Al
Al (1050)	0.004	0.008	0.236	0.120	0.028	0.020	0.030	Balance

2. Experimental Details

2.1 Sample Preparation and Chemical Composition

The investigated samples were supplied by military factory 63 (Cairo, Egypt). These samples were commercial pure Al-99.5% (1050). The as-received samples (the starting material) were annealed at 773 K for 6 h and cooled down to RT in the furnace (type MLW Model LM 3211, Nuclear-Solid State Lab., Physics Dept., Ain Shams University, Cairo, Egypt) to be fully recrystallized.

The deformation was accomplished at RT by rolling to various extents of thickness reduction: (16%, 37%, and 54%). After deformation, the samples were cut into a square with sides of 1 cm and polished by abrasives of grades 400, 600, 800, 1000, 1200. Isochronal annealing of the specimens was made in air in steps of 323 K with a hold time of 1 h at each annealing step, from RT to 823 K with accuracy ±3. The cooling of the samples to RT was performed slowly in the furnace to avoid quenching effects.

The samples were chemically analyzed by an optical emission spectrometer (BAIRD Model DV 6, USA). The chemical compositions of the samples are given in Table 1.

2.2 Positron Annihilation Lifetime Measurements

The PAL spectrometer was a fast-fast timing coincidence system with a time resolution full-width at half maximum

(FWHM) of 230 ps. A 20 microcurie (μCi) source of ^{22}Na Cl deposited on a thin Kapton foil (7 μm) was sandwiched between two identical specimens (1 × 1 cm). For each spectrum, more than one million counts were accumulated. After source and background corrections, the lifetime spectra were fitted to a sum of exponential decay components convoluted with the Gaussian resolution function of the spectrometer.

By using the computer program PATFIT (Riso Nat. Lab., Denmark), the lifetime spectra were analyzed into two components. The component characterized by the lifetime τ_1 with intensity I_1 represents the positron annihilating in monovacancies and dislocations. The lifetime τ_2 with intensity I_2 corresponds to positrons trapped and annihilating in three-dimensional vacancies and vacancy clusters.

2.3 Doppler Broadening Annihilation Radiation Measurements

The DBAR measurements were measured using a hyper-pure germanium detector. The measured FWHM was established to be 1.2 KeV at 662 KeV ^{137}Cs. The energy dispersion of the equipment was 0.049 KeV per channel. The number of channels included in the annihilation peak area was 300. The same positron source as mentioned before for PAL measurements was used and sandwiched between two identical pieces of the sample. The total number of counts in the measured spectrum is about 10^7. The line shaped, *S*-parameter was measured as the number of counts lying within an energy interval of 1.3 KeV centered at the peak of the annihilation line. The wing parameter *W* is the sum of counts lying within the energy

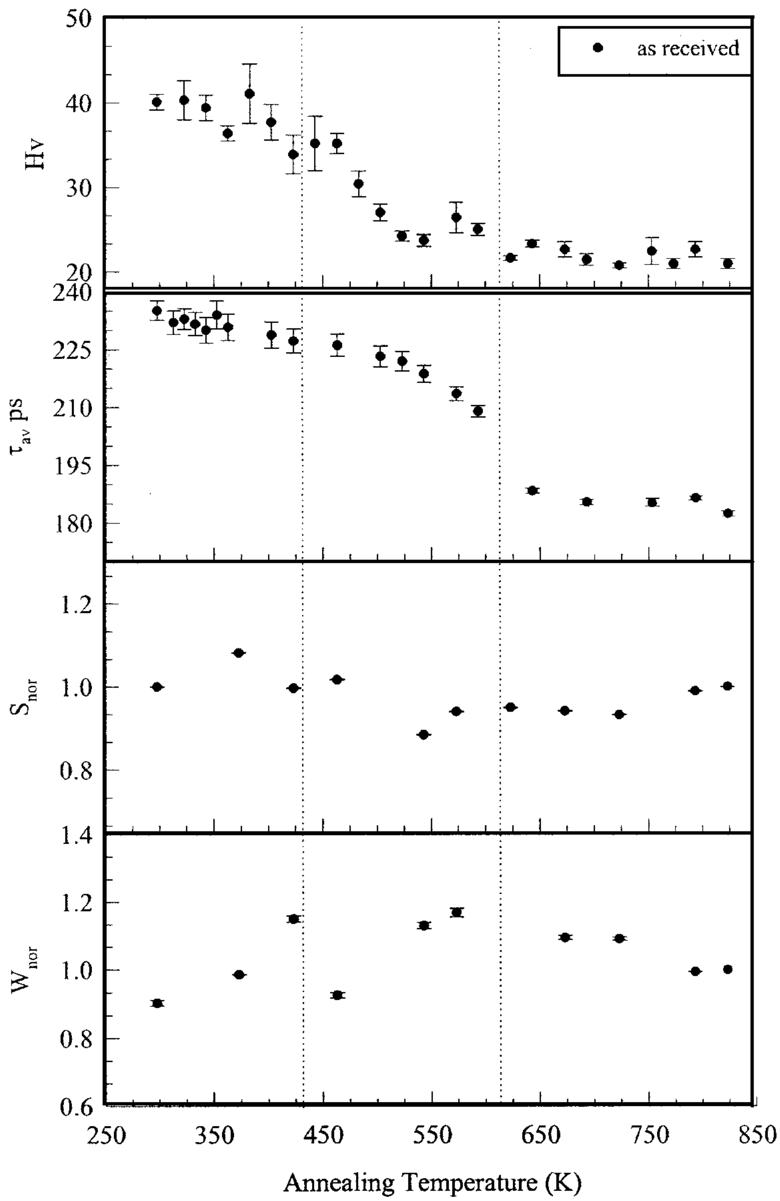

Fig. 1 Hv, τ_{av}, *S*-parameter, and *W*-parameter as a function of annealing temperature for the as-received commercial pure Al (1050)

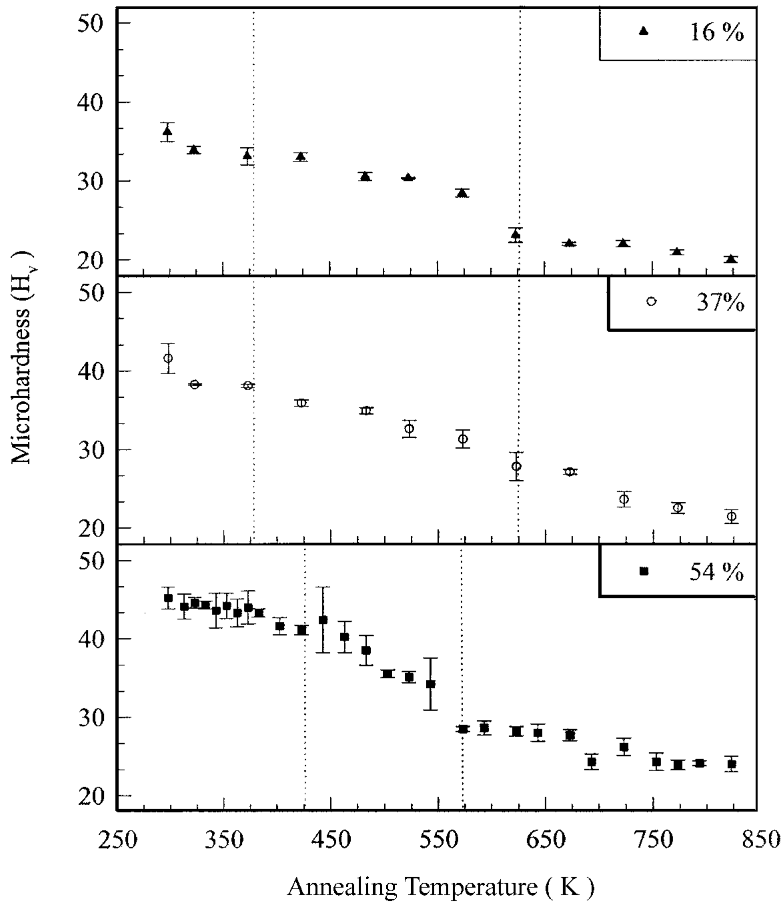

Fig. 2 Hv as a function of annealing temperature for 16%, 37%, and 54% cw commercial pure Al (1050) samples

interval 3.4-5.1 KeV from the peak center on either side of the peak. All parameter values refer to the unit potopeak area. The parameters S_{nor} and W_{nor} are determined from the ratio of S/S_{ref}, and W/W_{ref} respectively. The S_{ref} and W_{ref} were obtained by measuring the line shape distribution using a fully annealed commercial pure Al (1050) sample.

2.4 Vickers Microhardness Measurements

Vickers microhardness (Shimadzu, Kyoto, Japan) measurements were performed by using applied load 50 g for 10 s. More than 10 readings were measured for every sample and the standard deviations were estimated. The measurements were taken at RT.

3. Results and Discussion

3.1 As-Received Commercial Pure Al (1050)

Figure 1 shows the variation of Hv and the average lifetime of positrons (τ_{av}) as well as the line shape S_{nor} and wing W_{nor} parameters with the annealing temperature for the as-received (the starting material) commercial pure Al (1050). From the figure, three stages can be distinguished. The first stage, attributed to recovery, is taken from RT to 427 K. In this stage, a slight decrease in Hv, τ_{av}, and S_{nor} is observed due to the movement of some impurities in commercial pure Al, which hinders the dislocations motion. In addition the high values of τ_{av} (ranging from 225-235 ps) indicated that the positrons could be annihilated in vacancy clusters, monovacancy, and dislocations with high concentrations.[8,9] On the other hand, the increase in W_{nor} indicates that the positron has high overlap with core electrons.[10] However, the decrease of S_{nor} and increase of W_{nor} indicate a decrease of the concentration of low-momentum electrons, mainly due to annihilation of defects and a partial recovery of dislocations.[11]

The second stage, ranging from 427-623 K is characterized by a rapid decrease in Hv, τ_{av}, and S_{nor} while an increase in W_{nor} is observed due to complete recovery and the beginning of partial recrystallization.

The third stage, taken from 623-850 K, is characterized by a saturation of Hv, τ_{av}, S_{nor}; W_{nor} indicates complete recrystallization. At the end of this stage, the values of S_{nor} and W_{nor} approach the bulk value, which is 1.00. On the other hand, τ_{av} saturates at 190 ps, reflecting complete annealing of some defects, and the positrons can be annihilated in grain boundaries with low concentrations.[12]

3.2 Deformed Commercial Pure Al (1050)

In Fig. 3-6, Hv, τ_{av}, S_{nor}, and W_{nor} parameters of cold-worked commercial pure Al (16%, 37%, and 54% thickness

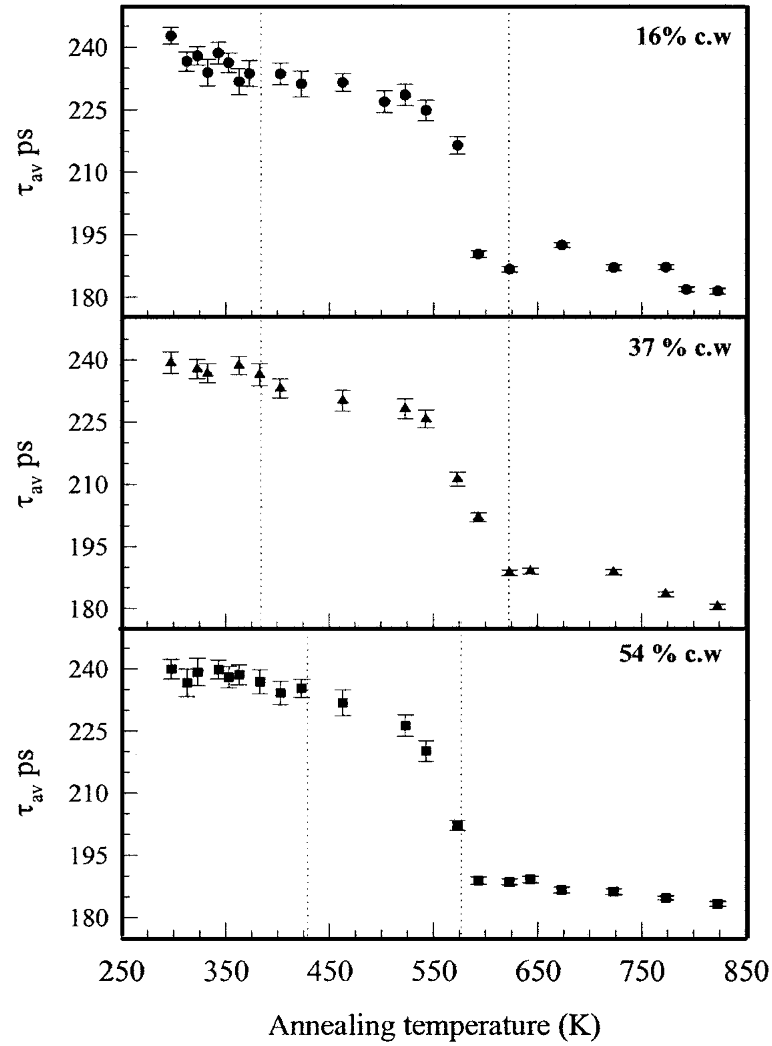

Fig. 3 τ_{av} as a function of annealing temperature for 16%, 37%, and 54% cw commercial pure Al (1050) samples

reduction) are shown, respectively. From the figures, the behavior can be divided into three stages. We observed that the recovery stage was extended to low temperatures for the light cold-worked samples while recrystallization was accelerated for the heavy cold-worked one. At low temperatures the high values of Hv and τ_{av} may be attributed to the cold rolling of Al (1050), which is characterized, by high amounts of dislocations. The values of S higher than the bulk value means that all positrons annihilated either in monovacancies or dislocations.

The first stage ranged from RT to 373 K for light cold-worked samples (16% and 37%) and delayed to 427 K for the heavy cold worked one (54%). This stage is related to the recovery process; it shows no variation in Hv and τ_{av} (Fig. 3, 4), a slight decrease in S_{nor} for all samples (Fig. 5), while W_{nor} shows an increase for 16% cw and a decrease for 37% and 54% cw (Fig. 6). The decrease in S_{nor} and the increase in W_{nor} in the case of the 16% cw sample indicate a decrease of the concentration of low-momentum electrons due to a partial recovery of dislocations.[11] However, the decrease in W_{nor} for 37% and 54% cw is due to a decrease of the average electron density indicating a recovery, which is retarded at a higher temperature for the more condensed cold worked one (54% cw). This behavior is due to the presence of two opposing processes acting at the same time, namely recovery and work hardening, which are found in 54% cw.

The second stage ranged from 373-625 K for 16% and 37% cw samples and from 427-575 K for the 54% cw sample. This stage, which is attributed to partial recrystallization, shows a rapid decrease of Hv for the 54% cw sample and a slight decrease for the 16% and 37% cw samples, attributed to a special rearrangement of dislocations as expected for these samples. These behaviors were confirmed by the trend of τ_{av}, S_{nor}, and W_{nor} in Fig. 4, 5, and 6, respectively. The τ_{av} decreases rapidly, approaching 190 ps due to the reaction of small voids from the migration of large vacancy defects, which is associated with the reduction of defect size and concentration. Therefore, the decrease of τ_{av} occurs when the dislocations near grain boundaries gradually recover and in grain boundaries in which the vacancy clusters decompose. S_{nor} tends to increase due to the migration of vacancies followed by a decrease for 16% and 37% cw samples and a decrease for the 54% cw

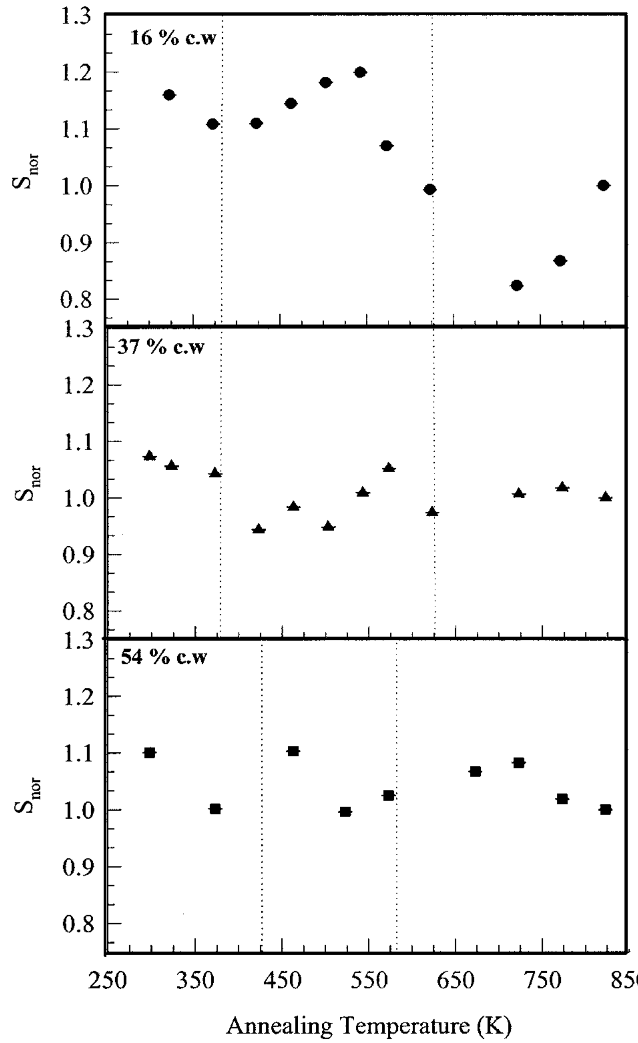

Fig. 4 *S*-parameter as a function of annealing temperature for 16%, 37%, and 54% cw commercial pure Al (1050) samples

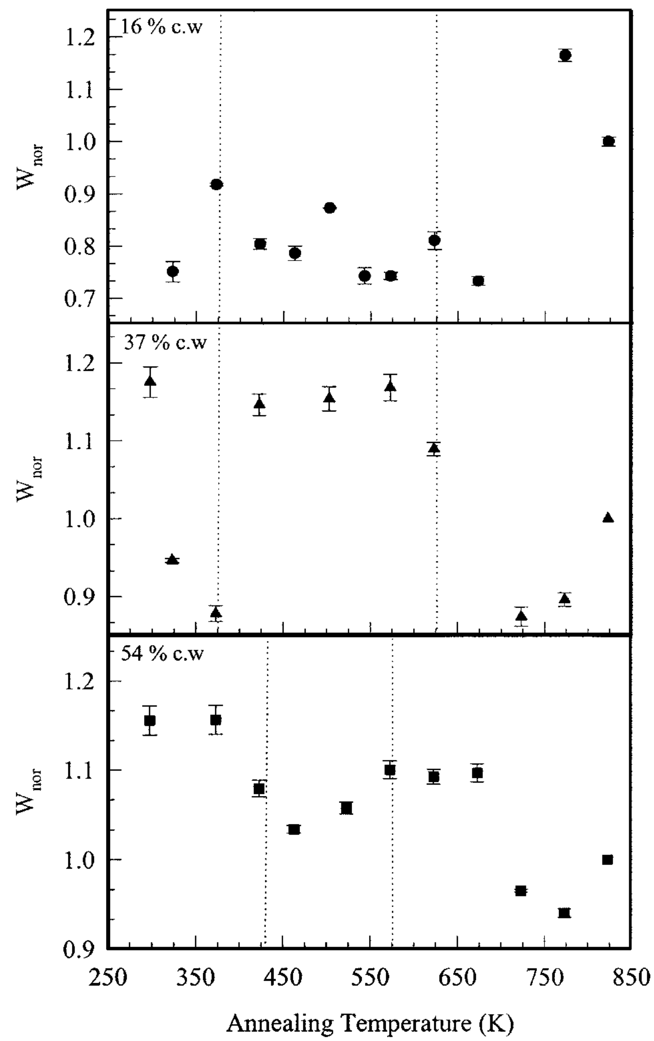

Fig. 5 *W*-parameter as a function of annealing temperature for 16%, 37%, and 54% cw commercial pure Al (1050) samples

sample. In Fig. 5, W_{nor} shows almost no change in this stage, indicating that the fraction of positrons annihilating with core electrons does not change very much as the trapping defect decreases in size. The values of τ_{av} ~ 190 ps and S_{nor} approaching S_{bulk} were observed already at 575 K for the 54% cw sample, which means that in the case of heavy cold working, recrystallization will take place with higher speed than that of light cold working.

The third stage is taken from 625-850 K for 16% and 37% cw samples and from 575-850 K for the 54% cw sample.

The figures show saturation in Hv, τ_{av}, and S_{nor} while a decrease is followed by an increase in W_{nor} for all samples (Fig. 6), which indicates complete recrystallization and grain growth. The values of S_{nor} approach the bulk value of 1.00, which is in good agreement with other results,[7] reflecting the decrease in defect concentrations and change in the defect formation or the atomic configuration around vacancies.[13] This supports the PAL and Hv measurements that the concentration of defects decreases causing less positron trapping. Also, the saturation of τ_{av} indicates that the dislocations defects disappear, which was confirmed by an increase in W_{nor}, where the positron can be annihilated with core electrons.

Figure 6 shows the S_{nor} and W_{nor} plot for as-received commercial pure Al, which shows a better view of the defects as demonstrated before by Abd El Wahab et al.[7] and Van Veen et al.[13] The points in the plot follow a linear trajectory, which indicates that one type of defect is present. The same trends were observed for the deformed samples.

4. Conclusions

- There are similar changes in τ_{av}, Hv, and *S* while a reverse behavior is observed in *W* during the stages of recovery of as-received and deformed commercial pure Al.
- It is shown that heavy cold working accelerated the recrystallization process, whereas light cold working accelerated the recovery process.
- At the last stage, both *S* and *W* approach the bulk value

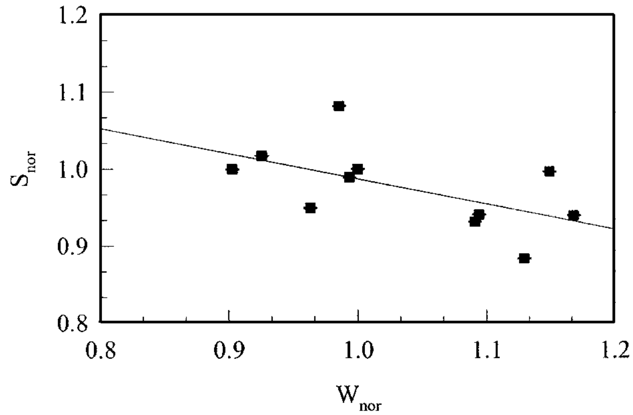

Fig. 6 *S-W* plot for as-received commercial pure Al (1050)

while τ_{av} showed saturation at 190 ps, reflecting complete annealing of defects.

- The *S-W* plot revealed the presence of one type of defect in commercial pure Al.

References

1. A.S. Taha, S. El-Houte, F.H. Hammad, and N. Hansen: "Recrystallization of Cold Worked Al-Al_2O_3 Alloy and Commercially Purity Aluminum" in *First International Spring School and Symposium on Advances in Materials Science*, Vol. 11, Atomic Energy Authority, Cairo, Egypt, 1994, pp. 297-312.
2. M. Mohsen, H, Ismail, A. Ashry, G. Brawer, and S. Mohammed: "Lattice Defects in Industrial Al Probed by Positrons," *Mater. Sci. Forum,* 2001, *363-365,* pp. 216-18.
3. M.S. Abd El Keriem, M. Mohsen, M.H. Kalil, M. Abd El Wahab, and A.S. Taha: "Application of PAS to Study Recovery and Recrystallization of Commercial Pure Al and Al-1wt% Mn Alloys," *J. Mater. Eng. Perform.,* 1998, *7,* pp. 673-76.
4. M. Iwami, E. Hashimoto, and Y. Ueda: "Change in the Positron-Trapping Efficiency of Dislocations in Al on Heating After Deformation," *Phys. Condens. Matter,* 1995, *7,* pp. 9935-42.
5. P. Hautojarvi, ed.: "Positron in Solids" in *Topics in Current Physics,* Vol. 12, Springer-Verlag, Berlin, Germany, 1979.
6. S. Mohammed, N. Mostafa, E. Gomaa, and M. Mohsen: "Application of Positron Annihilation Spectroscopy to Study the Recovery of Commercial Pure Al and Al-0.9wt% Si Alloys," *J. Mater. Eng. Perform.,* 2003, *12*(1), pp. 95-98.
7. M. Abd El Wahab and W. Arafa: "Application of DBAR to Evaluate the Microhardness Variation During Isochronal Annealing of Al and Al(Mn) Alloys," *J. Mater. Eng. Perform.,* 2000, *9,* pp. 355-58.
8. P. Hautojarvi, J. Heinio, M. Manninen, and R. M. Nieminen: "The Effect of Microvoid Size on Positron Annihilation Characteristics and Residual Resistivity in Metals," *Philos. Mag. A,* 1977, *35,* pp. 973-81.
9. S. Lui, B. Wang, C. Tang, W. Qui, and F.Tien: "The Investigation of High Temperature Substeady Creep in Pure Al by Positron Annihilation," *Mater. Sci. Forum,* 1995, *175-178,* pp. 443-46.
10. M. Harting, A, Hempel, M. Hempel, R. Bucher, and D. T. Brittan: "Defect Structure Characterisation of Plasma-Sprayed Bioactive Ceramic Coating," *Mater. Sci. Forum,* 2001, *363-365,* pp. 502-04.
11. N.G. Chechenin, A. Van Veen, R. Escobar Galindo, H. Schut, A. Chezan, and D.O. Boerma: "Positron Annihilation in Gaseous Nitrided Cold-Rolled Fe Ni Ti Films," *Mater. Sci. Forum,* 2001, *363-365,* pp. 493-95.
12. P. Hautojarvi, J. Heinio, M. Manninen and R.M. Nieminen: "The Effect of Microvoid Size on Positron Annihilation Characteristics and Residual Resistivity in Metals," *Philos. Mag, A,* 1977, *35,* pp. 973-81.
13. A. Van Veen, A.C, Kruseman, H. Schut, P.E. Mijnarends, B.J. Kooi, and J.Th.M. De Hosson: "Positron Analysis of Defects in Metals," *Mater. Sci. Forum,* 1997, *255-257,* pp. 67-80.