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POSITRON LIFETIME MEASUREMENTS AS A
NON-DESTRUCTIVE TECHNIQUE TO MINOR FATIGUE DAMAGE

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Division of Materials Science and Engineering

University of Utah

Salt Lake City, Utah 84112

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Annual Progress Report

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Project Personnel: Dr. J. G. Byrne*, Principal Investigator
Dr. R. W. Ure, Jr., Faculty Associate
Mr. K. G. Lynn, Graduate Student
Mr. F. Alex, Graduate Student

* Dr. R. W. Ure, Jr., served as Acting Principal Investigator for the academic year 1972-73,

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ABSTRACT

This report summarizes the activities of the second year of effort on applying measurements of positron lifetime distributions to characterize plastic deformation. The critical parts (electronics in the base of the photomultipliers) of the electronics have been brought under temperature control of within $\pm 0.1^\circ\text{C}$ thus minimizing drift problems. In data analysis a computer program is functioning in which there are no fixed experimental parameters. This permits much more accurate results. Samples of pure Ni and a Ni - 65% Co alloy have been fatigued to failure. Positron lifetime and X-ray particle size were measured at various fatigue lives and transmission electron microscopy was performed after failure. Good correlation was found between the three techniques and it appears that positron lifetime can be used as an effective tool to study fatigue mechanisms. It is a more sensitive non-destructive measurement than is X-ray particle size. It has been established that grain boundaries make a distinct contribution to the positron lifetime. Positron lifetime increases with plastic tensile strain in a manner dependent on initial grain size and in a manner consistent with existing work hardening theories. There are indications that the positron lifetime may be sensitive to hydrogen embrittlement or hydrogen assisted cracking (HAC) in steels.

I. INTRODUCTION

The long range objective of this study continues to be the utilization of measurements of the distribution of positron lifetimes to measure the nature and degree of damage in metals, whether the origin of the damage be cyclic or monotonic deformation, or irradiation. To reach this objective, it is essential to distinguish experimentally between point defects, line defects, complexes of point defects and other entities such as grain boundaries, stacking faults and twins. During this past year, progress has been made in the following areas: equipment, analysis, fatigue studies, grain size studies, and hydrogen embrittlement studies.

II. TECHNICAL REPORT

A. Equipment

Some important changes have been made in the fast-slow coincidence circuitry used for the measurement of positron lifetimes.

A new data acquisition system consisting of a Digital Equipment PDP-8 (small computer system), a Nuclear Data 560 Analog to Digital Converter and an ASR-33 teletype has been made operational. During a run the data are accumulated in the memory of the PDP-8. On completion of the run, the data are recorded on an x-y plotter and punched onto paper tape by the ASR33 teletype. The paper tape is then read directly into a large digital computer (the Univac 1108) for analysis.

Early data were often inaccurate because of drift in the electronics caused by temperature changes in the laboratory. To rectify this, a temp-

erature control system was built to control the temperature of the electronics. The most sensitive part of the system (the electronics in the base of the photomultiplier) has temperature control to less than $\pm 0.1^\circ\text{C}$ while the remainder of the system is controlled to within $\pm 0.5^\circ\text{C}$. More details of these modifications are given in the Technical Progress Report, September 1972, pp. 1-4, and in the balance of Section A which follows.

The sandwich consisting of the radioactive Na^{22} (10 μCi) source and two identical specimens was located in a holder built to produce a positioning accuracy of ± 0.01 inches. This allowed the sandwich to be positioned an equal distance from each of the two scintillating crystals on each of the photomultiplier tubes. Time of flight errors of the annihilating photons from the Na^{22} source were thus minimized.

The block diagram of Figure 1 describes the current setup. Cylindrical plastic scintillators of KL-236 with diameters of 1 inch and RAC 8575 photomultiplier tubes were used to detect the resulting photons.

The delayed-coincidence system employs a time to amplitude converter in which the artificial delay and the elapsed time between the emission and the annihilation of the positron is converted to a proportionate pulse. The pulse then goes through a coincidence gate into an ADC, the digitalized output of which is stored in a PDP-8 computer. These digitalized outputs are stored in finite width channels each corresponding to a particular range of pulse heights.

The distribution of time or voltage intervals is an exponential decay curve where the reciprocal of the slope is equal to the mean lifetime of the positron (when the electronic noise and background and subtracted from the spectrum).

The voltage gates on the single channel analyzers were set to 15% of the 0.51 and 1.28 MeV linearly amplified voltages. The single channel analyzers were used to decrease the number of random coincidences and to insure coincidence between the correct voltages of the voltage spectrum. If the pulses were within the specified voltages of the single channel analyzers and within 1.7 μ sec of each other at the coincidence gate, the event was considered valid and stored in the respective channel.

The time resolution of the system was determined by measuring the pulse height distribution from a Co^{60} source which emits two time coincident gamma rays of approximately equal energy. The FWHM (full width half maximum) of the Co^{60} distribution curve was typically 200 psec although it was found that a fluctuation occurred in the Co^{60} FWHM from run to run. The FWHM of the Na^{22} was larger due to the difference in energy of the gamma rays (0.51 and 1.28 MeV); i.e., there was an energy dependence of the voltage spectrum which broadened the Na^{22} spectrum by approximately 110 psec over the FWHM of the Co^{60} which typically is 220 psec. Since the FWHM of the Na^{22} was considered a floating parameter in the computer analysis both the above mentioned problems were solved explicitly.

To stabilize the electronics, the photomultiplier tubes, bases and scintillating crystals were controlled to $\pm 0.1^\circ\text{C}$ and the remainder of the fast-slow coincidence system was controlled to $\pm 0.5^\circ\text{C}$. This in turn reduced the fluctuations in the FWHM of the Co^{60} curve.

B. Analysis

A variety of computer programming techniques based on current literature and our own experimentation have been evaluated in order than an adequate

analysis of the experimental data might be accomplished. In fitting the experimental data the prompt resolution function must be unfolded and the number of exponential components determined.

The first program employed was the model of Lichtenberger, et al.⁽¹⁾ and proposed that the prompt resolution function could be unfolded and approximated by a double-sided exponential. If a Gaussian rounding at the peak is ignored, a good approximation is obtained. In our analysis it was found that ignoring the Gaussian rounding was unjustified since the most important part of the prompt curve is within 2 or 3 channels of the peak. A second problem encountered in this program was the consideration of T_0 (time zero) as a constant. Our research showed that T_0 changed from run to run and could not be fixed. As a result T_0 was made a floating parameter. A good fit, however, was still not obtained due to the Gaussian rounding of the peak.

The Lichtenberger program was then replaced by the "positronfit" model proposed by Kirkegaard and Eldrup⁽²⁾. This model assumes that the lifetime spectrum consists of a sum of exponential terms and that the prompt curve is Gaussian. With the Gaussian rounding at the peak, this assumption seemed to be more reasonable. In fact, the prompt and Gaussian curves with the same FWHM coincide to within 3% of the peak value, which are the important parts of the prompt curve. In both programs discussed so far a technique due to Marquardt⁽³⁾ is used which combines in an almost optional fashion, the method of Gauss-Newton and the method of steepest descent.

The parameters fitted in Positronfit are the annihilation rates, λ_j ; channel number equivalent to time zero, T_0 ; intensities, I_{0j}/λ_j ; and back-

ground, B. The background and the intensities are assumed linear whereas time-zero and annihilation rates are non-linear. The FWHM of the prompt curve is experimentally approximated and assumed constant.

The number of positrons annihilating at rate λ_j is assumed to be a decaying exponential function:

$$\begin{aligned} I_j(t) &= I_{0j} \exp(-\lambda_j t) & t \geq 0 \\ I_j(t) &= 0 & t < 0 \end{aligned}$$

where t is related to time by the channel number. The final result after folding $I_j(t)$ with the Gaussian is:

$$f_i = B + \sum_{j=1}^{k_0} F_{j,i}$$

with

$$F_{j,i} = \frac{I_{0j}}{2\lambda_j} \left[\gamma_{j,i} - \gamma_{j,i+1} - \operatorname{erf}\left(\frac{t_i - T_0}{\sigma}\right) + \operatorname{erf}\left(\frac{t_{i+1} - T_0}{\sigma}\right) \right]$$

and

$$\gamma_{j,i} = \exp \left[-\lambda_j (t_i - T_0) + \frac{\lambda_j^2 \sigma^2}{4} \right] \left[1 - \operatorname{erf}\left(\frac{\lambda_j \sigma}{2} - \frac{t_i - T_0}{\sigma}\right) \right]$$

where

$$\sigma = \frac{\text{FWHM}}{2\sqrt{\ln 2}}$$

In this model the FWHM of the prompt curve is not included as a fitting parameter.

In applying this program to our data it was found that the FWHM is strongly coupled with the lifetimes (Figure 2). As a result the initial determination of the FWHM is very critical and assumes no fluctuations in the electronic noise or the prompt curve. Even with good temperature control ($< \pm 2^\circ\text{F}$), fluctuations of ± 10 psecs in the FWHM occurred during successive runs in the equipment. These fluctuations caused the lifetimes to vary depending on the initial value of the FWHM used.

With these difficulties in mind the solution seemed to point to a modification of Positronfit that would include FWHM as a non-linear fitting parameter. Upon completion of this alteration τ_0 and FWHM were found to be strongly dependent on one another. This strong dependency caused Marquart's algorithm to be inadequate in resolving all of the parameters. The net result was that although all of the parameters were fitted, the results were dependent upon the initial approximated values of the parameters introduced into the program. The modified program was simply not adequate in solving this problem.

It was then ascertained that a good fit depended on the initial approximation of FWHM. Therefore, when the value of FWHM was approximated correctly the best fit between the original and fitted spectrum were obtained. Fits were measured by $\phi_{\min}$ (Marquart's parameter) or Variance of Fit (Kirkegaard and Eldrup) the later being directly related to $\phi_{\min}$. Marquart's parameter ϕ is:

$$\phi = \sum w_i (y_i - f(b))^2$$

with $f(b)$ representing the count rate in channel No. i predicted by the

model. Y_i is equivalent to the number of counts in channel no. i . The weighting factor equal to w_i in the model is represented by $1/Y_i$. $\phi_{\min}$ is found to correspond to the best fit by adjusting the linear and non-linear fitting terms. The Variance of Fit is defined as:

$$S^2 = \frac{\phi_{\min}}{q}$$

where $q = n - k_{\text{free}}$. n is equivalent to the number of channels analyzed and k_{free} to the number of estimated free intensities and lifetimes in the parameter vector b . Since Variance of Fit varies in a near parabolic manner when plotted with FWHM, it is possible to find the minimum value of the parabola. After the minimum value of the Variance of Fit is calculated, the corresponding lifetimes can be extracted. (See Figure 3). This was accomplished by leaving the same fitting parameters in "Positronfit" but using it as a function routine.

The final modified program has a new driver program which calls an external function routine termed $X_{\min}$. $X_{\min}$, in turn, calls Positronfit as a function routine where $x_{\min}$ minimizes the amount of computer time needed to find the minimum Variance of Fit with a specific error externally specified.

This modified program has been completed and seems to do a much better job in analyzing the data. A further advantage of this program is that there are no fixed parameters in the analysis and the results are independent of the initial input. To our knowledge this is the only program in the field where there are no fixed experimental parameters in the analysis.

C. Fatigue Studies in Ni and a Ni - Co Alloy

Introduction

Recently it has been established that vacancies and dislocations increase the lifetime of a positron in a deformed metal⁽⁴⁻⁷⁾. For example, a charge redistribution and a consequent electric dipole are created by the strain field which occurs around an edge dislocation⁽⁸⁾. The negative part of the dipole repels electrons and has a coulombic attraction for the positron. As shown by Brown⁽⁹⁾ there is a redistribution of conduction electrons at the core of a dislocation. Due to this redistribution and attraction to the positron a bound state may be formed between the positron and dislocation core, thus increasing the mean positron lifetime.

Adamenko, et al.⁽¹⁰⁾ performed angular correlation of positron annihilation photon experiments in deformed nickel alloys. These researchers found a change of the angular distribution curve from the annealed to the highly deformed state. It was suggested due to the change that in the highly deformed state that annihilations occur when the positron is in the core of the dislocation. Grosskreutz and Millet⁽¹¹⁾ performed positron experiments on fatigued Al and Cu at room temperature. After fatiguing, these investigators found an increase in the mean lifetime of 54 and 82 psec for Al and Cu, respectively. It was concluded that the enhancement in the lifetime had its origin in the dislocation network.

The current research is aimed at developing a procedure whereby positron annihilation may be used as a completely nondestructive method for detecting early fatigue damage. X-ray line broadening analysis and transmission electron microscopy have also been used to characterize fatigue damage in Ni and a 35% Ni - 65% Co alloy.

In the Fourier analysis of X-ray line broadening⁽¹²⁾, the structure of cold-worked metals is described in terms of small coherently diffracting domains within which there may be microstrains. The particle size and microstrain are closely related to the dislocation density and arrangement⁽¹³⁾ and hence are of interest in connection with the current positron annihilation results.

Experimental Procedure

The pure nickel and nickel-cobalt alloys used in this research were obtained from the International Nickel Company. Nickel (99.99+% purity) and a binary 35% Ni - 65% Co alloy were machined into constant stress plane bending fatigue specimens. These samples were annealed in argon at 600°C for one hour and furnace cooled to give grain sizes of 1.1×10^{-3} inches and 7.3×10^{-5} inches for the Ni and the 35% Ni - 65% Co alloy, respectively. A Sontag SF-2-U fatigue machine with a fixed frequency of 1800 cpm was employed for cycling. All specimens were electropolished after annealing with a Boilman solution. Both samples were fatigued at a maximum bending stress of 45 ksi.

The positron lifetime data were analyzed as discussed in Section C.

The data were corrected for a linear background level and annihilation times appropriate for both the annihilations in the mylar and active sodium salt. Fatigue specimens were measured for 20.0 hours and had approximately 7.5×10^5 coincident events accumulated during this time. The fitted background to peak height ratio was 7.5×10^3 counts.

All X-ray measurements were performed with a G.E. XRD-5 unit as described in an earlier paper⁽¹³⁾. After fatiguing the specimens a specified

amount, positron annihilation and X-ray data were taken. The same procedure was repeated at various intervals until the specimen fractured.

The Warren-Averbach Fourier analysis⁽¹²⁾ was used for the comparison of the X-ray peaks in the standard annealed condition to that of the fatigued condition. A computer program by DeAngelis⁽¹⁴⁾ was used for calculating the Stokes-corrected Fourier coefficients of the broadened k_{α_1} profile. From these profiles the particle size and root mean square microstrains were obtained according to Warren's⁽¹²⁾ derivation.

Transmission electron microscopy (TEM) specimens were taken from the maximum stress surface region. Both electropolishing and jet polishing techniques were used to thin the material. All foil specimens were examined with an RCA EM-U 3 6T electron microscope.

Results

The major part of the fatigue damage in cyclic bending is concentrated in regions close to the surface of the specimen where bending stresses are greatest. In the present case the deformation was plastic and thus the principal defects are dislocations. At 295°K when vacancies are in thermal equilibrium their contribution to the positron lifetime is negligible⁽¹⁵⁾. Grosskreutz and Millet⁽¹¹⁾ also concluded for copper and aluminum that the observed enhancement in lifetime had its origin in the dislocation network and not in vacancies induced during fatigue. Crack initiation and first stage crack propagation generally occur in the specimen surface, and since most of the positrons annihilate near the surface regions, measurements of annihilation times should be a valuable non-destructive method for studying fatigue damage.

The positron lifetimes were found to have large changes during the initial cycling stages of both the Ni and Ni - 65% Co as can be seen from Figures 4 and 5, respectively. The mean positron lifetime increased with increasing number of cycles until saturation. As was mentioned earlier this indicates that positrons are being trapped in the defects of the sample, where the traps cause the positron lifetime to increase.

The total change of the mean lifetime was 86 psec for pure Ni and 93 psec for the Ni-Co alloy. The difference in the fatigue life noted in Figures 4 and 5 between Ni and the Ni-Co alloy, respectively, is largely attributable to the change in stacking fault energy (SFE). The importance of the SFE is due to the importance of dislocation cross slip. The basic idea concerning the effect of SFE on fatigue life is that a low SFE material, the Ni-Co alloy, permits a wider separation of partial dislocations than a high SFE material, pure Ni. In the case of the 35% Ni - 65% Co alloy it is then harder to force the two partial dislocation together to permit cross slip of the dislocation. According to Backofen, et al.^(16,17) the stacking fault energy influences the fatigue life more than the yield strength of the material.

In the Ni and Ni - 65% Co alloy specimens, the slope changed drastically approximately at 7000 and 22,800 cycles, respectively (Figures 4 and 5). This saturation occurred after approximately 7% of the total fatigue life in both samples. In angular correlation studies of Fe cold rolled at room temperature, Snead, et al.⁽¹⁸⁾ found a tendency toward saturation of the angular correlation curve between 8 and 16% reduction of thickness. These investigators also noted the rate of increase in the curves to be approxi-

mately linear up to deformation of 8%. Saturation effects have also been observed in plastically deformed Ni⁽¹⁹⁾. In this study it was found that the angular correlation for Ni deformed 45% was nearly identical to that for Ni deformed 16%.

We will now describe the TEM structures in the maximum-stress surfaces for our Ni and Ni - 70% Co alloy specimens. After fracture in Ni, the density of dislocations is higher and bands composed of elongated rectangular cells are found. The size of these cells is approximately 2000 Å long by 1000 Å wide. This is approximately equal to the cell size obtained from the X-ray measurements by Moll and Ogilvie⁽²⁰⁾ for Ni fatigued at the same stress level on the same type of fatigue machine.

After fatigue failure in the 65% Co alloy a much higher dislocation density relative to the annealed condition was again observed and cells close to 500 Å in diameter were found, which value again is close to the X-ray particle size (approximately 460 Å) in this material at fracture.

In the X-ray line broadening study, the particle size in the $\langle 111 \rangle$ direction in the Ni - 65% Co (Figure 6) decreased from 1600 Å at 10,000 cycles to 440 Å at 400,000 cycles (prior to fracture). In the $\langle 100 \rangle$ direction the particle size decreased from 800 Å to 480 Å as shown in Figure 7.

The root mean square strain normal to the reflection planes were also measured in the Ni - 65% (Figure 8). The root mean microstrains $\langle \epsilon^2 \rangle$ were independent of the crystallographic orientation. As shown by Williamson and Smallman⁽²¹⁾ and Smallman and Westmacott⁽²²⁾ the dislocation density can be calculated in terms of the particle size and microstrain measured by X-ray analysis. In the absence of extensive pile-ups or polygonization the dislocation density ρ is given by:

$$\rho = \frac{2\sqrt{3} \langle \epsilon^2 \rangle^{1/2}}{Db}$$

where $\langle \epsilon^2 \rangle$ is the mean squared microstrain, D is the particle size and b is the Burger's vector of the dislocation. The Burger's vector being taken in the $\langle 111 \rangle$, Figure 9 shows how the dislocation density, calculated by the above relationship, varies as a function of fatigue.

Conclusions

1. TEM and X-ray observations during fatigue gave qualitative agreement with the positron annihilation data obtained for Ni and a Ni - 65% Co alloy.
2. It appears that positron annihilation lifetime data can be used as an effective tool for studying fatigue mechanisms. Since the trapping rate is directly proportional to the defect concentration, the researcher may use positron annihilation as a non-destructive method for calculating dislocation density.
3. The X-ray particle size saturated at approximately the same number of cycles at which the mean positron lifetime changed slopes. This could mean that the defect concentration or dislocation density reached saturation before the trapping rate had saturated. This particular aspect needs to be studied more thoroughly.

D. Grain Size Effects in Cu

Introduction

Since the grain size of a metal determines to a great extent the degree of work hardening during deformation it is essential that we understand how positrons interact with grain boundaries before and during deformation. The

only literature citation concerning the existence of a grain boundary - positron interaction is a brief mention by Weisberg and Berko⁽²³⁾ that different lifetimes were noted for single and polycrystals of each of Fe and Be. The current phase of study therefore was aimed at measuring the positron lifetime in Cu specimens of different grain sizes before, and after increasing amounts of tensile strain.

Experimental Procedure

Flat tensile specimens 1.3 mm thick were machined from 99.999+% Cu which had been unidirectionally cold rolled 90.7%. Specimens were annealed for times of 1, 5, 25 and 30 minutes at 600°C in an Ar atmosphere and slowly cooled to ambient temperature in Ar. The resulting recrystallized grain diameters were 3.3, 169, 264, and 309 μ , respectively. The positron lifetime also was determined for a Cu single crystal of the same purity as the polycrystals. Positron lifetime data were gathered and analyzed as described earlier in the current report. Tensile deformation was applied with an Instron machine at a strain rate of 0.0125 min⁻¹.

Results

Table 1 shows that the positron lifetime increases steadily from about 157 to 176 psec as one goes from a single crystal to progressively finer grain size material. This indicates that grain boundaries act as trapping sites for positrons, increasing the mean positron lifetime.

The behavior of the positron lifetimes of the 169, 264, and 309 μ grain sizes was in accordance with existing work hardening theories in which the dislocation density increases faster the smaller the grain size since for a given total strain a smaller grain size should experience more dislocation

Table 1

Effect of Grain Boundaries on
Positron Lifetime in 99.999% Copper

Average Grain Size	(τ_1) psec (mean lifetime)	Treatment
Single Crystal	157	Melt grown
309 μm	162	Cold rolled 1300% and annealed 30 minutes at 600°C
264 μm	164	Cold rolled 1300% and annealed 25 minutes at 600°C
169 μm	173	Cold rolled 1300% and annealed 5 minutes at 600°C
3.0 μm	176	Cold rolled 1300% and annealed 1 minute at 600°C

generation and interaction with consequent increased positron trapping. This can be seen from Figure 10 where the mean lifetime increased with increasing defect concentration, but saturation began at a concentration dependent upon the trapping rate and the grain size. The 169, 264, and 309 μ grain size started to saturate at approximately 10, 14, 15%, respectively. These results are similar to Kusmiss, et al.⁽⁴⁾ where saturation occurred somewhere between 8 and 16% deformation of Pt.

Since trapping at dislocations should be similar to that at vacancies, the present trapping models of Bergensen and Scott⁽²⁴⁾, Connors and West⁽²⁵⁾, and Seeger⁽²⁶⁾ should be applicable. In the trapping models two lifetimes are expected; one for perfect material, τ_f , and one for trapping, τ_t , which do appear at strains before saturation. Table 2 shows the intensity of the two lifetimes as a function of strain in the 264 μ grain size Cu. These intensities have been corrected for the annihilations in the active sodium salt and the mylar of the positron source. The positron lifetime in the strain free lattice, τ_f , was found to be 157 psec in the single crystal and the trapped positron lifetime, τ_t , was 230 psec in the 40% deformed sample. This is assuming that for τ_f there are essentially no trapped positrons in the single crystal and for τ_t that essentially all of the positrons are trapped at saturation. Then τ_f and τ_t were considered fixed and the intensities were taken as floating parameters.

The variance of fit improved from or was equal to that of the previous analysis (Section 8) where only a single lifetime - the mean lifetime was used. This suggests that the present trapping models are applicable.

Table 2

Percents of Positron Annihilations in the Lattice (τ_f) and at Defects (τ_t) for
the 264 μm Grain Size Material

% Strain	0	5	10	15	20	40
Intensity τ_f	96.3	71.2	37.0	4.4	0	1.1
Standard Deviation τ_f	2.70	2.57	2.45	2.52	2.18	2.41
Intensity τ_t	3.7	28.8	69.0	95.6	100	98.9
Standard Deviation τ_t	5.27	5.05	4.15	4.18	3.53	4.12
Variance of Fit	.930	1.029	1.362	1.029	1.129	1.508

In the present case of plastic deformation, the principal defects are dislocations. As shown by Kuribayashi, et al.⁽²⁷⁾, excess concentrations of vacancies in copper above the saturation produced by the non-conservative motion of jogs of dislocations had a small effect on the angular correlation of positron annihilations. Grosskreutz and Millet⁽¹¹⁾ also concluded for copper that the observed enhancement in lifetime had its origin in the dislocation network and not in vacancies.

The change in mean positron lifetime with grain size indicates that, when researchers using polycrystalline specimens attempt to determine vacancy formation energy, by the present trapping models^(24,25), the grain diameter should be included. Further, if grain growth occurs, the change in total grain boundary area should be considered. This would only be needed for higher accuracy since the effects on the lifetime data would be small.

With the 3.3 μ grain size behavior during straining was found to be anomalous to any existing theories. The positron lifetime in this sample did not reach 230 psec until approximately 42% strain, near fracture. The only explanation that can be presented at this writing is that the strain was not homogenous, or small amounts of grain boundary sliding occurred during straining. If grain boundary sliding did occur, this will keep the dislocation density at a much lower level than is expected for specific strains. Since grain boundary sliding should not occur at room temperature even with this small grain size, the first explanation seems more reasonable.

E. Hydrogen Embrittlement

Introduction

One of the significant problems affecting high strength steels is absorption of hydrogen in nascent form. This results in a serious change in the mechanical properties of the steel. This phenomena has been called "hydrogen embrittlement", although several authors suggest that the steels are really not more brittle. Therefore, we prefer the term hydrogen assisted cracking (HAC).

HAC or "hydrogen embrittlement" is a reversible, or temporary, damage which occurs when a material is exposed to a source of nascent hydrogen. It results in a loss of ductility, ultimate tensile strength, and notch toughness when an affected material is loaded to stresses below the yield strength for sustained periods. Properties may appear quite normal when specimens are tested under normal strain rates, but damage manifests itself by sudden failures at stresses below ultimate tensile strength after some time delay or "incubation period".

The amount of hydrogen in the steel which we are considering here are not the large amounts which result from exposure of the steel to hydrogen at high temperatures and/or pressures and which produce permanent loss of mechanical properties as a result of decarburization, intergranular cracking, and blistering. The phenomena discussed here are the result of small amounts of hydrogen in the ppm range which are absorbed by the steel during plating or chemical treatments in which nascent hydrogen is present.

The form and location of the hydrogen in the metal is still unknown. The mechanism by which hydrogen causes cracking is also unproven. In

addition, no method has been found until now to detect the hydrogen in situ in steel. The methods to detect hydrogen or its damage involve mass spectrographic analysis of gases evolving from a specimen being fused in a vacuum or placing specimens believed to be "embrittled" under stress and measuring time to failure.

We have measured the positron annihilation lifetime in steel samples which have been "hydrogen embrittled" and have found changes in the lifetime which appear to correlate with the "hydrogen embrittlement".

A series of tests was performed using standard HEP* specimens which were treated in a variety of ways describes in the tests and results section of this report. The purpose of this series of tests was to:

- 1) Determine stability of existing equipment and reproducibility of results.
- 2) Determine whether absorption of hydrogen causes a change in the positron lifetime and the extent of such a change.
- 3) Determine if the positron lifetime in cadmium is different than in steel and the depth at which positrons annihilate.
- 4) Determine whether the measurable effect, if any, dissipates with time in the presence of a dense, bright cadmium plating on the surface and with this cadmium removed.
- 5) Determine if measurable effects can be removed by low temperature bakes and if the presence of a bright cadmium on the surface hinders this.

*Manufactured by Associated Spring Corporation, Wallace Barnes Division, Bristol, Connecticut.

6) Determine if glass blasting mechanically distorts the material near the surface enough to be measured by an increase in positron lifetime and whether this effect can be removed by low temperature bakes. Approximately thirty tests were conducted using the fast-slow coincidence circuit in the configuration described in the Technical Progress Report for Contract No. AT(11-1)-2128 dated September 1972. A Na^{22} source of approximately 10 μCi strength was placed between two strips comprising each specimen. The scintillation crystals were placed as close to the sample as possible and runs made for approximately 16 hours. Between 750 and 850 counts per minute were obtained for most the tests. The peak count to background ratio was 8,000:1 or greater.

Results

Each test employed two HEP test strips from batch 0167. These strips were prepared as required for each specific test, cut in half with one half being used as the primary specimen and the other half being set aside for repeat tests, if necessary.

The HEP specimens were 0.0215" thick by 0.469" wide by 7.47" long strips conforming to AISI type 1070 or 1075 steel. Composition of this specific batch is given in Table 3. They were heat treated to a hardness of Rc 52.

The source was made from a solution consisting of Na^{22}Cl in water. An appropriate amount of diluted solution was deposited and sealed between two 0.002" thick sheets of mylar.

All lifetimes given in this section refer to the short component and are given in picoseconds (psec).

Table 3
Chemical Composition (%)

C	< 0.75
Cr	< 0.01
Ni	0.01
Si	0.12
Mn	0.80
Cu	0.03
Mo	< 0.01
V	< 0.002

Table 4
Rerun of Standard

Test	Date	Lifetime
1a	3 Oct	178.1
1b	7 Oct	176.4
1c	11 Oct	189.3
1d	16 Oct	180.1
1e	22 Oct	177.3

1. Stability. In order to determine how stable or reproducible the equipment was, two "as received" HEP specimens were used as a standard and rerun every third or fourth test. Table 4 is a tabulation of lifetimes derived on various days.

It should be noted that the drastic temperature excursion of the equipment occurred on October 11. If this result is neglected, the maximum variation from lowest to highest reading is 3.7 psec. The arithmetic average is 177.9 with a maximum variation of less than ± 2 psecs.

The unweighted average of all measurements gives:

$$L = \frac{1}{N - 1} \sum (L - \bar{L})^2 = 177.9 \pm 1 \text{ psecs.}$$

This is in good agreement with lifetimes of 170 ± 20 and 171 ± 4 psecs. reported by Jones and Warren⁽²⁸⁾, and Weisberg⁽²⁹⁾, respectively, using the mean lifetime of aluminum as a reference. It is also in fairly good agreement with values of 160 ± 5 obtained by Weisberg⁽²⁹⁾ when using the slope method.

Reproducibility is also demonstrated by results obtained by rerunning an electropolished and bright cadmium plated set of strips, sample 26, as reported later. If test 26d, which appears to be erratic, is neglected, the maximum variation from high to low reading is 3.8 psec.

It should also be noted that all electro polished specimens after stripping, except Test 4, gave fairly consistent results with a maximum variation of 5.6 psec, see Table 5.

2. Electropolishing. All specimens to be cadmium plated were prepared by either electropolishing or glass blasting. The following

Table 5
Results of Electropolished Specimens

Test	Date	Lifetime
2	4 Oct	192.0
4	9 Oct	178.8
28	17 Oct	192.3
30	19 Oct	188.8
32	21 Oct	194.4

Table 6
Reruns of Cadmium Plated Sample

Test	Date	Lifetime
26a	12 Oct	190.6
26b	13 Oct	188.6
26c	15 Oct	192.4
26d	21 Oct	199.3

solution and conditions were used for electropolishing:

Electropolishing Solution:

10 parts 99.1% $C_2H_4O_2$
1 part 60% $HClO_4$

Current:

1.5 amps/in²
Anode, Specimen
Cathode, 300 series stainless steel

Temperature:

Approximately 70°F

Time:

1 minute

All specimens were rinsed with distilled water following all polishing and plating operations and wiped dry with a soft tissue. Except for those kept in liquid nitrogen until testing, all were wrapped in tissue and stored subject to ambient environmental conditions.

Test 2, involving an electropolished specimen gave a short lifetime of 192.0 psecs; an increase of 14.1 psecs over the mean lifetime of the "as received" standard.

Sample 2 was then baked at $400 \pm 5^\circ F$ for 24 hours. The lifetime was then determined (Test 5) to be 180.9 psecs which is within experimental error of the "as received" condition.

3. Cadmium Plating. One set of strips, sample 3, was first electropolished and cadmium plated to a very fine finish using the following electroplating solution and conditions:

Electroplating Solution:

CdO	3.5 oz/gal
NaCN	16 oz/gal
NaOH	1-2 oz/gal

Current:

0.24 amps/in²

Temperature:

Approximately 70°F

Time:

7 minutes

This resulted in a thickness of 0.0035" per surface.

The test (Test 3) was started within three hours of plating and yielded a lifetime of 194.2 psecs.

When required to be stripped, all cadmium was removed by immersing specimens in a 10% solution by weight of NH_4NO_3 in water and rinsing in distilled water.

The cadmium was stripped from sample 3 four days after plating and the sample run (Test 4). A lifetime of 178.8 psecs was obtained.

In order to determine if the effect of hydrogen dissipates with time, another set of specimens was prepared similarly to sample three with the following exceptions:

- 1) Prior to plating, the sample was chemically embrittled by subjecting it to a 1:1 mixture of concentrated HCl and water for one minute.

- 2) 1 gm/gal of sugar was added to the electroplating solution given above to further enhance the tendency towards embrittlement. Sample 26 was then run periodically and gave the results listed in Table 6.

On stripping this sample and testing (Test 32), a lifetime of 194.4 was obtained. This tends to indicate that Test 4 was in error as the effect of hydrogen is still present. Test 26d is also somewhat high.

Since Test 32 failed to substantiate results of Test 4, another set of specimens was plated identically to sample 26, cut in half and placed in liquid nitrogen. It was kept in liquid nitrogen until just before Test 28 was begun, at which time it was stripped and run. The resulting lifetime was 192.3 psecs confirming results of Test 32.

Rinebolt⁽³⁰⁾ and Raring and Rinebolt⁽³¹⁾ report that cadmium plating resulting in delayed fractures as low as 50% of the notched tensile strength while cathodically charged specimens failed at stresses as low as 16% of the notched tensile strength. In order to see if cathodic charging produces a greater increase in lifetime, another set was prepared by electropolishing and cathodically embrittling in a 4% by weight solution of H_2SO_4 in water at a current density of 0.15 amps/in². The specimens were then kept in liquid nitrogen until just before testing. A misrun resulted and the specimen was not tested until after it was at room temperature for seven days (Test 30). This gave a lifetime of 188.3 which is nonconclusive.

4. Annihilation in Cadmium. The effect of cadmium on positron lifetime was determined by the results of the following tests as compared to the results from some of the tests on cadmium plated specimens above.

At the time of preparation of sample 3, a second set was prepared identically and baked at $400 \pm 5^\circ F$ for 24 hours. Test 6 yielded a lifetime of 186.7 psecs for a decrease of 7.5 psecs as a result of baking.

The specimens from this test were then stripped and tested (Test 34) resulting in a lifetime decrease of 11.1 psecs to 175.6.

A 0.0007" thick film was placed on each side of the source so that they were between the source and the two strips comprising sample 1. Test 8 indicated an increase in lifetime of 20.2 psecs to 198.1.

A set of glass blasted specimens was electroplated with 0.0025" of cadmium per surface. Upon testing (Test 27), a lifetime of 206.9 psecs resulted.

When an extra 0.007" thick layer was interposed between the source and the specimens from Test 27, the run (Test 31) resulted in a lifetime of 220.8 psecs. Figure 11 is a graph of lifetime versus thickness of cadmium.

In order to determine if the decrease when resulted in Test 6 by baking was due to some change in the cadmium produced by baking, the 0.0007" thick layers were baked at $400 \pm 5^{\circ}\text{F}$ for in excess of 24 hours. These were then interposed between the source and strips comprising sample 1 and run (test 33). A lifetime of 203.1 resulted which is 5 psecs higher than Test 8 but still within the realm of experimental error.

5. Glass Blasting and Vacuum Cadmium Plating. All glass blasting was done using 200 to 375 mesh spherical glass beads.

One set of specimens was glass blasted and tested (Test 9) giving an increase in lifetime of 17.8 psecs to 195.7.

In order to determine if the effect of this blasting could be relieved, samples from Test 9 were baked at $400 \pm 5^{\circ}\text{F}$ for 24 hours. Test 25 gave a lifetime of 180.9 which is within experimental error of the lifetime in the "as received" standard.

A glass blasted set of strips was vacuum cadmium plated to a thickness of 0.00025" per surface. Test 10 gave a lifetime of 192.1 psecs.

Discussion

A summary of all test results of this series is given in Table 7. These results are those which occur when the raw data is deconvoluted by a slope type computer program similar to that provided by Lichtenberger and Stephens⁽³²⁾. It was modified by Lynn⁽³³⁾ and improved according to suggestions of Goland and Hall⁽³⁴⁾ so that absolute lifetimes result.

Although the results section is concerned with the short lifetime only, the short-short lifetime and three component lifetimes and their intensities are listed in Table 7. The short-short lifetime is supposedly related to the resolving ability of prompt events. By comparing the short and short-short lifetimes of measurements from a single sample, it can be seen that this lifetime varies in a similar fashion to the short lifetime. Table 7 lists the short lifetimes obtained from repeating sample 1 in order of ascending value. The short-short lifetimes also vary directly to them.

By comparing the short to short-short lifetimes for all tests in Table 8 it is noted that although not always the case, there is a trend for tests with longer short lifetimes to exhibit longer short-short lifetimes.

No correlation exists between the short lifetime and the intermediate and long lifetimes. We would expect the long lifetime to be related to the source and possibly surface effects. Since the same source was used throughout, for those tests where the surface is not altered, we would expect similar long lifetimes. This was not the case.

Table 7
Summary of Results

TEST ⁵	DATE ⁶	SHORT - SHORT LIFETIME ¹	INTENSITY ²	SHORT LIFETIME ¹	INTERMEDIATE INTENSITY ²	LIFETIME ¹	LONG INTENSITY ²	LIFETIME ¹	FITTING FACTOR ³	PEAK MAXIMUM ⁴
1a	3 Oct	149.5	0.756	178.1	0.202	628.2	0.040	2557	916	224
1b	7 Oct	148.4	0.750	176.4	0.201	588.7	0.049	2280	856	224
1c	11 Oct	160.7	0.826	189.3	0.139	741.1	0.035	2521	1474	225
1d	16 Oct	151.3	0.763	180.1	0.192	613.8	0.045	2365	893	224
1e	22 Oct	148.9	0.752	177.3	0.201	603.9	0.049	2243	700	223
2	4 Oct	154.4	0.809	192.0	0.156	703.8	0.035	2681	1584	223
3	5 Oct	160.8	0.724	194.2	0.226	583.4	0.049	2336	659	223
4	9 Oct	150.1	0.779	178.8	0.178	653.0	0.043	2382	777	224
5	9 Oct	152.0	0.793	180.9	0.169	670.2	0.038	2605	1102	224
6	7 Oct	154.5	0.699	186.7	0.241	540.6	0.059	2051	611	224
8	8 Oct	162.2	0.666	198.1	0.271	513.9	0.063	1948	486	224
9	6 Oct	159.0	0.828	195.7	0.141	748.5	0.031	3040	1459	223
10	18 Oct	159.2	0.750	192.1	0.200	606.9	0.050	2375	568	224
24	10 Oct	164.8	0.763	197.8	0.193	622.0	0.045	2365	813	224
25	15 Oct	151.9	0.762	180.9	0.191	619.4	0.047	2335	744	224
26a	12 Oct	158.8	0.716	190.6	0.230	565.9	0.053	2241	664	226
26b	13 Oct	157.0	0.680	180.6	0.256	513.9	0.065	1966	450	224
26c	15 Oct	159.1	0.712	192.4	0.231	543.0	0.057	2054	613	224
26d	21 Oct	165.8	0.768	199.3	0.191	643.2	0.040	256	1044	222
27	16 Oct	168.4	0.546	206.9	0.383	453.6	0.070	1960	258	225
28	17 Oct	155.3	0.810	192.3	0.153	727.2	0.036	2635	744	224
29	19 Oct	164.3	0.688	199.3	0.253	531.7	0.065	1966	450	224
30	26 Oct	151.8	0.817	188.3	0.148	722.0	0.034	2735	2289	223
31	25 Oct	176.1	0.647	220.6	0.292	502.9	0.061	1980	463	222
32	21 Oct	157.3	0.829	194.4	0.144	773.8	0.028	3512	2000	222
33	24 Oct	167.0	0.729	203.1	0.220	584.3	0.051	2322	916	222
34	23 Oct	146.7	0.734	175.6	0.208	558.5	0.057	2091	640	223

NOTES:

1. In 10^{-12} seconds, rounded off to four significant figures
2. Fraction of total events attributed to this lifetime, rounded off to third decimal place
3. Summation of differences of actual count in each channel from theoretical fit
4. Channel number in which maximum count occurs
5. Numbers omitted were tentatively planned but not run in this series
6. Date during which test was started; runs were made for approximately 16 hours

Table 8
Short versus Short-Short Lifetimes

Test	Date	Short Lifetime	Short-Short Lifetime
1b	7 Oct	176.4	148.4
1e	22 Oct	177.3	148.9
1a	3 Oct	178.1	149.5
1d	16 Oct	180.1	151.3
1c	11 Oct	189.3	160.7

The above might tend to indicate either the resolving ability is changing and affecting the short lifetime or the computer program is not separating the lifetimes adequately.

The current tests gave a reproducibility of ± 2 psecs, as did those in July 1972. The increase in lifetime attributed to cadmium was also the same but the increase in lifetime due to hydrogen is now established at 14.1 ± 2 psecs versus 17 to 21 in July. It should be noted, however, that the program has been modified since then so that the lifetime in steel is now 177.9 versus 263 in July.

The oxide present on the "as received" specimens does not affect the lifetime as suggested in July. The previous effect was probably due to extreme temperature problems encountered in July.

Also the presence of bright cadmium does not seem to interfere with desorption of hydrogen as previously suspected.

The discrepancies encountered in Tests 4, 26, and 30 are unexplained and may need additional work. Even so, the conclusions reached in the following section appear to be valid.

Conclusions

1. The existing equipment and computer program gives reproducible results of ± 2 psecs on the same sample, run a number of times over a period of two to three weeks.

2. Subjecting steel to conditions which normally cause absorption of hydrogen causes an increase in short lifetime. Tests 2, 28, 30, and 32 reveal that this occurs whether this "embrittlement" is a result of cadmium plating or electrochemical reaction when the specimen is either the cathode

or anode. This last result is quite important is that the nascent hydrogen normally credited with "hydrogen embrittlement" is released at the cathode not the anode. The anodic effect may explain why exposing a steel part to caustic solutions is embrittling even though there is a predominance of hydroxyl ions. The electropolished and electropolished and cadmium plated specimens experienced an increase in lifetime of 14.1 ± 2 psecs when tested shortly after preparation or immediately after stripping of the cadmium. It should be noted, however, that Test 34 indicates that the stripping operation does not produce an increase in lifetime.

3. The annihilation lifetime in cadmium is appreciably greater than in steel. Tests run so far are not adequate to completely determine it, but it appears to be at least 220 psecs. This is contrary to results obtained by Weisberg⁽²⁹⁾ of 190 ± 4 psecs, but is within 199 ± 21 reported by Jones and Warren⁽²⁸⁾. The current tests indicate the majority of the annihilation occurs within the first two or three thousandths of the surface. This can be better established once the lifetime in cadmium is determined. It is important to note that the presence of cadmium increases the lifetime sufficiently so that on a cadmium plated specimen it is difficult to determine the extent of lifetime change in the base metal unless exact thickness of cadmium is known.

4. Test 26 did not reveal any trend indicating a decrease in lifetime during the first seven days following plating. Fletcher and Elsea⁽³⁵⁾ indicate that in sixty days hydrogen will reduce to equilibrium at room temperature in a one inch square coupon of carbon steel. The diffusion rate varies inversely to the square of the thickness thus it should be expected

to evolve from thin sections much faster. Sample 26 will be rerun at later times to determine if a decrease in lifetime occurs. The results of Test 30 may indicate hydrogen is lost at room temperature when plating is removed.

5. The entire increase in lifetime as a result of absorption of hydrogen can be removed by baking at 400°F for twenty four hours whether or not there is bright cadmium on the surface. This was demonstrated by comparing the results of Tests 5, 6, and 34.

6. Glass blasting apparently increases dislocation content sufficiently to increase the lifetime almost identically to that when a part is subjected to "embrittling" conditions. This effect can be removed by baking at 400°F for twenty-four hours.

REFERENCES

1. P. C. Lichtenberger and J. R. Stevens, Can. J. Phys., 50, 345 (1972).
2. P. Kirkegaard and M. Eldrup, Computer Physics Comm., 3, 240 (1972).
3. D. W. Marquardt, J. Soc. Ind. Appl. Math., 11, 431 (1963).
4. J. H. Kusmiss, A. N. Goland and C. L. Snead, Jr., Phys. Stat. Sol. b, 50, 33 (1972).
5. R. M. J. Cotterill, K. Peterson, G. Trumpy and J. Traff, J. Phys. F. Metal Phys., 2, 459 (1972).
6. A. A. Adamenko, Y. Ya. Dakhtyar and V. S. Mikhalenkov, Sov. Phys. Dokl., 13, 702 (1969).
7. P. Hautajarvi, A. Tamminen and P. Jauho, Phys. Review Letters, 24, 459 (1970).
8. A. H. Cottrell, S. C. Hunter and F. R. N. Nabarro, Phil. Mag., 44, 1064 (1953).
9. R. A. Brown, Phys. Rev., 141, 568 (1966).
10. A. A. Adamenko, Y. Ya. Dekhtyar and V. S. Mikhalenkov, Phys. Letters, 26A, 288-9 (1968).
11. J. C. Grosskreutz and W. E. Millet, Phys. Letters, 28A, 621 (1969).
12. B. E. Warren, Progress in Metal Physics, Vol. 8, (B. Chalmers, ed.) Pergamon Press, 1959, p. 147.
13. C. M. Wan and J. G. Byrne, Proceedings of the Second International Conference on Fracture, Paper 52, 598 (1969).
14. R. J. DeAngelis, Local Atomic Arrangements Studied by X-Ray Diffraction, (J. B. Cohen and J. E. Hillard, eds.) Gordon and Breach, New York, 1965, Vol. 136, p. 271.
15. I. K. Mackenzie, T. L. Khoo, A. B. McDonald and B. T. A. McKee, Phys. Rev. Letters, 19, 946 (1967).
16. D. H. Avery and W. A. Backofen, AIME Fracture Conference, 1962, p. 172.
17. G. A. Miller, D. A. Avery and W. A. Backofen, Trans. AIME, 236, 236 (1967).

18. C. L. Snead, Jr., A. N. Goland, J. H. Kusmiss, H. C. Huang and R. Meade, Phys. Rev., 133, 275 (1971).
19. C. L. Snead, Jr., A. N. Goland, H. C. Huang, C. D. Esseltine, R. Meade, and J. H. Kusmiss, Bull. Am. Phys. Soc., 15, 300 (1970).
20. R. E. Ogilvie and S. Moll, Inco. Seminar, Duquesne Club, Pittsburgh, Penn., 1960.
21. G. K. Williamson and R. E. Smallman, Phil. Mag., 1, 34 (1956).
22. R. E. Smallman and K. H. Westmacott, Phil. Mag., 2, 669 (1957).
23. H. Weisberg and S. Berko, Phys. Rev., 154, 249 (1967).
24. B. Bergersen and M. J. Scott, Sol. State Comm., 7, 1203 (1969).
25. D. C. Connors and R. N. West, Phys. Letters, 30A, 24 (1969).
26. A. Seeger, Phys. Letters, 41A, 267 (1972).
27. K. Takagi, K. Kuribayashi, S. Nanao, S. Tanigawa and M. Doyama, Sol. State Comm., 9, 2155-6 (1971).
28. G. Jones and J. B. Warren, Can. J. Phys., 34, 1933 (1956) as reported in Weisberg.
29. H. L. Weisberg, Ph.D. Thesis, Brandeis University, 1965.
30. J. A. Rinebolt, Naval Research Laboratory Memorandum Report 345 (March 1953 to July 1954).
31. R. H. Raring and J. A. Rinebolt, Naval Research Laboratory Memorandum Report 452 (April 1955).
32. P. C. Lichtenberger and J. R. Stevens, A computer program entitled "The Analysis of Counting Distributions with a Complex Exponential Character," Department of Physics, University of Guelph, Guelph, Ontario.
33. K. G. Lynn is a graduate students in the Division of Materials Science and Engineering, University of Utah, also doing work on positron annihilation.
34. From a conversation between J. G. Byrne and A. Goland and T. Hall, Brookhaven National Laboratory, Stonybrook, August 1972.
35. E. E. Fletcher and A. R. Elsea, Defense Metals Information Center, Columbus, Ohio (March 26, 1964).

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Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10.

Lifetime vs Cadmium Thickness.