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

Technical Progress Report

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ABSTRACT

The year 1974 has seen the blossoming of a number of publications and talks based on our positron lifetime experiments including a particularly significant oral presentation at the TMS AIME Spring Meeting concerning fatigue of steel below its yield point. The importance of the latter is the fact that positron lifetime was found to be a gradually increasing parameter which in the case of elastic fatigue can be a true N.D.T. technique for failure prediction. A cryostat has been constructed which is about to be used to measure the positron lifetime changes associated with the earliest stages of vacancy coalescence and void formation following electron irradiation. This will be the first work of its kind, since current positron annihilation information in connection with changes due to irradiation is all in the large void range after neutron damage. Considerable equipment and computer analysis modifications have been effected to improve the reliability, speed, and interpretive power of the experiments in general.

I. TECHNICAL REPORT

A. Equipment

The system suffered considerable down time this year as a result of photomultiplier base problems. The original photomultiplier bases (ORTEC Model 270) were inadequate in that in March 1974 we experienced intermittent behavior and returned the bases to ORTEC. These bases use a tunnel diode as a constant fraction discriminator (C.F.D.). The diodes were found to be faulty and were not replaceable on an "off the shelf" basis, in addition, there is a possible design error in the C.F.D. Therefore we were forced to abandon these photomultiplier bases and fabricate a complete new system. We turned to an ORTEC 265 photomultiplier base and on the "slow" side of the fast-slow circuit changed to an ORTEC 113 pre-amplifier. In addition, a completely new C.F.D. was added.

We utilized a design of T. Hall, formerly of Stony Brook--Brookhaven - now at Cornell, and two boards were built and made to work. Since Mecl III integrated circuits were used, we found we had a very acute temperature problem. The circuit layout was changed and two new boards fabricated. The new boards have integrated circuits coupled via BeO to a large Cu block heat sink whose heat capacity is such that air cooling is sufficient for stabilization. In the near future we plan to thermoelectrically cool the Cu block. In addition to the C.F.D. on each board the circuit has two energy discriminators which allow selection of the

0.511 MEV gamma rays or the 1.28 MEV gamma rays within ± 30 percent. Once the thermoelectric cooling has been installed these discriminators will be set so as to allow the complete removal of the slow coincidence side⁽¹⁾ of the measurement system. Hopefully, this will decrease the down time of the system and increase the count rate so as to permit a run in 10 hours or better.

B. Fatigue Studies in 4340 Steel

Duplicate Sonntag cantilever bending fatigue specimens were prepared in each of the hardened and tempered (Rockwell C 51), and annealed (Rockwell C 27) conditions. The intention of the fatigue experiment on these is to cycle each condition through a maximum stress of two-thirds of the yield point. We are looking for fatigue hardening of the softer condition and fatigue softening of the harder condition. We have completed the fatigue hardening part of this experiment and the consequent positron lifetime changes are seen in Figure 1. One notes that the increase is not precipitous, as had been the case for severe plastic strain range cycling of Ni and Ni-Co alloys,⁽²⁾ but rather is gradual and does not saturate even at fracture. In our previous work⁽²⁾ the early saturation of the positron lifetime during severe plastic cycling made the technique perhaps an overly sensitive indicator of accumulating lattice damage, i.e., one might view it as overly sensitive if his objective were to utilize such a material to a fatigue life closer to the failure point because the flat saturation region does not

provide an indication of approaching failure. The linear regions of positron lifetime increase seen in Figure 1 are ideal for ultimate material "calibration" for failure predictive purposes. We regard this as a very important development in terms of the ultimate goal of this project.

The hardened condition of 4340 steel has been partially tested in fatigue and positron lifetime, however, the experiment had to be interrupted due to damage caused by an unexpectedly large power surge in the building which got through our protective circuit and destroyed some integrated circuits. The system now is "up" again and as soon as FWHF adjustments and recalibrations are completed, cycling and lifetime measurements will be resumed. Up to the time of the problem the 4340 had been cycled 6000 times and positron lifetimes measured at intervals of 1000 cycles. No change in positron lifetime had been observed. We plan on going to increments of 5000 cycles. It will be extremely interesting if fatigue softening and a decrease in positron lifetime are observed. This might be expected on the basis of the simplification observed in the dislocation substructure during fatigue of initially hard materials.

C. Electron Irradiation Studies

In preparation for an experimental program on positron lifetime measurements during the early stages of vacancy coalescence and void formation following electron irradiation and annealing, we

have completed the following work. A cryostat was designed and built which will permit the electron irradiation of Cu single crystals at 77°K and their subsequent electrical resistivity and positron lifetime measurement during pulse annealing experiments. Preliminary calculations of damage rates indicate that in order to develop a significant amount of damage, eight hours of electron irradiation will be required with a 6 MEV beam. At that time we should have damage of between 10 and 100 parts per million. The cryostat has a 2.6 liter liquid nitrogen capacity and must be refilled every 13 hours. A cold finger has a stainless steel jacketed heater coil; thus, the sample temperature is controlled by the joule heating of the coil balanced against heat leaks up the cold finger to the LN₂. The sample is monitored via two copper-constantan thermocouples.

The cryostat was made compatible with both the 8 MEV linear accelerator of the University of Utah Physics Department and our positron lifetime system (in the plexiglas temperature-controlled box holding the detector crystals and photomultiplier bases).

Great care was taken in preparing a number of single crystal Cu specimens of low dislocation density. After electropolishing, the Cu crystals are 0.070 inches thick with areas of 0.4 inches by 0.4 inches. Each sample has provision (etched groove 0.02 inches by 0.04 inches by 0.20 inches) for internal containment of the Na²² positron source, i.e., the grooves are aligned and the

pair of Cu crystals are diffusion bonded to provide a channel into which we will place 100 μ curies of Na²². This means that the sample itself, rather than mylar, will be the receptacle for the positron source, which will eliminate our need to consider annihilations in the mylar as in the fatigue experiments. This is made possible by the fact that the source and specimens need never leave the dewar until the entire experiment (radiation, pulse annealing, and measurement) is over when it can be disposed of by the radiological health department.

Estimation of the vacancy concentration in the sample will be made possible by monitoring the change in resistivity of two 0.003 inch Cu wires. These are situated in front of and behind the specimen with respect to the electron beam; this will also give us a damage profile for the sample.

The cryostat has been coupled to the linear accelerator and alignment has been done on the sample holder with a dummy sample. The cryostat also was coupled with the positron lifetime measurement system and counting experiments have been performed on the dummy specimen.

At this writing the damage created by the power surge (see Part C) has been repaired, the lifetime system is being recalibrated, the Cu crystals have been etched and only the diffusion bonding and Na²² injection remain prior to the start of the electron irradiation.

D. Hydrogen Embrittlement

Previous work⁽³⁾ on the role of hydrogen embrittlement and dislocations and other defects suggested that, in fact, these defects are trapping sites for the hydrogen. A number of tests were initiated to establish this relationship, however, equipment problems that developed during the lifetime measurements negated the results.

Proposed experiments to follow through on this matter are described in the renewal proposal concurrently being submitted.

E. Analysis of Positron Lifetime Data

The purpose of this work is to enable us to make a comparison of the various mathematical models currently used to analyze data on the lifetimes of positrons in deformed metal lattices. Each run of the program will fit a given model to the experimental lifetime data. By comparing the respective reduced chi-squares (a measure of the goodness of fit) as well as other data, we hope to be able to conclude that one of the models is better than the rest for data from our experimental system. Similar runs and conclusions could then be made on data from other experimental systems in the field. Consistent results might then allow some judgment to be made as to the relative value of these mathematical models.

The program is written in Fortran and run on a UNIVAC 1108 computer. A subprogram defines the model to be fitted. Changing

from one model to another requires the replacement of this subprogram with the correct version for that model. The program is flexible in that the introduction of a new model only necessitates the writing of an additional version of the subprogram.

Two methods of fitting are utilized: (1) Marguardt's iterative technique⁽⁴⁾ of least squares, an almost optimum combination of the method of Gauss-Newton and the method of steepest descent; and (2) A multiple linear regression analysis subprogram⁽⁵⁾. The two separate techniques enable the program to handle a greater number of parameters than otherwise possible. One Marguardt least square iteration is performed on the non-linear parameters while holding the linear parameters constant at their current values, then the multiple linear regression analysis fits the linear parameters while holding the non-linear parameters constant at their current values. This process requires initial guesses for the non-linear parameters which includes the lifetimes, t_0 (time equals zero), and others depending on the particular model being used. Guesses for the linear parameters (which are the respective intensities of the lifetimes and also a constant background) are not needed. At present the final printout includes an alphameric title, all input variables, values of parameters at the end of each iteration, the final fitted values, a certain amount of error analysis, and a table of the experimental data, each corresponding fitted value and their ratio.

The models so far include three for the instrumental resolution spectrum and five for the lifetime spectrum. They should be integrated between channels since the spectra represent counts averaged over the width of one channel. The instrument resolution models represent the response of the instrument. Four instrumental resolution models (all are assumed to have the linear background added) have been used in this work. They are:

(1) A Poisson distribution,

$$F(t_i) = I \frac{\mu^n}{n!} e^{-\mu},$$

where $n = \frac{t_i}{\text{channel width}}$. This was run without integrating between channels and will be corrected. The non-linear parameter is μ (mean of the distribution), and the linear parameters are I and background.

(2) A Gaussian distribution,

$$F(t_i) = I e^{-\frac{(t_i - \mu)^2}{2\sigma^2}}.$$

This was also run without integrating between channels and will be corrected. The non-linear parameters are μ (mean) and σ (standard deviation). The linear parameters are I and background.

(3) The double-sided exponential function used by Lichtenberger, Stevens, and Newton.⁽⁶⁾

(4) A combination of the Gaussian convoluted with the double-sided exponential, similar to that used by Hall.⁽⁷⁾ We have called this the slope-sided resolution function. This was

originally given as a convolution integrad, but solved by us to the form

$$F(t_i) = I_- \left[Y_{i+1,-} - Y_{i,-} + \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_{i+1}-t_0)}{W} \right) - \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_i-t_0)}{W} \right) \right] \\ - I_+ \left[Y_{i+1,+} - Y_{i,+} - \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_{i+1}-t_0)}{W} \right) + \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_i-t_0)}{W} \right) \right]$$

$$\text{where, } Y_{i,-} = \exp \left(\frac{t_i-t_0}{\tau_-} + \frac{W^2}{16\tau_-^2 \ln 2} \right) \left(1 - \operatorname{erf} \left[\frac{2\sqrt{\ln 2}(t_i-t_0)}{W} + \frac{W}{4\tau_- \sqrt{\ln 2}} \right] \right)$$

$$\text{and } Y_{i,+} = \exp \left(-\frac{t_i-t_0}{\tau_+} + \frac{W^2}{16\tau_+^2 \ln 2} \right) \left(1 - \operatorname{erf} \left[-\frac{2\sqrt{\ln 2}(t_i-t_0)}{W} + \frac{W}{4\tau_+ \sqrt{\ln 2}} \right] \right)$$

The non-linear parameters are τ_- , τ_+ (reciprocal slopes of left and right hand sides of the function when plotted on a semilog graph), t_0 (time zero), and W (full width at half maximum). The linear parameters are I_- , I_+ , and background.

The instrument resolution model should fit data taken with Co^{60} as a source, since this isotope emits two γ -particles simultaneously. We have fit Co^{60} data using models 1, 2, and 4.

We have used two models to represent the decay of the positron in the samples. The first is a sum of three exponential terms,

$$F(t) = \sum_{j=1}^3 I_j e^{-t/\tau_j}.$$

The second is similar to the first but incorporates a description of the time dependence of the trapping of the positron at a defect. At present, we have assumed only one type of defect, presumably a dislocation.

The final mathematical model which is used to represent the data is obtained by convoluting one of the models for positron decay with one of the instrument resolution models. There are eight possible models with this procedure. However, we have concentrated our research so far on only five of these.

The five lifetime models we have studied (all have been integrated between channels) are:

(1) The summation of three exponential decay terms convoluted with a Gaussian resolution function. This is the model used by the program Positronfit⁽⁸⁾, a modified version of which normally analyzes our data. The model is of the form:

$$F(t_i) = \sum_{j=1}^3 I_j \left[Y_{ij} - Y_{kj} - \text{erf}\left(\frac{2\sqrt{\ln 2}(t_i - t_0)}{W}\right) + \text{erf}\left(\frac{2\sqrt{\ln 2}(t_{i+1} - t_0)}{W}\right) \right]$$

where,

$$Y_{ij} = \exp\left(-\frac{t_i - t_0}{\tau_j} + \frac{W^2}{16\tau_j^2 \ln 2}\right) \left(1 - \text{erf}\left[\frac{-2\sqrt{\ln 2}(t_i - t_0)}{W} + \frac{W}{4\tau_j \sqrt{\ln 2}}\right]\right)$$

and $k = i+1$. The non-linear parameters are τ_1, τ_2, τ_3 (the lifetimes), t_0 (time zero), and W (full width at half maximum).

The linear parameters are I_1, I_2, I_3 , and background.

(2) The same as (1) above but with the inclusion of two additional terms incorporating Tom Hall's slope-sided resolution function. Letting $\tau_4 = \tau_+$,

$$F(t_i) = I_- \left[Y_{i+1,-} - Y_{i,-} + \text{erf}\left(\frac{2\sqrt{\ln 2}(t_{i+1} - t_0)}{W_{1/2}}\right) - \text{erf}\left(\frac{2\sqrt{\ln 2}(t_i - t_0)}{W_{1/2}}\right) \right] \\ + \sum_{j=1}^3 I_j \left[Y_{ij} - Y_{kj} - \text{erf}\left(\frac{2\sqrt{\ln 2}(t_i - t_0)}{W_{1/2}}\right) + \text{erf}\left(\frac{2\sqrt{\ln 2}(t_{i+1} - t_0)}{W_{1/2}}\right) \right]$$

where $Y_{i,-}$ and Y_{ij} are as previously defined and $k = i+1$.

The non-linear parameters are τ_- , τ_+ , τ_1 , τ_2 , τ_3 , t_0 and W as previously defined. The linear parameters are I_- , I_+ , I_1 , I_2 , I_3 , and background.

(3) The same as (2) above but with the deletion of the τ_- term. Indications are that the τ_- term, characterizing the left-hand side of the curve, contributes very little to the actual lifetimes (τ_1 , τ_2 , and τ_3). There is a good chance that the removal of the τ_- term, thereby reducing the number of fitted parameters by two, will not significantly affect the final results. The equation is:

$$F(t_i) = \sum_{j=1}^4 I_j \left[Y_{ij} - Y_{i+1j} - \operatorname{erf}\left(\frac{2\sqrt{\ln 2}(t_i - t_0)}{W_{1/2}}\right) + \operatorname{erf}\left(\frac{2\sqrt{\ln 2}(t_{i+1} - t_0)}{W_{1/2}}\right) \right]$$

where Y_{ij} is as previously defined. The non-linear parameters are τ_+ , τ_1 , τ_2 , τ_3 , t_0 , and $W_{1/2}$. The linear parameters are I_+ , I_1 , I_2 , I_3 , and background.

(4) The double-sided exponential as given by Lichtenberger, Stevens, and Newton⁽⁶⁾. This function is a summation of exponentials of the form:

$$F(t_i) = \sum_{j=1}^4 I_j \exp\left(-\frac{t_i - t_0}{\tau_j}\right)$$

The non-linear parameters are τ_- , τ_1 , τ_2 , and τ_3 (interpreting the τ_4 term as τ_-). The linear parameters are I_- , I_1 , I_2 , I_3 , and background (I_- will be negative).

(5) The same as (2) above but with the incorporation of a trapping model. At present, we are using only one type of defect (dislocation). The form of the function is identical to (2) except that the τ_1 term ($j=1$ in the summation) is replaced with:

$$I_D \left[Y_{iD} - Y_{kD} - \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_i - t_0)}{W} \right) + \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_{i+1} - t_0)}{W} \right) \right] \\ + I_L \left[Y_{iL} - Y_{kL} - \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_i - t_0)}{W} \right) + \operatorname{erf} \left(\frac{2\sqrt{\ln 2}(t_{i+1} - t_0)}{W} \right) \right]$$

where,

$$Y_{iD} = \exp \left(-\frac{t_i - t_0}{\tau_D} + \frac{W^2}{16\tau_D^2 \ln 2} \right) \left(1 - \operatorname{erf} \left[-\frac{2\sqrt{\ln 2}(t_i - t_0)}{W} + \frac{W}{4\tau_D \sqrt{\ln 2}} \right] \right),$$

$$Y_{iL} = \exp \left(-s[t_i - t_0] + \frac{s^2 W^2}{16 \ln 2} \right) \left(1 - \operatorname{erf} \left[-\frac{2\sqrt{\ln 2}(t_i - t_0)}{W} + \frac{sW}{4\sqrt{\ln 2}} \right] \right),$$

$$k = i+1, \text{ and } s = \frac{1}{\tau_L} + k_D.$$

The non-linear parameters are τ_L (lifetime in lattice),

$\tau_2, \tau_3, \tau_-, \tau_+, k_D$ (the trapping rate of dislocation), t_0 ,

and W . The linear parameters are $I_D, I_L, I_2, I_3, I_-, I_+$,

and background.

Data used for the debugging of the instrumental resolution models came from runs on our experimental system using a Co^{60} source.

Data used for the debugging of the lifetime models came from runs on our system using a Na^{22} source and a 4340 steel sample with 8000 cycles fatigue at two-thirds yield point. A final programming check will be carried out using artificially generated data. We

will generate this data for each model considered by first selecting typical values for the parameters and then storing the cumulative totals,

$$T_i = \sum_{j=1}^i F(t_j) \quad i = 1, N$$

where N is the number of data points desired. Finally, a random number, n , $0 \leq n \leq 1$, will be generated and multiplied by the total number of counts, T_N , and a count added to that channel i for which $T_{i-1} \leq nT_N \leq T_i$. These final series of steps will be repeated T_N times, then generating a distribution with random fluctuations according to each model.

In general, the models involving Tom Hall's slope-sided resolutions function appear better than the others. The deletion of the τ_0 term in (3) seems to have no great effect which indicates that the trapping model (5) should be tried without a τ_0 term. The results, however, are inconclusive since all bugs have not yet been ironed out.

F. Other Activities on the Contract

1. University support was obtained for a complete Materials Test System (MTS) fatigue machine. This will enable closed loop fatigue work. It is now operational with the exception of grips which are on order.
2. The following papers have appeared recently or are in press:
 - a. *Positron Trapping at Stacking Faults in Metals*, Phys. Stat. Solidi (b) 60, K117 (1973) by K. G. Lynn,

R. W. Ure, and J. G. Byrne.

- b. *Positron Lifetime Measurements of Fatigue Damage in Ni and a Ni-Co Alloy*, Phys. Stat. Solidi (a) 22, 731 (1974) by K. G. Lynn, C. M. Wan, R. W. Ure, and J. G. Byrne.
 - c. *The Effect of Plastic Deformation on Positron Annihilation in Copper of Varying Grain Size*, Acta Met. (In Press) by K. G. Lynn, R. W. Ure, and J. G. Byrne.
3. An oral presentation was given at the Spring Meeting of the Institute of Metals Division of AIME, Pittsburgh, 1974, on the fatigue studies of 4340 steel. Oral discussion was also given to two positron contributions from Columbia University. I (J. G. Byrne) think it is no exaggeration to say that Dr. K. G. Lynn taught those authors a great deal about positron annihilation in that and subsequent discussions.
4. A Siemens 1A transmission electron microscope has been purchased by this department. This should be a great help to the project.
5. It is a pleasure to announce that the Ph.D. thesis of K. G. Lynn was completed early in January 1974, and that after serving as a postdoctoral investigator with us through August, he is joining Brookhaven National Laboratory as an Assistant Physicist. He will continue to work on

positron annihilation there with Dr. Alan Goland. Dr. Lynn's new title is a reflection of his tremendous efforts here and his potential since he was not trained as a physicist.

6. Dr. Lynn was invited to present several seminars at the ORTEC Company on circuitry modifications and advances made here. We understand that our circuitry was then described by ORTEC to a number of laboratories including the Stuttgart group under Dr. Alfred Seeger, where it will be adopted.
7. Dr. M. Wechsler of Ames is interested in collaborating with us on positron studies of neutron irradiated Vanadium specimens. We will do our best to accomplish this as it relates to our own void and vacancy studies.
8. Dr. I. Kramer formerly of Martin-Marietta Aerospace and now with the Naval Ship Research and Development Center, Annapolis, who pioneered many surface damage studies, i.e. surface work hardening being different from that in the bulk, is also interested in pursuing joint work utilizing our positron lifetime background. This would also be highly desirable to fit in as time permits. It's fatigue aspects connect closely with the goals of this contract.
9. Specimens of Al containing various ppm contents of Au were obtained from Dr. Vandermeer of Oak Ridge National Laboratory. These will be used to examine positron trapping at solute atoms and at precipitates as time permits.

10. F. Seppi of Hill Air Force Base has offered to let us measure positron lifetimes on shotpeened specimens at various stages of fatigue life. This would tie in nicely with our N.D.T. objectives in fatigue and also with shot-peened studies of the writer⁽⁹⁾.
11. The review article on positron annihilation mentioned in COO AT(11-1)-2128-5, page 23, is still scheduled to be done. Its publication scheduling has been set back a year, however, by Dr. Herman, the editor.
12. We are impressed with the contributions of an undergraduate student, R. Waukie, with whom Dr. Lynn spent considerable training time on computer work. We hope to keep him connected part time with the contract. He is only a sophomore and shows much promise.
13. Dr. Byrne was invited to speak to the Bonneville Chapter of ASM on September 12, 1974, in Ogden, Utah, on this AEC sponsored research. The title was *New Non-Destructive Testing Possibilities with Positrons Annihilation*.
14. Papers which are, or will shortly be, in various stages of preparation treat: computer analysis of data, elastic fatigue of 4340 steel, fatigue of Al, and computer fits of various theories in the field to our data.

II. TIME INFORMATION ON PRINCIPAL INVESTIGATOR

The principal investigator, Dr. J. G. Byrne, has spent one month, full time during the summer of 1974 on this research. During the 1973-1974 academic year he spent the equivalent of one month, full time on this research (at no cost to AEC).

Dr. R. W. Ure, Jr., Faculty Associate, spent one-half month, full time during the summer of 1974. During the 1973-1974 academic year he spent the equivalent of one-half month, full time on this research (at no cost to AEC).

Figure 1.

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