A Review of Recent Positron Annihilation NDE Applications

S. Panchanadeeswaran
University of Utah

Po-We Ure Jr.
University of Utah

J. G. Byrne
University of Utah

Follow this and additional works at: http://lib.dr.iastate.edu/cnde_yellowjackets_1979

Part of the Materials Science and Engineering Commons

Recommended Citation
http://lib.dr.iastate.edu/cnde_yellowjackets_1979/19

This 4. Eddy Currents, Techniques and Phenomena is brought to you for free and open access by the Interdisciplinary Program for Quantitative Flaw Definition Annual Reports at Iowa State University Digital Repository. It has been accepted for inclusion in Proceedings of the DARPA/AFML Review of Progress in Quantitative NDE, July 1978–September 1979 by an authorized administrator of Iowa State University Digital Repository. For more information, please contact digirep@iastate.edu.
A REVIEW OF RECENT POSITRON ANNIHILATION NDE APPLICATIONS

S. Panchanadeeswaran, Po-We Kao, R. W. Ure, Jr., and J. G. Byrne
Department of Materials Science and Engineering
University of Utah
Salt Lake City, Utah 84112

ABSTRACT

This review will treat two recent applications of positron annihilation to metallurgical studies and will involve the measurement of the Doppler effect associated with the gamma rays emitted during positron annihilation. The applications will be: studies of the interactions of dislocations with pre-precipitates in aged Al-4 weight percent Cu single crystals and studies of the effect of hydrogen charging into polycrystalline nickel.

INTRODUCTION

Earlier, it was reported that a commercial aluminum base age hardening alloy, 7075, responded to Doppler broadening measurements by exhibiting higher values of the peak to wings shape parameter for harder conditions. Thus, the Doppler shape factor was behaving in much the same way in which it would respond to plastic deformation in a pure metal; that is, in a pure metal subjected to damage the Doppler peak shape becomes sharper because dislocations and vacancies provide locations for positron trapping which are lacking in ion cores. Hence, positrons attracted to and trapped in such regions will tend more to annihilate with lower energy conduction electrons. Such annihilations cause a smaller Doppler shift from the central energy value of 511 keV than would be caused were the positron to annihilate with a more energetic core electron. Thus, the curve representing the number of annihilations versus plus and minus deviations in annihilation gamma ray energy about a central value of 511 keV (the value if the electron-positron center of mass were stationary) sharpens with rising defect concentration and, conversely, the shape broadens with the annealing out of damage. The gamma rays emitted on annihilation enter a Ge(Li) spectrometer capable of measuring their energy.

RESULTS

To have a better chance of understanding the response of positrons to changes during age hardening, we set out to produce well-documented aged states in a known alloy; that is, to look at G.P. zones, transition precipitates (8'), and the equilibrium precipitate (8) in both single and polycrystalline Al + 4 weight percent Cu alloy. The heat treatments used were: solution treated (S.T.) at 550°C for 1 hour + water quench (Q) for the supersaturated solid solution; S.T. + Q + 130°C for 48 hours for G.P. I zones; S.T. + Q + 190°C for 5 hours for G.P. II zones; S.T. + Q + 240°C for 24 hours for 8' and S.T. + Q + 400°C for 24 hours + 315°C for 48 hours for 8 (the equilibrium precipitate).

When peak to wing (P/W) Doppler comparisons were made, both Na22 and Ge68 were used as positron sources. The Na22 produces positrons with a maximum energy of 0.546 MeV and the Ge68 produces positrons with a maximum energy of 1.9 MeV; thus, the former positrons will sample events closer to the specimen surface than the latter positrons.

P/W data for an aged single crystal utilizing a Na22 positron source are shown in Fig. 1, plotted against the principal aging temperature. One sees a rise in P/W ratio from the solid solution level to the G.P. zone conditions and a later decline as 8' and 8 are formed.

Figure 2 exhibits P/W data taken on the same samples, but with Ge68 as the positron source. One sees a similar trend (except for the G. P. II point) in the data, all of which lie in a lower magnitude range. When these experiments were repeated with polycrystalline samples and the Ge68 positron source, similar data trends to those of Fig. 2 resulted (with the exception of G. P. II).

A number of conclusions emerge at this point. For the "bulk" data obtained with Ge68, the P/W values for the polycrystals were usually lower point for point than the corresponding values for the single crystal experiments. This would suggest that in the quenched condition fewer point defects are present in the polycrystal because of leakage to grain boundary sinks. In subsequent aging this would lead to a lower density of pre-precipitate nucleation sites in the polycrystal. This would account for the lower P/W ratios in a G. P. zone condition because the coherency strain or hardness of the region measured would be lower.

Again, the case of the G. P. II zone containing single crystal Ge68 data violates this scheme. The same data point will later be seen to be at variance with the interpretation given to the G.P. I condition; that is, it can reasonably be regarded as a bad data point, but for reasons as yet unclear.

For 8' and 8 data, the lower nucleation frequency again seems to explain the lower values of P/W ratio for the polycrystalline samples. However, the basis for the values themselves no longer can be ascribed to coherency strains, but rather may be reflective of positron trapping at the 8' and 8 matrix-particle interface.

In every case, P/W values measured closer to the surface (Na22) exceeded those measured at greater depths (Ge68). This may be related to the higher quenching rates and point defect concentrations of the surface region which in turn would on aging produce a higher nucleation rate.
of precipitation.

Independent of depth, the quenched solid solution was always lower in P/W ratio than either the G.P. zone or overaged conditions. This implies that the stain effect of G.P. zones is much more interactive with positrons than is the discreet point defect distribution created by quenching. The η' and η conditions are also more interactive with positrons than is the quenched state—again probably due to interfacial rather than strain field trapping reasons.

In comparing G.P. I and G.P. II zones, it seems (with the one exception previously noted) that the trapping of positrons is greater for the more highly stained G.P. II condition. As one then passes to the partially coherent (less strain) η' situation, the positrons are less effectively trapped than by the coherent G.P. II particles. Finally, in comparing η' and η, it seems that the completely incoherent η particles have a more effective positron trapping interface than do the η' particles.

We now proceed to more recent single crystal experiments in which the interaction of various of the above aged states in single crystals respond to tensile deformation. It is known that for G.P. zone states dislocations must rigidly cut through the zones. For the η' and η situations, on the other hand, the dislocations bow out between the particles, wrap around, and pass on as in the zone or averaged conditions. This may be seen via microhardness. Yet, the existence of the defects could be seen via microhardness.

The second subject to be discussed is the hydrogen charging of metals. Earlier work in this laboratory showed a hydrogen-positron relationship in steel which was appropriate for the nondestructive detection of hydrogen embrittlement. In the subsequent work, nickel (after various amounts of cold work) was cathodically charged with hydrogen. The sharpness of the Doppler peak increased at first and to a greater extent the greater the amount of initial deformation. Following the initial sharpening, a broadening occurred. A mechanism which could explain this is one in which protons migrate to dislocations introduced by cold work and subsequently form gaseous hydrogen molecules which produce enough pressure to generate new dislocations at these locations. The broadening of the Doppler peak, following the initial narrowing, is attributed to protons reducing the attractive potential between positrons and dislocations. Figure 7 shows the undulating character of the positron-proton-dislocation relationship for two current densities for samples deformed 10.7 percent in tension.

A 70 percent cold rolled sample was cathodically charged for three hours and then measured as a function of time at 300°K. Figure 8 shows how P/W increased during this period. This is attributed in part to the diffusion of protons out of the sample. This would unscreen some dislocations and thus raise P/W. Another cause may be that protons detrapped from dislocations or delivered by dislocation short-cut diffusion to inclusions or grain boundary locations may combine to form H₂ and generate new dislocations by exerting pressure.

Figure 9 shows a schematic diagram of microhardness measurement directions on special samples. The central impression was made with a 1 kg load and the two orthogonal directions enabled 25 gram microhardness measurements to be made as a function of distance from the large impression with and without hydrogen charging. Figure 10 shows on the upper curve (H₁) how the microhardness varies with distance from a high dislocation density after charging and after a one hour anneal at 365°K (curve H₂); that is, with no charging.

Cathodic charging produced no change in the P/W shape factor of annealed Ni; yet, the microhardness of annealed Ni increased with charging as is seen by the right end of curve H₁ (higher than right end of curve H₂). This suggests that the P/W ratio was seeing an exact balancing between defect generation and defect screening by protons during charging. Yet, the existence of the defects could be seen via microhardness.

ACKNOWLEDGMENTS

The authors wish to acknowledge the financial support of the Air Force Office of Scientific Research which made this work reported in this review possible.
Fig. 1. Doppler P/W shape factors for an Al + 4 percent Cu single crystal in various aged states. Na\textsuperscript{22} positron source.

Fig. 2. Doppler P/W shape factors for an Al + 4 percent Cu single crystal in various aged states. Ge\textsuperscript{68} positron source.

Fig. 3. Doppler peak to total (I_v) and wings to total (I_c) shape factors versus tensile strain for an Al + 4 percent Cu single crystal containing G. P. I zones.

Fig. 4. Doppler peak to total (I_v) shape factor versus tensile strain for an Al + 4 percent Cu single crystal containing G. P. II zones.
Fig. 5. Doppler peak to total ($I_v$) and wings to total ($I_c$) shape factors versus tensile strain for an Al + 4 percent Cu single crystal containing $\theta$ transition precipitates.

Fig. 6. Doppler peak to total ($I_v$) and wings to total ($I_c$) shape factors versus tensile strain for an Al + 4 percent Cu single crystal containing $\theta$ equilibrium precipitate particles.

Fig. 7. Doppler peak to wings shape factor versus hydrogen charging time in polycrystalline nickel.
Fig. 8. Doppler peak to wings (P/W) shape factor versus decay time at ambient following cathodic charging of hydrogen into nickel.

Fig. 9. Microhardness measurement directions and sample size for results shown in Fig. 10. Paths 1 and 2 should be used before and paths 3 and 4 after cathodic charging.

Fig. 10. Microhardness versus distance from a 1,000 gm impression before (curve H₂) and after (curve H₁) cathodic charging of hydrogen into nickel.
References


