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Nondestructive Examination of Deformed Steel and Nickel by Positron Annihilation *

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Positron Annihilation (PA) Doppler broadening measurements have been carried out on cold-rolled and fatigue cycled 316 stainless steel. In addition, PA measurements were done after isochronal annealing steps following deformation at 294 K of both 316 SS and high purity nickel. In order to understand better the response of PA to microstructural changes, specimens were examined by transmission electron microscopy as well. It is concluded that in nickel both vacancies and dislocations produced by cold working can act as positron traps. In 316 SS, however, the PA response is principally due to vacancies, and only small effects can be attributed to trapping at dislocations.

The response of positron annihilation (PA) to vacancies and dislocations has been observed for many metals [1]. Several studies have shown large changes in PA parameters due to the production of these defects during plastic deformation [2]. In order to apply PA as a monitor of deformation or fatigue in metals, and to correlate the information obtained from PA with changes in mechanical properties, it is necessary to know what changes in microstructure determine PA response and how these changes relate to the mechanical properties. In the present work we are concerned with determining the extent to which PA is sensitive to the density and structure of dislocations produced by cold working and fatigue cycling of 316 stainless steel and by cold working high-purity nickel. 316 SS is a single phase alloy with the same fcc crystal structure as nickel.

The Doppler broadening data were analyzed in terms of two parameters. S, the lineshape parameter, is defined as the counts in a central portion of the annihilation line, corresponding to about one third of full width at half maximum, divided by the total number of counts in the line, times 100. S measures a total response due to different types of trapping defects. In addition, the defect-specific R-parameter, defined and discussed by Triftshäuser [3] and by Mantl and Triftshäuser [4], is used for analysis. The apparatus is described in reference [5]. The steel samples were commercial 316 SS; the nickel for our measurements, nominally 99.995% pure, was obtained from Materials Research Corporation.

Figure 1 shows the measured values of S for fatigued 316 SS as a function of the number of cycles. The standard round bar fatigue samples, of 0.64 cm diameter, were sectioned after the fatigue tests and electro-polished to remove surface damage from the cutting. The PA results show

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an increase of S with increasing number of cycles up to a saturation value which is reached at a lower number of cycles for the higher strain range. The saturation value for the sample fatigued at 866 K is much lower than the value for the samples fatigued at room temperature.

TEM studies of the same samples showed an increase in dislocation density with number of cycles and the development of a dislocation

cell structure characteristic of fatigue cycling [6]. There was not, however, a consistent correlation between the values of S and dislocation density for samples fatigued at different strain ranges ($\Delta\epsilon$) or temperatures. For example, the sample fatigued at $\Delta\epsilon = 0.6\%$, $T = 294$ K, 47173 cycles had a value of S close to that for the sample fatigued at $\Delta\epsilon = 1.8\%$, $T = 294$ K, 4000 cycles, but showed a much lower dislocation density. Furthermore, samples fatigued for more than 1000 cycles at 866 K showed relatively high dislocation densities but only small increases in S over the value for undeformed samples.

These results suggested strongly that the PA response was affected by lattice vacancies generated during the deformations. In order to separate vacancy and dislocation effects, isochronal annealing measurements are useful, since vacancies usually become mobile and migrate to sinks at temperatures where dislocations are sessile. An annealing study was therefore done on cold-rolled 316 SS and on cold-rolled pure nickel, chosen as a well characterized model material.

Figure 2 shows the values of S and R, always measured at room temperature, after isochronal annealing steps with 30 minute holding times, for polycrystalline Ni rolled at room temperature to 25% thickness reduction. The S-parameter is seen to decrease in two distinct stages, occurring at nearly the same temperatures as the annealing stages of residual resistivity measured for quenched Ni by Wycisk and Feller-Kniepmeier [7]. From this close correspondence with resistivity measurements we conclude that the same mechanisms are operative; i.e., that the first stage occurs where vacancies become mobile and that the second stage is due to the disappearance of dislocations (recrystallization). This interpretation was confirmed by TEM [6]: micrographs of 25% cold worked nickel samples showed no change in the dislocation structure after annealing at 575 K but did show a low dislocation density after annealing at 800 K. Thus the PA data show clearly that positrons are sensitive to dislocations in nickel and that the drop in S near 700 K is due to the disappearance of the dislocations. The decrease of the R-parameter between 375 K and 550 K is then associated with a change in the dominant type of trapping site from vacancies to dislocations; as the dislocation density decreases during the second annealing stage (550 K to 725 K) no further change in R is observed. The PA response before annealing is due to positron trapping at vacancies

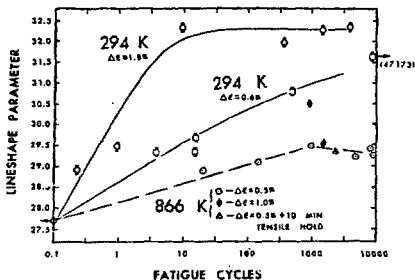


Fig. 1 PA lineshape parameter for fatigue-cycled 316 SS, strain ranges $\Delta\epsilon$.

alone or at both vacancies and dislocations; the response after annealing above 550 K is due to positron trapping principally at dislocations.

The results of a similar annealing experiment on 316 SS, also cold-rolled to 25% thickness reduction, are shown in Figure 3. Here the decrease of S begins at a higher annealing temperature than for nickel and proceeds more gradually without being resolvable into two distinct stages. The R-parameter remains nearly constant, showing only a slight drop at the highest annealing temperatures. Microhardness measurements on similarly annealed samples showed a decrease of Knoop hardness number only after annealing at temperatures above 1000 K. TEM on 316 SS samples cold worked at room temperature to 75% reduction in thickness showed little change in the dislocation structure after annealing at 873 K, but showed a low dislocation density after annealing at 1073 K [6]. The disappearance of the dislocations is responsible for the decrease in hardness but was found to have little effect on the PA response. Thus the decrease in S on annealing to 873 K is primarily due to vacancy annealing, and the nearly constant R is consistent with single-defect PA response.

The lack of PA response to dislocations in 316 SS implies either that dislocations trap positrons only weakly, or else the presence of alloying elements causes the S-parameter for positrons annihilating at dislocations to be similar to the S-parameter for positrons annihilating in the bulk. In either case positrons are not strongly sensitive to the dislocations. This explains the lack of correlation between S and dislocation density observed earlier in the PA response to fatigue cycling.

Conclusions:

- 1) In 316 SS and nickel cold worked at room temperature, positrons are primarily trapped at lattice vacancies produced by the cold work.
- 2) In nickel the vacancies anneal out by about 550 K, leaving dislocations. In this case PA is sensitive to the dislocation density.
- 3) In 316 SS positrons are insensitive to the dislocation density. The changes in PA caused by cold work or fatigue cycling at room temperature are mainly due to lattice vacancies or defect complexes produced during plastic deformation that are not observed by TEM.

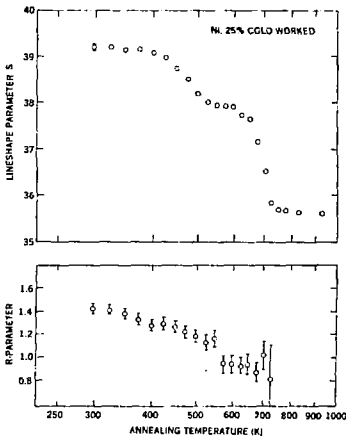


Fig. 2 The parameters S and R as a function of annealing temperature for cold worked nickel.

- 4) For applications of PA as a monitor of deformation it is essential to obtain defect-specific information, such as is provided by the R-parameter in the case of pure nickel.

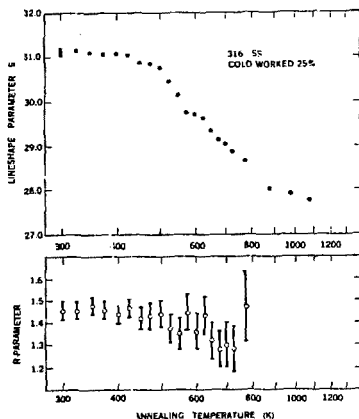


Fig. 3 The parameters S and R as a function of annealing temperature for cold worked 316 SS.

References

- [1] Documentation is found in the proceedings of the previous Positron Conferences.
- [2] See, e.g., G. Dlubek, O. Brümmer, and E. Hensel: *Phys. Stat. Sol. (a)* 34 (1976) 737.
- [3] W. Triftshäuser: *Phys. Rev. B* 12 (1975) 4634.
- [4] S. Mantl and W. Triftshäuser: *Phys. Rev. B* 17 (1978) 1645.
- [5] W. R. Wampler and W. B. Gauster: *J. Phys. F: Metal Phys.* 8 (1978) L1.
- [6] A full discussion of the TEM results will be published elsewhere.
- [7] W. Wycisk and M. Feller-Kniepmeier: *Phys. Stat. Sol. (a)* 37 (1976) 183.