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

FINAL REPORT

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ABSTRACT

Positron studies have been applied successfully to cyclic fatigue in steel and copper and have shown a capability to non-destructively detect fatigue softening and fatigue hardening. In the case of elastic high cycle fatigue in initially soft steel the fatigue hardening is identified with point defects. For cyclic plastic range high cycle fatigue cyclic hardening and softening are correlated with changes in dislocation substructure.

Positron measurements during the pulse annealing of copper single crystals following electron irradiation at 77° K have revealed the formation of multi-vacancy complexes which constitute the precursors of radiation induced voids.

Positron studies have been applied to hydrogen embrittlement in 4340 steel and more recently in nickel. The technique can non-destructively detect hydrogen embrittlement and gage its extent. U. S. Patent #4064438 was issued on this basis.

SUMMARY OF ACCOMPLISHMENTS & PUBLICATION STATUS

A. Fatigue Results

The first effort on the fatigue of steel showed that 4340 steel in initially hard and soft conditions may be followed successfully by positron lifetime measurements during cyclic fatigue softening and hardening respectively. (K. G. Lynn and J. G. Byrne, Met. Trans. 7A (1976) p. 604). This work was quoted and shown in two slides by Dr. R. M. J. Cotterill in an invited review paper entitled, "Crystal Defect Studies Using Positron Annihilation: An Appraisal of the Current Situation" at the 4th International Conference on Positron Annihilation, Helsingør, Denmark, 23-26 August, 1976. More recently these same data were included in another review paper by C. E. Coleman of AERE, Harwell, entitled, "Positron Annihilation - A Potential New NDT Technique" in NDT International, October 1977, p. 227-239. In this same review four other references to data generated by this group were made. These will be enumerated later in this report.

Follow-up work on fatigue in steel was recently submitted to Metallurgical Transactions ("Positron Lifetime & X-Ray Particle Size Measurements During High Cycle Fatigue of AISI 4340 Steel" by P. Alexopoulos and J. G. Byrne). This is in final review now, a response to reviewer comments has been made and we assume acceptance will soon occur. Reprints will, of course, be forwarded immediately upon receipt here. A manuscript is appended to this report.

Earlier fatigue work on Ni and a Ni-Co alloy ("Positron Lifetime Measurements of Fatigue Damage in Ni and a Ni-Co Alloy," K. G. Lynn, C. M. Wan, R. W. Ure, and J. G. Byrne, Phys. Stat. Sol. (a) 22, 731 (1974)) was also quoted, and figures described in the above mentioned review of C. E. Coleman.

At this writing a Cu fatigue paper ("Positron Lifetime Changes During the Fatigue of Cu" by P. Alexopoulos and J. G. Byrne) is in review with Metallurgical Transactions. Initial reviewer comments have been received and the response is being prepared. This work describes fatigue softening and hardening in Cu and shows, as for the steel work, the utility of positron annihilation applied to fatigue. A manuscript is appended to this report.

B. Hydrogen Embrittlement Results

Hydrogen embrittlement in 4340 steel was studied with positron lifetime measurements and reported in the International Conference on the Effect of Hydrogen on Behavior of Metals, held at Moran, Wyoming in 1975. The report was entitled, "Positron Annihilation Studies of Hydrogen Embrittlement" (P. Alex, T. D. Hadnagy, K. G. Lynn and J. G. Byrne, p. 642, Editors A. W. Thompson and I. M. Bernstein, publ. by AIME, 1976). Up-dated results were later presented at the 4th International Conference on Positron Annihilation, Helsingør, Denmark, 1976 ("Positron Studies of Fatigue, Hydrogen Embrittlement and Radiation Damage in Metals" by J. G. Byrne, P. Alexopoulos, P. Alex, T. D. Hadnagy, R. Waki, G. R. Miller and R. W. Ure, p. 59). This effort resulted in the issuance of U. S. Patent #4064438 and the work has also been cited by the earlier mentioned NDT review by C. E. Coleman of AERE Harwell.

More recently hydrogen studies were shifted to nickel and a M. S. thesis (P. W. Kao) was completed in May 1978. This thesis is appended to this report and the material is currently being written up for publication.

C. Electron Irradiation Results

A study of the electron irradiation of Cu single crystals at 80°K was completed and submitted in June 1978 for publication to Physica Status Solidi

(B). A multivacancy complex was detected during pulse annealing at about 660°K. This complex is a likely precursor of larger radiation induced voids in solids.

D. Grain Size & Tensile Strain Results

The positron lifetime of samples of varying grain size Cu was measured as a function of plastic tensile strain (K. G. Lynn and J. G. Byrne, *Acta Met.* 22, (1974) p. 1075). Subsequent analysis of these data by H. P. Leighly (*Appl. Phys.* 12, (1977) 217) suggests a linear relation between internal grain boundary surface area per unit volume and positron lifetime.

E. Annealing

Data showing the positron lifetime as a function of annealing temperature for Cu and α -brass, each deformed by rolling 10% and 50%, and for Ni rolled 69% are included in a manuscript in review at *Physics Status Solidi* (manuscript appended to this report). The same manuscript contains data on the increase of positron lifetime as a function of tensile strain.

F. Stacking Fault Results

The lack of positron trapping at stacking faults was reported (K. G. Lynn and J. G. Byrne, *Physics Status Solidi* (B) 60, (1973) K117).

G. Miscellaneous

The writer was invited to summarize the positron aspects of Cold Work and Annealing for the TMS-AIME Symposium on Recovery, Recrystallization and Grain Growth in Materials, in Chicago Oct. 24-27, 1977. A similar invitation has been received for the February, 1979 AIME Meeting.

H. Equipment Development

1. Introduction

A number of improvements and major changes have been made in the positron lifetime measurement systems. In the past several years, we have had continuing troubles with the Stabilized Timing Discriminators which had been built following the design of T. N. Hall. When they were working, they performed well, but we had continual problems with them failing. In the past year, a second set of boards and a frame to hold them have been built. A number of improvements which allow easier servicing and easier adjustment of the controls were incorporated in the new design. These have proved to be very satisfactory.

A dual Floppy Disk System and 16,000 additional words of core storage have been added to the PDP/8 system which is used to accumulate data from the positron lifetime equipment. This will allow several major improvements in our experiments and data reduction. (a) It allows much more rapid storage of data pending complete analysis on the computer. (b) It allows us to get the lifetimes from the raw data using the PDP/8 rather than having to punch paper tape and take it to the central computer. (c) It allows us to analyze the data as it is accumulated and to reject data which shows evidence of serious drift in the equipment. This rejection is done before each block of data is added to the rest of the data accumulated during the run and thus allows us to get data which is more accurate.

Finally, two Ortec 473A Constant-Fraction Discriminators have been purchased and used at times in place of the Hall Stabilized Timing Discriminators.

A picture of the two scintillation detectors and phototubes is shown in Fig. A. The sample and positron source sits in the crack between the two tubes at the center of the photo.

2. Stabilized Timing Discriminators

Several years ago, we built two printed circuit boards using the lifetime system with stabilized timing discriminators described by Thomas N. Ball, (Nuclear Instruments and Methods 117, 253-9 (1974)). They worked well for several years, but as they grew older troubles developed. Shorts or leakage between leads on the board developed continually. This system requires two identical boards. In our original system, the two boards were mounted one below the other making it difficult to get at the lower board or even the under side of the upper board for testing. Another problem with the older boards involved the four adjustable pots on each board which are used to adjust the position of the energy windows in the energy discriminator and to adjust the constant-fraction discriminator for minimum walk. On the older boards, these adjustments were made with screwdriver adjust potentiometers on the individual boards. This proved to be awkward.

We have redesigned and built new boards and a new rack for the boards, as shown on Fig. B. The boards are held side by side for ease in trouble shooting. The adjustments are made with 10 turn potentiometers mounted on the front panel. The new system is much easier to service and adjust. The downtime has been significantly lower than for the original system.

3. PDP/8 System

A dual Floppy Disk memory and 16,000 additional words of core memory have been added to the PDP/8 system giving a total of 20,000 words of core storage. The PDP/8 is shown on Fig. C and the Floppy Disk Memory in Fig. D.

The program Positronfit, which we have been using to analyze our data, has been rewritten so that it can run on this system. This program is written in FORTRAN and compiled on the system. This program analyzes the data and extracts numerical values of positron lifetime from the data by least squares fitting procedures.

4. Ortec Constant-Fraction Discriminators

Two Ortec Model 473A Constant-Fraction Discriminators have been purchased. These have been used primarily when the Hall system has been down for servicing. They have only lower level discriminators, so they pass all signals having an energy above the lower level. Although they have not been tested extensively, they appear to give very good performance.

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Revised

LIST OF APPENDICES

1. "Positron Lifetime & X-Ray Particle Size Measurements During the High Cycle Fatigue of AISI 4340 Steel" by P. Alexopoulos and J. G. Byrne.
2. "The Removal of Defects from Solids as Observed with Positron Annihilation" by M. L. Johnson, S. Saterlie, D. Boice and J. G. Byrne.
3. "Positron Lifetime Changes During the Fatigue of Cu" by P. Alexopoulos and J. G. Byrne.
4. "Positron Lifetime Studies of Electron Irradiated Copper" by T. D. Hadnagy, J. G. Byrne and G. R. Miller submitted to Physica Status Solidi.
5. "Positron Annihilation Studies of Hydrogen Embrittlement of Nickel" by Po-We Kao as an M. S. Thesis, University of Utah.

NOTE: We have just been notified that manuscript No. 2 above is accepted for publication in Phys. Stat. Solidi August, 1978, issue 2, vol. 48.

FIGURE A

Two scintillation detectors and photubes

FIGURE B

New printed circuit boards and rack

FIGURE C

PDP/8 System

FIGURE D

Floppy disc memory system