UNITED STATES
NUCLEAR REGULATORY COMMISSION
WASHINGTON, D. C. 20555

AUG 5 1980

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MEMORANDUM FOR: Harold R. Denton, Director
Office of Nuclear Reactor Regulation

FROM: Robert J. Budnitz, Director
Office of Nuclear Regulatory Research

SUBJECT: RESEARCH INFORMATION LETTER # 95 POSITRON ANNIHILATION FOR NON-DESTRUCTIVE EXAMINATION

REFERENCE: J. A. Van Den Avyle, W. B. Jones, W. B. Gauster, and W. R. Wampler, "Assessment of Positron Annihilation as a Non-destructive Examination Technique,: NUREG/CR-1129 March 1980.

Introduction and Summary

This memorandum transmits the results of completed research on the ability of positron annihilation (PA) to measure "damage" quantitatively in metals. Although optimistic claims had been made in the literature for PA as an NDE technique, this research shows that it cannot be recommended for in-service inspection of components which have been operated at elevated temperatures. The availability of extensive equipment at Sandia from non NRC projects and fatigue test specimens from the NRC Elevated Temperature Program enabled a very cost effective study of the PA technique to be undertaken for temperatures to 1100°F for 316SS. The research results from this study show that for temperatures above ∿700°F, where vacancies in 316SS become mobile, adequate sensitivity does not exist in the PA technique to recommend it as a NDE technique for 316SS that has been deformed or fatigued in the temperature range of 700°F to 1100°F. The reference documents the PA technique, quantifies the sensitivity, provides a good basis for assessing PA as an engineering tool, and shows that it is not applicable to stainless steels for the operating temperature range of breeder reactors.

The ability of positron annihilation measurements to measure metal "damage" from fatigue is a result of the formations of vacancies and dislocations in the crystalline lattice structure. The positrons are preferentially annihilated by the higher density of electrons at such sites, thus providing a source of coincident photons which may be detected and counted. Correlation of the numbers of such sites with fatigue or plastic deformation would potentially provide a NDE measurement of component life.

Structural design rules which utilize "damage accumulation" assign life fractions to segments of creep and fatigue. During creep deformation, voids can form and during fatigue, dislocation densities can increase or decrease. Local accumulation of such damage results in crack initiation. The difficulties encountered in utilizing ultrasonics to evaluate crack growth in 316SS emphasized the need for a new method for monitoring damage at elevated temperatures. The PA technique had been used successfully to measure dislocation density changes in cold worked metals, and changes of vacancy concentrations and vacancy clustering in quenched metals.

The Nuclear Regulatory Commission (NRC) program at Sandia Laboratories on "Elevated Temperature Design Assessment" provided metallic specimens (316SS) with precise deformation histories and thorough metallography. Equipment was also available from another project for the PA measurements. Thus, precise tools and specimens were available at Sandia to assess PA as a non-destructive examination method for 316SS at elevated temperatures. An evaluation of the technique was undertaken and completed.

The goals of the Sandia research were: (1) to assess the sensitivity of the technique for stainless steels and the applicability at elevated temperatures; (2) to correlate positron annihilation readings with observed microstructural changes; and (3) to determine correlations among positron annihilation measurements and fraction of life or damage.

The results of the research showed that PA measurements of fatigue could be measured to 886 K (1100°F) but that the sensitivity decreased to an impracticable level. Practical sensitivity levels are limited to the temperature at which vacancies become mobile, approximately 650 K (710°F) for 316SS. Also, the accumulation of "damage" which modifies the positron annihilation is limited to about 10% of fatigue life. However, with the design factor of safety of 20 on cycles, it is noted that this is beyond expected fatigue application for the normal case.

Results

PA measurement results are reported in terms of change of the "lineshape" parameter. This parameter is a measure of the shape of the coincident photon peak at 611 keV. A higher value means the peak is higher with respect to the total count summation over all energy channels. The technique is sensitive to defect concentrations of 10^{-7} to 10^{-6}.

Initial scoping measurements were made by cold rolling 316SS in stages to 75% reduction. Room temperature PA measurements with counting statistics error limits of $\pm 2\%$ showed almost identical behavior with measurements on pure nickel. Measurements from low cycle fatigue specimens of 316SS at room temperature and 866 K (1100°F) were made next (Figure 1). The major observations from these tests are:

(1) Saturation in lineshape parameter (S) occurs at about 10% of fatigue life.

(2) At room temperature, the change in lineshape parameter from the initial condition to saturation is less for small-strain range tests than for the high-strain range tests.

(3) Much less change in lineshape parameter is measured for 866 K (1100°F) tests than for room temperature tests.

(4) Tests with several combinations of hold periods show PA saturation values equivalent to non-hold period runs although fatigue life was reduced by a factor of 4 for the tensile hold case.

Examination of the samples from the saturation regime of each test condition of Figure 1 showed that the lineshape parameter was not a unique function of the dislocation density; the final dislocation density at the higher temperature is equal to or greater than it is for room temperature The following studies established the basis for changes or lack of change.

<u>Annealing response of cold worked material</u>. Pure nickel and 316SS were evaluated to sort out effects of alloying elements in the 316SS. Two stages of annealing were defined (Figure 2). The <u>first</u>, lower temperature, annealing stage occurs when vacancies become mobile; the <u>second</u> stage is due to the disappearance of dislocations (recrystallization). The interpretations were inferred from lineshape parameter measurements and confirmed by transmission election microscopy.

<u>Annealing response of fatigued 316SS</u>. The annealing curve generated at room temperature (Figure 3a) exhibits a response similar to the room temperature curve for 25% cold rolled 316SS. However, the curves for cold rolled 316SS steadily decrease starting at 500 K and reach the well annealed level at 800 to 900 K. In contrast, annealing of a fatigued sample cycled at 866 K (Figure 3b) does not produce any significant decrease in ΔS (a normalization to the room temperature, large strain, lineshape parameter value). This indicates that vacancies created during fatigue cyclying at 866 K are mobile and diffuse to sinks leaving few residual vacancies to anneal.

Discussion of Results

The lack of PA response to dislocations in 316SS implies that dislocations either trap positrons weakly or the presence of alloying elements cause the lineshape parameter for positron annihilation at dislocations to be similar to that in the undisturbed lattice. The additional work required to investigate this phenomena is considered to be outside the objectives of this study. Alternate potential positron traps are small (.02 μm) carbides which precipitate heterogeneously during fatigue of this alloy at 866 K.

PA response to low temperature fatigue and monotonic deformation shows good sensitivity to 10% of life or 20% cold work. Since the nominal factor of safety on cyclic life is 20, knowledge of truly exceeding 10% of fatigue could be useful in low (room) temperature applications.

The experiments performed here (Figure 1) with various hold periods show that PA does not correlate with reduction of life due to creep/fatigue interaction. Samples with or without hold times produce the same PA results. This is consistent with the observation that creep alone does not provide large numbers of defects.

Conclusions

1. In 316SS cold worked or fatigued at room temperature, positron annihilation (PA) measurements primarily show sensitivity to vacancies generated during deformation.

2. In 316SS PA is insensitive to dislocation density.

3. For 316SS the PA lineshape parameter increases monotonically with damage and saturates at 20% strain for cold work and at 10% of fatigue life for low or elevated temperature. However, much lower sensitivity in terms of change in S is observed for elevated temperature than for room temperature.

4. Non-destructive monitoring of early stages of deformation near the surface is practicable, particularly at lower temperature. Potentially such near surface damage could be correlated to fatigue crack initiation.

5. The PA technique is limited to the investigation of specific points (i.e. small volumes). Measurement at a point requires about 30 minutes and thus large volumetric surveys are not practical.

Recommendations

The positron annihilation technique is not recommended for the purpose of monitoring fatigue damage or creep-fatigue interaction at elevated temperature. The enclosed report, NUREG/CR-1129, quantitatively defines the limited ability at elevated temperatures. Alternate applications, from room temperature to the temperature at which vacancies become mobile, 650 K (710°F) for 316SS, would produce results with practical sensitivity in the measured change of the lineshape parameter.

For further information on these results, please contact Dr. T. J. Walker of my staff.

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Robert J. Budnitz, Director
Office of Nuclear Regulatory Research

Enclosure: NUREG/CR-1129

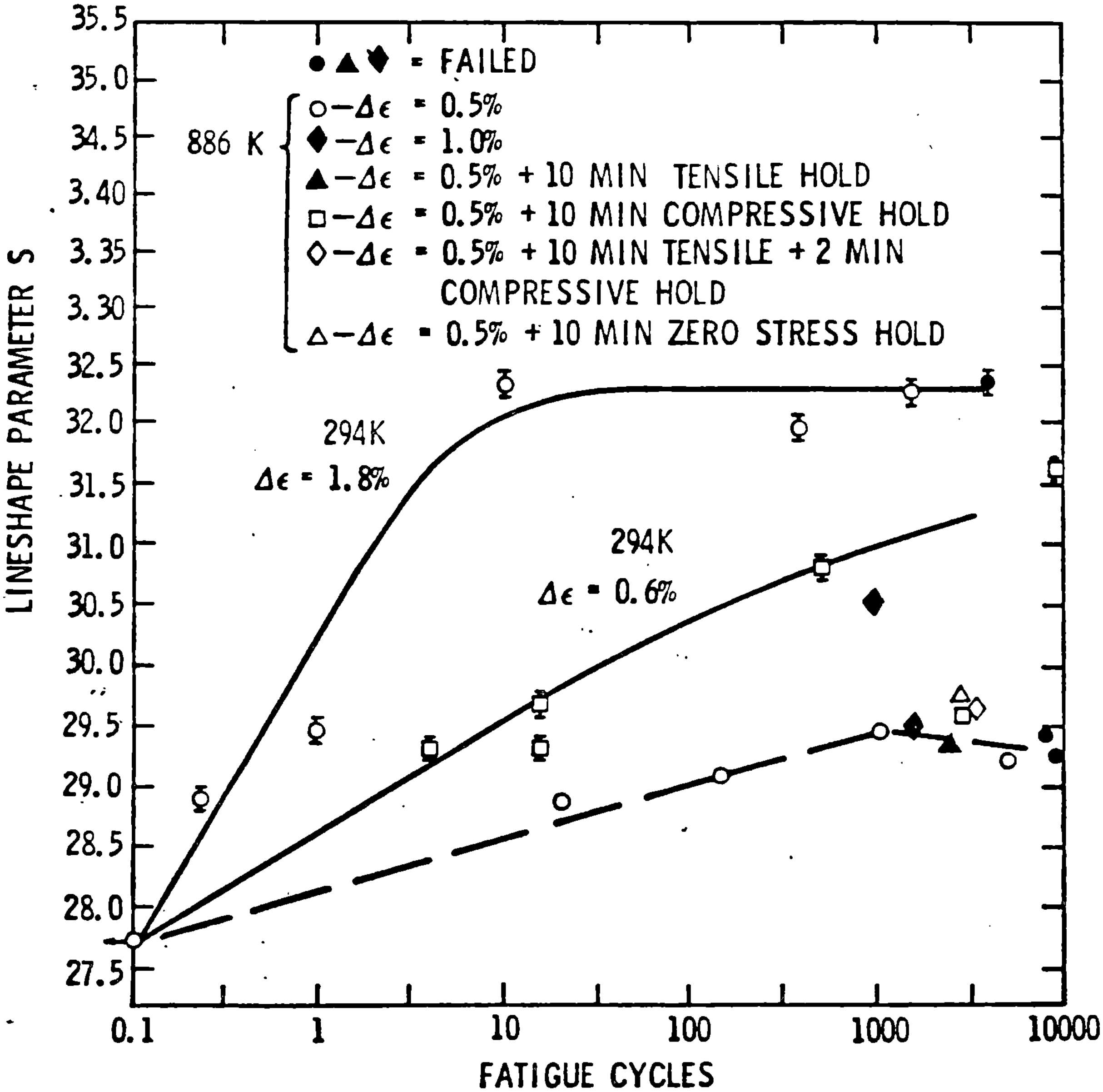
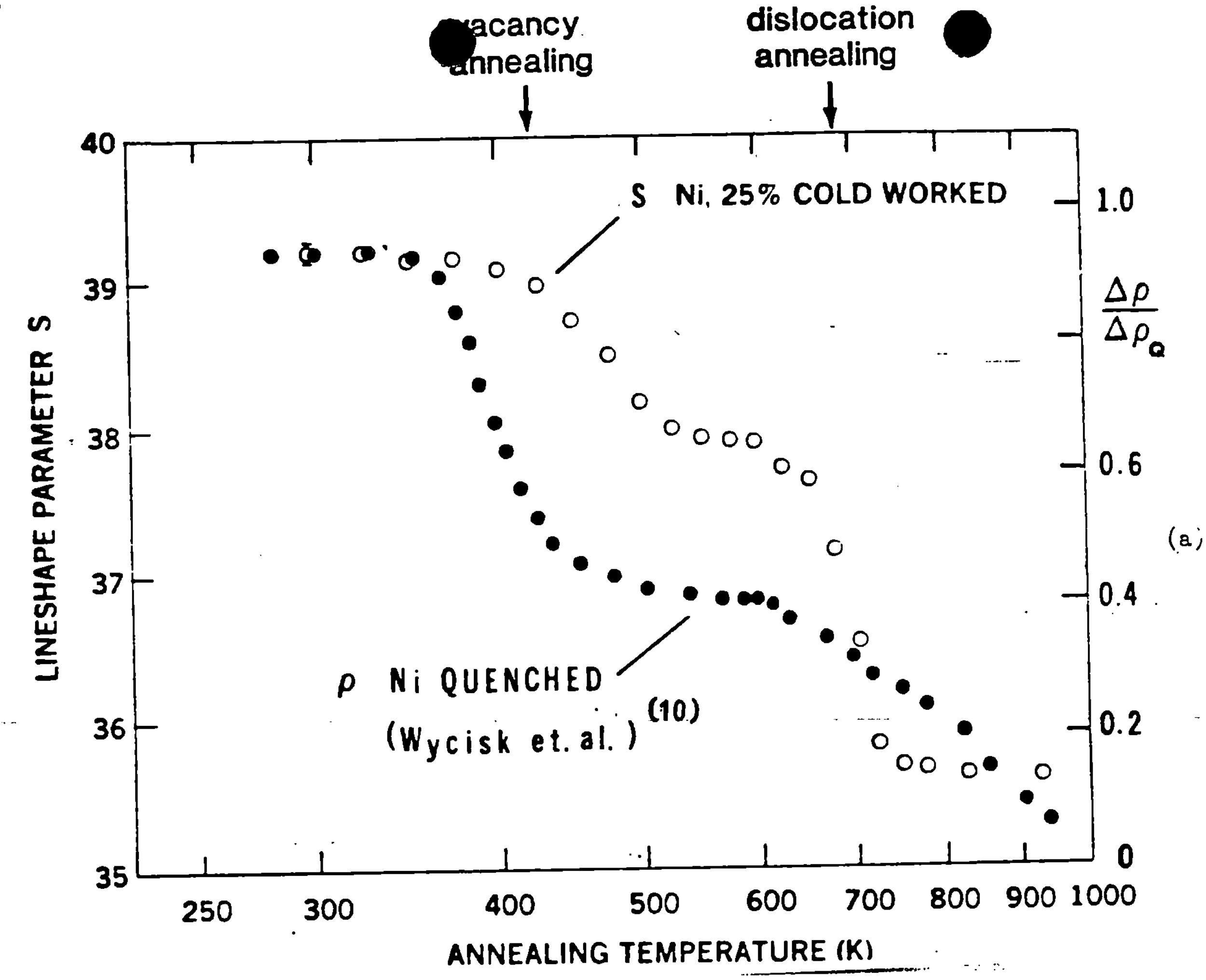


FIGURE 1. Positron annihilation response of 316 stainless steel fatigued at 293 K and 866 K.



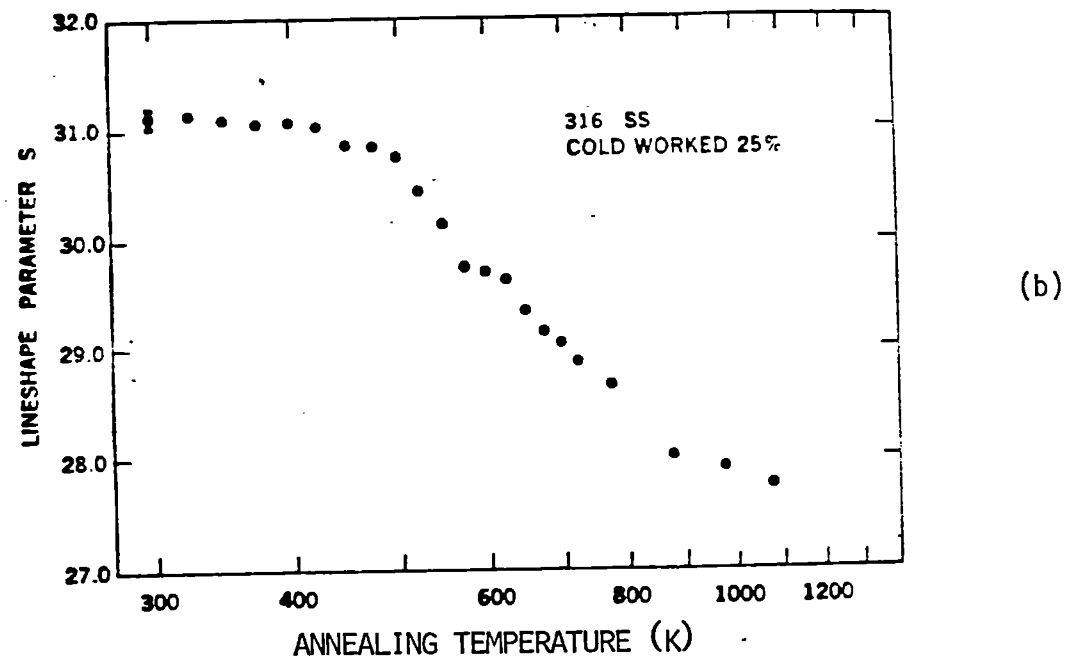


FIGURE 2. (a) Isochronal annealing response of pure nickel; S-parameter measurements of cold worked nickel with resistivity data for quenched nickel

(b) Isochronal annealing response of 25% cold worked 316 stainless steel; S-parameter versus annealing temperature

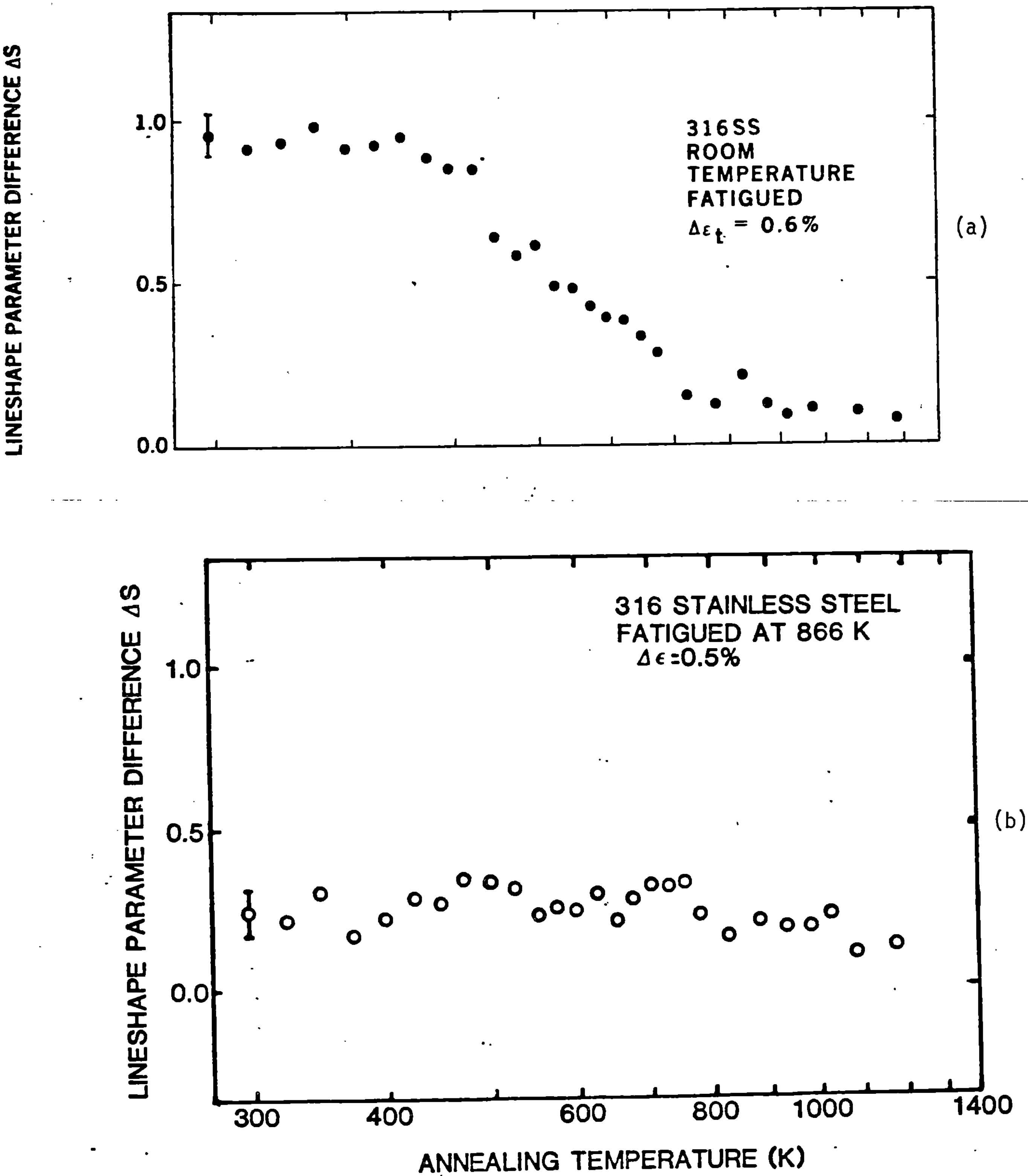


FIGURE 3. Isochronal annealing response of fatigued 316 stainless steel:

(a) fatigued 293 K, $\Delta\varepsilon$ = 0.6%, N = 47143 cycles (prior to failure);

(b) fatigued 866 K, $\Delta\varepsilon$ = 0.5%, N_f = 10078 cycles

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T. E. Murley / Acting

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Enclosure: NUREG/CR-1129

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