

W. B. Jones, J. A. Van Den Avyle, W. B. Bauster and W. R. Wampler
 Sandia Laboratories
 Albuquerque, NM 87185

ABSTRACT

The positron annihilation technique can provide a sensitive measure of defect density in metals. In this program the technique has been used to monitor defects generated during plastic deformation by cold work or fatigue cycling. The primary goals have been 1) to assess the degree of sensitivity of the technique, 2) to correlate positron annihilation readings with observed microstructural changes to better understand the physical basis for these readings, and 3) to determine correlations between positron annihilation measurements and number of fatigue cycles.

Examination of fatigued samples by transmission electron microscopy indicates some correlation between dislocation density and positron annihilation lineshape parameter (determined by the Doppler broadening technique). However, annealing studies of deformed samples indicate that positron annihilation response in 316 stainless steel is sensitive primarily to excess vacancies generated during the deformation and is less sensitive to dislocation density. Data on deformed nickel show sensitivity to both vacancies and dislocations. In general, lineshape parameter values tend to achieve a constant level at approximately 10 per cent of fatigue life.

INTRODUCTION

Elevated temperature design for advanced nuclear reactor components must incorporate creep and fatigue and their interaction. Current ASME Design Codes utilize the concept of damage accumulation to treat combined creep-fatigue loadings. This approach assumes that bulk changes occur during the service life that represent "damage". One question addressed by this study has been: Is there a measure of the bulk changes that occur during service that could be correlated to the accumulated "damage"? Flaw detection is not the purpose of such a technique.

Several candidate schemes have been surveyed with positron annihilation appearing to have more promise than the others (1,2). Positron annihilation has been shown (3) to be sensitive to vacancies, vacancy clusters and dislocations induced by irradiation or deformation of metals.

RESULTS AND DISCUSSION

Positrons injected into a metal annihilate with electrons emitting two gamma photons. The momentum of the annihilating electron causes a Doppler shift in the energy of the emitted photons. The sensitivity of positron annihilation to vacancy and dislocation concentrations in crystals arises from differences between electron momentum distributions in the perfect lattice and at defects. Figure 1 shows a schematic drawing of the apparatus used to measure the Doppler broadening of the annihilation line. Positrons are produced in the source by the β^+ decay of a naturally radioactive isotope. For this study, ^{22}Na was the isotope used. These positrons penetrate relatively short distances into the metal sample (Fig. 2) where they annihilate with electrons within about 100 psec. Test specimens examined in this study were sectioned normal to the stress axis so that the material sampled by the positrons was originally at the interior of the gauge section.

The result of the Doppler broadening measurement is a distribution of gamma energies around the 511 keV line. The parameter chosen to characterize this curve is the lineshape parameter, S. Details

concerning this characterization can be found in reference 1, however, it is important to note that the value of S depends on the density of dislocations and vacancies. The larger the value of S, the higher is the density of vacancies and dislocations.

To initiate this study, a number of fatigue tests on type 316 stainless steel were conducted at room temperature and the dislocation density monitored for comparison with lineshape data. The lineshape parameter, S, is plotted versus number of fatigue cycles in Fig. 3 together with transmission electron micrographs of representative dislocation substructures. For these test conditions the dislocation density and the lineshape parameter both increase with increasing number of fatigue cycles.

Figure 4 shows the results of fatigue tests conducted under several different conditions. For each set of conditions, the lineshape parameter followed the dislocation density changes; however, comparison of the values of S and the dislocation densities among the several conditions did not reflect a correlation (Fig. 5). In addition, the observed changes in S saturated at about 10% of life.

As pointed out earlier, the positron annihilation response is known to be sensitive to the presence of both dislocations and vacancies. In order to distinguish the relative magnitude of each contribution, isochronal annealing was conducted on samples of cold worked 316 stainless steel and pure nickel. Figure 6 shows the changes in lineshape parameter induced by cold work for both materials. In the present study, a pure Ni specimen was cold rolled to a 25% reduction in thickness and isochronally annealed (Fig. 7). Together with electron microscopy results, these data show that after annealing the Ni to 600 K to remove the excess vacancies, the lineshape parameter has noticeably decreased due to the loss of vacancies. However, about 60% of the total initial increase remains due to the dislocations. Figure 8 shows that after annealing 316 stainless steel at 873 K, the lineshape parameter has nearly completely recovered whereas electron microscopy and microhardness have shown that the dislocation structure has not

changed. From this we conclude that most of the initial response was due to the vacancies and only a very small fraction could be caused by the dislocations in the 316 stainless steel.

Figures 9 and 10 show the annealing response of 316 stainless steel cycled at room temperature and 866 K. As anticipated, the material fatigued at room temperature undergoes significant annealing of the lineshape parameter due to the large number of excess vacancies present after cycling. In contrast, cycling at 866 K results in a minimal accumulation of excess vacancies and, accordingly, the annealing treatment produced very little change in the lineshape parameter.

CONCLUSIONS

This study has shown that positron annihilation sensitivity to dislocation density must be established for each alloy. Even when such sensitivity is present, the response may be dominated by the presence of excess vacancies. Transmission electron microscopy has shown that the density and distribution of dislocations reaches a steady-state condition in the first 10% of the fatigue life so that, even with good dislocation sensitivity, the positron annihilation response would saturate at about 10% of total fatigue life.

ACKNOWLEDGEMENTS

Sandia Laboratories is a U. S. Department of Energy facility. This article was sponsored by the U. S. Nuclear Regulatory Commission under contract DE-ACO4-DPO0789.

REFERENCES

1. W. B. Gauster, W. R. Wampler, W. B. Jones, and J. A. Van Den Avyle, Sandia Laboratories Report SAND-77-1570, May 1978.
2. J. A. Van Den Avyle, W. B. Jones, and J. H. Gieske, Sandia Laboratories Report SAND-77-1557, July 1978.
3. C. F. Coleman and A. E. Hughes, in Research Techniques in Nondestructive Testing, R. S. Sharpe, ed., (Academic Press, New York) Vol. 3, 1977.
4. G. Dlubek, O. Brummer, and E. Hensel, *Phys. Stat. Sol. (a)*, 34, 737 (1976).
5. W. Wysick and M. Feller-Kniepmeier, *J. of Nuc. Mat.*, 69 and 70, 616 (1978).

POSITRON ANNIHILATION DOPPLER BROADENING

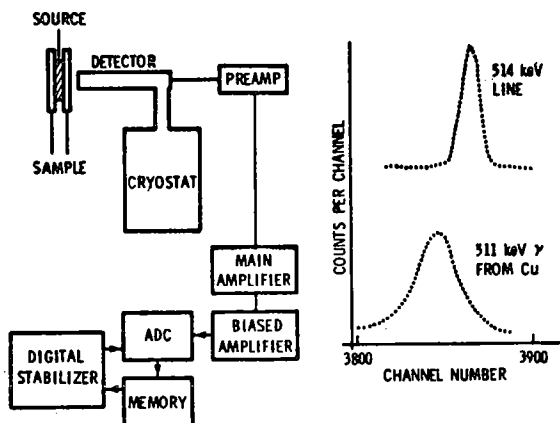


Figure 1.

Depths of Penetration of Positrons in Copper (Values for Steel will be ~10 to 20% Higher)

| Source | e-folding distance* | maximum penetration |
|------------------|--------------------------------|---------------------|
| ^{22}Na | 23.1 μm = 0.0009 in | 0.31 mm = 0.012 in |
| ^{68}Ge | 165 μm = 0.006 in | 1.42 mm = 0.056 in |

* Depth in material at which positron flux is $1/e$ (37%) of its value at the surface.

Figure 2.

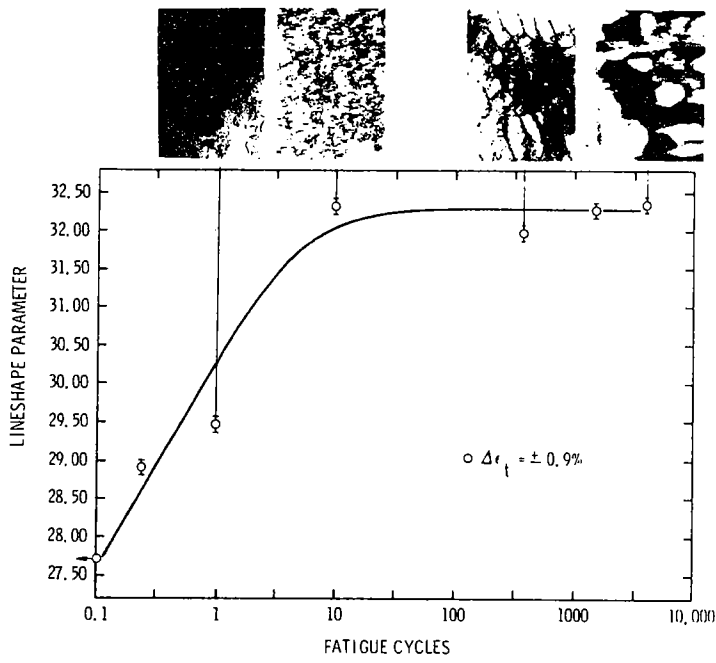
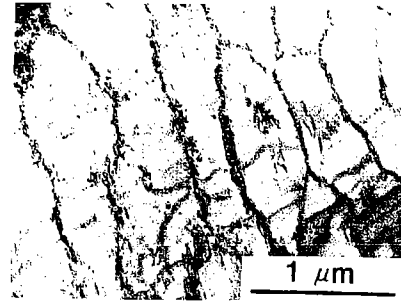
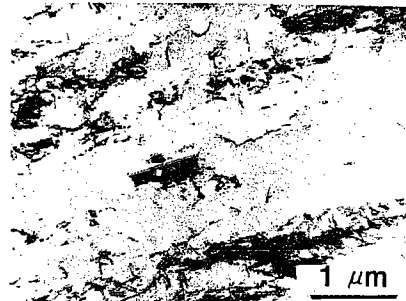


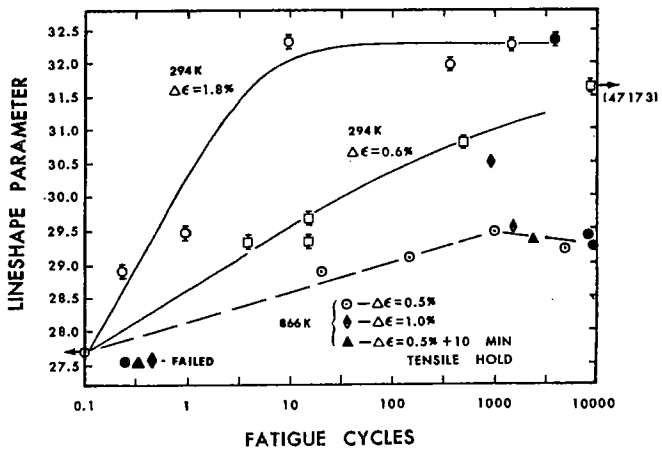
Figure 3.



294 K
 $\Delta\epsilon = 1.8\%$
 $N = 3700$
 $S = 32.3$

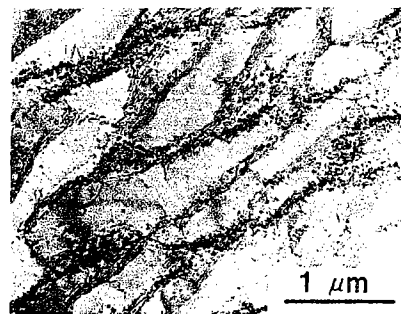


294 K
 $\Delta\epsilon = 0.6\%$
 $N = 47173$
 $S = 31.6$



**POSITRON ANNIHILATION RESPONSE
 OF FATIGUED 316 STAINLESS STEEL**

Figure 4.



866 K
 $\Delta\epsilon = 0.5\%$
 $N = 10000$
 $S = 29.2$

Figure 5.

POSITRON ANNIHILATION RESPONSE
OF COLD WORKED 316 STAINLESS STEEL AND NICKEL

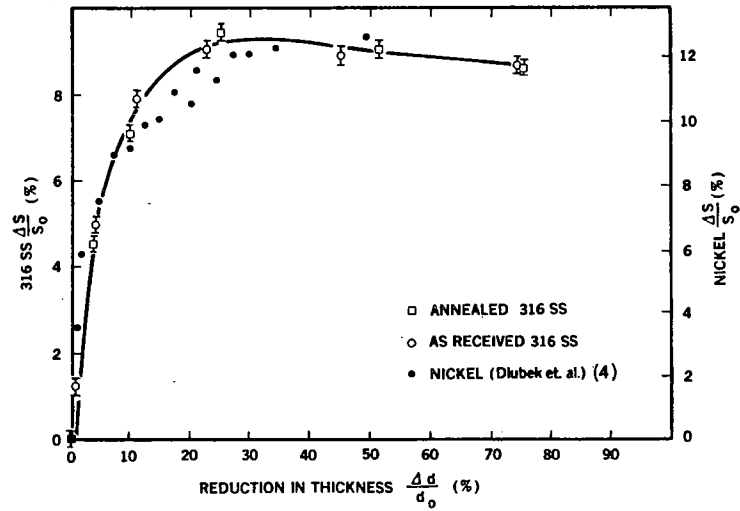


Figure 6.

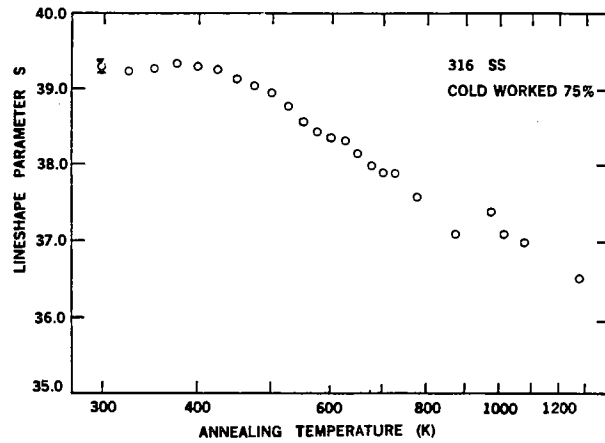
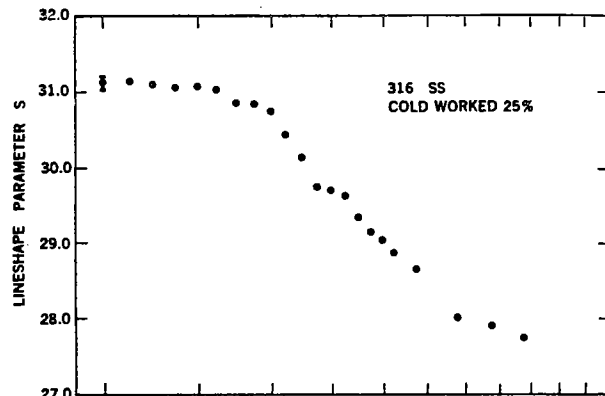
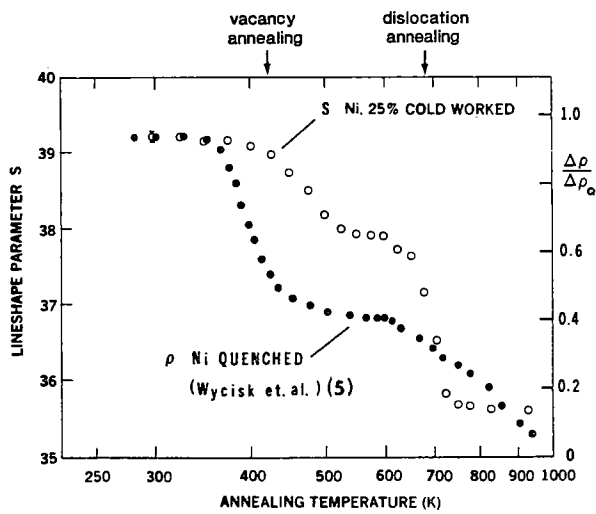


Figure 7.

Figure 8.

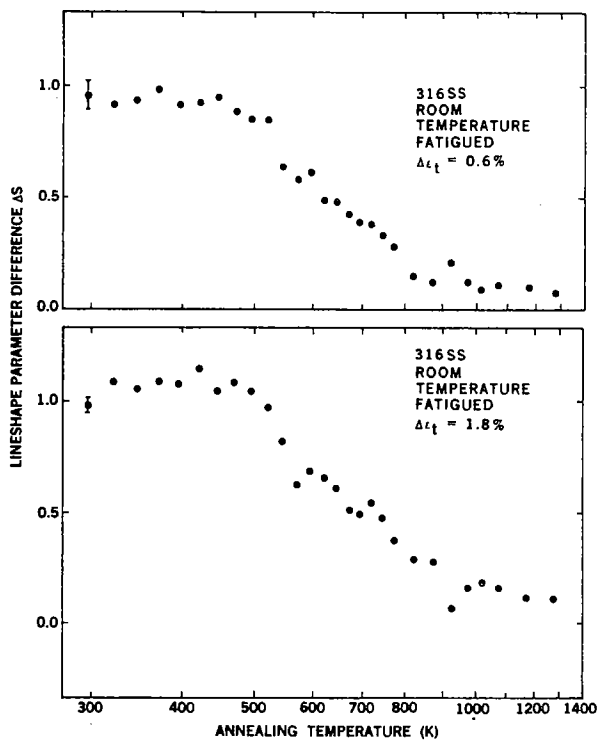


Figure 9.

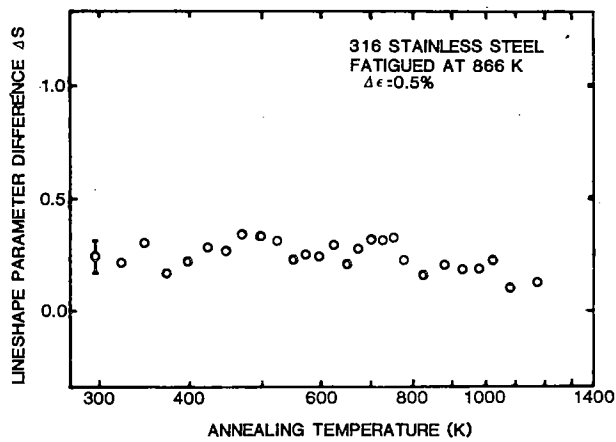


Figure 10.