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

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ABSTRACT

The critical parts (electronics in the base of the photomultipliers) of the electronics have been brought under temperature control of within ± 0.1°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 - 66.5% 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. Experiments to study lifetime changes during cyclic softening and cyclic hardening of AISI 4340 steel are beginning.

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 monatonic 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 voids. During 1973 progress has been made in the following areas: equipment, analysis, fatigue studies, grain size studies, and hydrogen embrittlement studies.

II. TECHNICAL REPORT

A. Equipment

The new data acquisition system consisting of a Digital Equipment PDP-8 (small computer system), a Nuclear Data 560 Analog to Digital Converter and and ASR-33 teletype is now completely independent of the Scipp 1600 multichannel analyzer which has been returned to the Physics Department.

Due to design difficulties encountered in the analog to digital converter, modifications were made with some help from the Nuclear Data Corporation. With these modifications the ADC is now functioning well.

Others who might wish to utilize a photomultiplier base using constant fraction timing, should be alerted to the fact that an inherent design difficulty exists. This difficulty, unforseen by the manufacturer, consists of a discriminator problem in the photomultiplier bases (controlled by the discriminator control on the time pick offs). The adjustment of the discriminators on the time pick offs causes the cross-over point to rise above the base line. This drastically limits the usefullness of the discriminators. With certain newly developed calibration steps, we can alleviate part of this problem and obtain reproducible lifetime data.

B. Analysis

A variety of computer programming techniques have been evaluated in order to optimize analysis of the experimental data. In fitting the data the prompt resolution function must be unfolded and the number of exponential components determined.

The first program employed was the model of Lictenberger, 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 that T_0 (time zero) is a constant in this program. Our research showed that T_0 changed from run to run and could not be fixed. As a result, we have modified the program to make T_0 a floating parameter which is adjusted to give the best fit to the data. A good fit, however, was still not obtained due to the Gaussian rounding of the peak.

The Lictenberger 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 by this program are the annihilation rates, λ_j; channel number equivalent to time zero, T_o; intensities, I_{o_j}/λ_j; and background, B. The background and the intensities are assumed linear whereas time-zero and annihilation rates are non-linear. The FWHM of the apparatus is an input parameter to the program and is determined by measuring the Co^{60} spectrum.

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

$$I_j(t) = I_{o_j} \exp(-\lambda_j t) \qquad t \geq T_o$$

$$I_j(t) = 0 \qquad t < T_o$$

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

$$f_i = B + \sum_{j=\ell}^{k_o} F_{j,i}$$

with

$$F_{j,i} = \frac{I_{o_j}}{2\lambda_j}\left[Y_{j,i} - Y_{j,i+\ell} - \mathrm{erf}\left(\frac{t_i - T_o}{\sigma}\right) + \mathrm{erf}\left(\frac{t_{i+\ell} - T_o}{\sigma}\right)\right]$$

and

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

where

$$\sigma = \frac{FWHM}{2\sqrt{\ell n\ 2}}$$

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

In applying this program to our dtat it was found that the lifetime which was obtained from this analysis depends strongly on the value of the FWHM which is assumed (see Fig. 1). Using this program, we have fit one set of our data assuming various values for FWHM. Values of the variance of fit S^2 and the lifetime τ obtained in this way are shown on Figure 1. Note that the lifetime which is obtained depends strongly on the value of FWHM which is assumed in the analysis. As a result the initial determination of the FWHM is very critical; any fluctuations in the electronic noise or other parameters of the apparatus which change the FWHM of the apparatus will cause large errors in the lifetime data. Even with good temperature control (< ± 2°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 value of the FWHM used in the analysis.

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 T_o and FWHM were found to be strongly dependent on one another. This strong dependencey caused Marquart's algorithm to be inadequate to resolve 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 to solve 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 ϕ_{min} (Marquart's parameter) or Variance of Fit (Kiregaard and Eldrup) the later being directly related to ϕ_{min}. Marquart's parameter ϕ is:

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

with f(b) representing the count rate in channel no. i predicted by the model. Y_i is the number of counts in channel no. i. The weighting factor w_i is taken as $1/y_i$. ϕ_{min} is the smallest value of ϕ found by adjusting the parameters of the model, b. The Variance of Fit is defined as:

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

where $q = n - k_{free}$. n is the number of channels analyzed and k_{free} is the number of estimated free intensities and lifetimes in the parameter vector b. Since it was not possible to get a reliable fit of the theory to the experimental data when FWHM was one of the fitting parameters in the Marquardt fitting routine, we have used the following procedure. We calculate the variance of fit S^2 for a number of values of FWHM using the original Positronfit program which takes FWHM as a fixed input parameter. We then pick the value of FWHM which gives a minimum for S^2. This search procedure is done on the computer by a standard one-dimensional minimization routine based on the Fibonacci numbers[4]. The final value of FWHM is obtained by fitting a parabola to the three values of S^2 vs FWHM which are closest to the minimum. Once this value of FWHM is obtained, a fit is done using this minimizing FWHM and values for T_o and the positron lifetimes are obtained.

This modified program has been completed and does a much better job of 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 [5-8]. For example, a charge redistribution and a consequent electric dipole are created by the strain field which occurs around an edge dislocation(9). The negative part of the dipole repels electrons and has an attraction for the positron. As shown by Brown(10) 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.(11) performed experiments on the angular correlation of the photons from positron annihilation in deformed nickel alloys. These researchers found a change of the angular distribution curve from the annealed to the highly deformed state. This change suggested that, in the highly deformed state, annihilations occur when the positron is in the core of the dislocation. Grosskreutz and Millet(12) 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 33.5% Ni - 66.5% Co alloy.

In the Fourier analysis of X-ray line broadening(13), 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(14) and hence are of interest in connection with the current positron annihilation results.

Experimental Procedure

The pure nickel and the nickel-cobalt alloy used in this research were obtained from the International Nickel Company. Nickel (99.99 + % purity) and a binary 33.5% Ni - 66.5% 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 33.5% Ni - 66.5% 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 Bollman solution. Both samples were fatigued at a mixImum bending stress of 45 ksi.

The positron lifetime data were analyzed as discussed in Section 3. The data were corrected for a linear background level and for 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 accumlated during this tjme. 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[14]. After fatiguing the specimens a specified amount, positron annihilation and X-ray data were taken. The same procedure was repeated a various intervals until the specimen fractured.

The Warren-Averbach Fourier analysis[13] 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[15] 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[13] 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 the density of vacancies is in thermal equilibrium, the number of vacancies is so small that their contribution to the positron lifetimes is negligible[16]. Grosskreutz and Millet[12] 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, 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 - 66.5% Co as can be seen from Figures 2 and 3, 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. It might be noted that the zero deformation lifetimes of the Ni and the Ni-Co alloy are different by less than 10 psec. This in itself is an important finding because it indicates that stacking faults are not very strong trapping sites for positrons.

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 2 and 3 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 33.5% Ni - 66.5% 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.[17,18] the stacking fault energy influences the fatigue life more than the yield strength of the material.

In the Ni and Ni - 66.5% Co alloy specimens, the slope of the curve of lifetime vs number-of-cycles changed drastically approximately at 7000 and 22,800 cycles, respectively (Figures 2 and 3). 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.[19] 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 approximately linear up to deformation of 8%. Saturation effects have also been observed in plastically deformed Ni[20]. 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 - 66.5% 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[21] for Ni fatigued at the same stress level on the same type of fatigue machine.

After fatigue failure in the 66.5% 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 <111> direction in the Ni - 66.5% Co (Figure 4) decreased from 1600 Å at 10,000 cycles to 440 Å at 400,000 cycles (prior to fracture). In the <100> direction the particle size decreased from 800 Å to 480 Å as shown in Figure 5.

The root mean square strain normal to the reflection planes were also measured in the Ni - 66.5% alloy (Figure 6). The mean square microstrains $\langle\varepsilon^2\rangle$ were independent of the crystallographic orientation. As shown by Williamson and Smallman[22] and Smallman and Westmacott[23] 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:

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

where $<\varepsilon^2>$ is the mean squared microstrain, D is the particle size and b is the magnitude of the typical f.c.c. Burgers vector. Figure 7 shows how the dislocation density, calculated by the above relationship, varies as a function of fatigue.

CONCLUSIONS

1. The X-ray particle size and the mean positron lifetime both change rapidly early in the fatigue process. Thus, it appears that positron annihilation lifetime data can be used as an effective tool for studying early fatigue damage. Since the intensity of defect trapping, I_d, is directly proportional to the defect concentration the researcher may use positron annihilation as a non-destructive method for estimating dislocation density before I_d saturates.

2. It was noted both in Ni and the Ni-Co alloy that the positron lifetime saturated earlier than did the X-ray particle size. However, it seems significant that both parameters saturate much sooner in Ni than in the Ni-Co alloy. This suggests that the defect density builds up more rapidly in the higher SFE materials, as would be expected.

3. A limiting X-ray particle size, independent of crystallographic direction, was reached during fatigue. The limiting particle size, however, was approached more rapidly along the <111> direction than along the <100> direction in the Ni - 66.5% Co alloy.

4. The mean positron lifetime saturated earlier than did the X-ray particle size or calculated dislocation density.

5. At fracture, agreement was found between the cell size observed in TEM and the X-ray particle size to within a factor of two.

6. Most of the cracks were initiated along grain boundaries in the Ni - 66.5% Co alloy; in the Ni, most cracks initiated along slip bands within the grains.

A paper describing this work has been prepared and submitted for publication in the Journal of Applied Physics.

D. Grain Size Effects in Cu

Introduction

Since grain size determines in large measure work hardening, it is important to study the positron-grain boundary interaction before and during deformation. The only literature citation concerning the existance of a grain boundary - positron interaction is a brief mention by Weisberg and Berko[24] that different lifetimes were noted for single crystals and polycrystals of Fe and Be. This 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

Early data were reported in Table 1 of COO AT(11-1)-2128-3, however additional and more accurate values are shown in the current Table 1. These new values are a result of the new computer analysis of the data. Clearly grain boundaries act as trapping sites for postrons, 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 more rapidly the smaller the grain size, since for a given total strain a smaller grain size should experience more dislocation generation and interaction with consequent increased positron trapping. Thus in Figure 8 the mean lifetime increased with increasing tensile strain. Saturation begins at a concentration dependent upon the grain size; the 169, 264, and 309 μ grain size materials saturated at approximately 10, 14, 15%, respectively. These results are similar to Kusmiss, et al.[5] where saturation occurred somewhere between 8 and 16% deformation of Pt. To make sure that no large voids were being produced during tensile deformation, all the Cu specimens were given a radiographic examination at the facility of the Universidad Central de Venezuela. No indication of voids was seen.

Since trapping at dislocations should be similar to that at vacancies, the trapping models of Bergensen and Scott[25], Connors and West[26], and Seeger[27] 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 in percent of the two lifetimes as a function of strain in the 264 μ grain size Cu.

Table 1*

Effect of Grain Size on
Positron Lifetime in 99.999% Copper

Average Grain Size (μ)	Mean Lifetime τ_1 (psec)	Treatment
Single Crystal	157	Melt Grown
309	162	Cold rolled 90.7%, Annealed 30 min. at 600°C
264	164	Cold rolled 90.7%, Annealed 25 min. at 600°C
169	173	Cold rolled 90.7%, Annealed 5 min. at 620°C
59	166	Cold rolled 90.7%, Annealed 15 min. at 600°C
40	170	Cold rolled 90.7%, Annealed 7 min. at 600°C
26	174	Cold rolled 90.7%, Annealed 3.5 min. at 600°C
3	176	Cold rolled 90.7%, Annealed 1 min. at 600°C

*Since COO AT(11-1)-2128-3 and COO AT(11-1)-2128-4 previously reported data have been reanalyzed using the new program and new grain size data added.

Table 2

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

% Strain	0	5	10	15	20	40
Intensity τ_f	96.3	71.2	31.0	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	100.0	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

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 assumes 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 analysis of section (B) where only a single lifetime - the mean lifetime was used. This suggests that the present trapping models are applicable.

In the present case of plastic deformation, the principal defects are dislocations. As shown by Kuribayashi, et al.[28], 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[12] 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[25,26], the effects of 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 sample, the 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 boundar sliding did occur, this will keep the dislocation density at a much lower level than is expected for specific strains. Since grain boundar sliding should not occur at room temperature even with this small grain size, the first explanation seems more reasonable.

E. Hydrogen Embrittlement

Introduction

Since a rather large section (pgs. 19-34) of COO AT(1101)-2128-4 "Final Progress Report 1/1/72 - 12/31/72" presented findings on hydrogen embrittlement since. The issuance of COO AT(11-1)-2128-3, "Technical Progress Report" dated September 1972, we would like to take the liberty of not repeating those findings here, but rather simply call attention to their location.

A series of experiments*, soon to be described, were performed during the period of January through April 1973. The purpose of these tests was to:

(1) Determine the realtionship of hardness to positron lifetime.

(2) Substantiate the effect of absorbed hydrogen on positron lifetime noted during the October 1972 tests and determine how hardness level affects this relationship.

*We will not burden the current report with the full details of the testing program, but rather will summarize them and present the full particulars in publications.

(3) Determine the effect of cold work on positron lifetime and relate any changes to those as a result of absorbed hydrogen.

(4) Further delineate the effect of cadmium thickness on the surface of a steel specimen.

Experimental Procedure

Except for reruns of HEP specimens , #1 and #26 from the October 1972 series all specimens in the current series were AISI 4340. Flat tensile specimens were machined from a 6" x 6" bar so that the broad sides of the specimens were perpendicular to the direction of rolling. This results in the longest dimension of the grains being perpendicular to the surface against which the positron source is placed. Spectrochemical and carbon analyses were performed.

Specimen Preparation

The steel specimens were tested in four different heat treatment conditions and before and after various stages of embrittlement, plating, stripping and straining as summarized below.

a. Heat Treatment.

The four different heat treated conditions used were: (1) annealed (Rb 90.5), (2) Rc 37, (3) Rc 43, and (4) Rc 49.5 to 51. The annealed samples correspond to the steel in the condition as received. The three remaining hardnesses were obtained by heating the machined specimens to 1550°F for one hour in an endothermic atmosphere with a dew point of 52°F. They were then quenched in oil and subjected to two one hour tempers to yield the appropriate hardness given above. Metallographic examination of a cross section revealed decarburization extending to less than 0.001" depth.

b. Electropolishing.

In order to remove any distorted layer from machining or decarburization approximately 0.005" per surface was electrochemically removed from the reduced section.

c. Baking.

All 4340 steel specimens except those used in tests 100 through 109 were baked at 375 to 450°F for 24 hours subsequent to electropolishing to insure removal of any hydrogen effects. This was performed in an argon atmosphere to prevent oxidation and minimize discoloration.

d. Embrittlement.

Sepcimens designated as "embrittled" were subjected to an electrolytic bath for 100 minutes during which the specimen was the cathode. The electrolyte consisted of IN H_2SO_4 with 0.2 grams As_2O_3 per liter added as a poison. A current density of 0.1 smps/cm^2 was applied.

e. Plating.

Some specimens were given a bright, embrittling cadmium plate approximately 0.0003" thick without subjecting them to a special embrittling process while others were plated immediately after embrittling as described above.

f. Stripping.

The cadmium was removed by immersing the sample in a 10% by weight ammonium nitrate aqueous solution for approximately 7 minutes at about 130°F. This was followed by rinsing with distilled water.

g. Straining.

The flat tensile specimens which were subjected to plastic elongation over a one inch gage length in an Instron tensile machine.

Positron Lifetime Results

a. Hardness vs. Positron Lifetime.

Figure 9 shows the relationship of lifetime to hardness. The annealed specimens yielded hardness values of R_B 90.5 or R_C 7. The circles correspond to specimens prepared by baking after electropolishing, while the "x'es" correspond to specimens where the baking was ommitted.

There is a definite trend for positron lifetime to increase with hardness although it is also apparent that baking has no consistent effect.

It is interesting that the hardness and lifetime decrease as tempering proceeds. This implies that the decreasing density of line and point defects is more of a controlling factor than is the increasing volume fraction of carbide precipitates during tempering. Another possible interpretation is that carbides provide sites for rapid annihilation.

b. Hydrogen Embrittlement.

Figures 10a through 10d show the relationship of positron lifetime to electrochemical processing. Here again definite trends are noted despite considerable variation. In all four hardness levels plating resulted in an increase in positron lifetime over the bare condition. Stripping the cadmium from the specimen resulted in a decrease in lifetime but the lifetime was still in excess of that prior to plating. For example, the arithmetic average of the annealed specimens increased 15 psec upon plating and remained 14 psecs above the starting point even after stripping. The Rc 37 specimens increased 24 psecs upon stripping but remained 12 psecs above the starting level upon stripping.

The arithmetic average of the Rc 43 specimens increased 17 psecs upon plating but still remained 12 psecs above the starting point upon stripping. A lesser number of tests were run on specimens in the Rc 49.5 - 51 hardness range so results are not as reliable. Even so, the positron lifetime increased 31 psecs upon plating and still remained 9 psecs above the starting level after stripping. There was no discernable increase in lifetime as a result of embrittleing prior to plating, as opposed to plating only, even though actual cracking resulted in the higher hardness levels as a result of the embrittlement treatment.

c. Relationship of Cold Work and Positron Lifetime.

As seen in Figure 11, there is a definite trend between positron lifetime and tensile strain. Upon initial straining of soft specimens the positron lifetime increases very rapidly and reaches a maximum around 3% elongation. It then levels off or even drops slightly. The harder materials generally exhibit a higher starting lifetime but a more gradual rate of increase of lifetime with strain.

The above results suggest that vacancies and/or dislocations may be near the saturation level in the higher strength level specimens. As related to the effect of the embrittlement treatment it may suggest the reason for the cracking of the high strength steel specimens.

d. Effect of Cadmium on Surface.

Results of tests with two thicknesses of cadmium interposed between source and specimen indicate an increase in lifetime with increased thickness. Except for possible surface effects, this increase in lifetime is probably due to the higher proportion of positrons which are trapped in the cadmium and the inherently longer lifetime associated with cadmium.

F. Other Activities on Contract

1. A manuscript describing the Ni and Ni-Co fatigue results has been submitted to the Journal of Applied Physics.

2. A short note on trapping of positrons at stacking faults is in preparation.

3. A paper on grain size effects in connection with deformation of copper is in preparation.

4. University support for an M.T.S. fatigue testing system is expected. This will greatly assist our fatigue studies.

5. Independent support for the purchase of angular correlation equipment is being sought. This would greatly amplify our capacity to study various defects.

6. This group is to prepare a review article on positron annihilation for the series "Treatise on Materials Science and Technology" - early in 1974.

7. Experiments are beginning of the fatigue hardening and fatigue softening of 4340 steel.

8. Some preliminary results on fatigue in Aℓ have been gathered.

9. Discussions and planning have begun with the linear accelerator staff at the University of Utah in order to examine positron annihilation at voids.

10. Arrangements have been made with Oak Ridge N. L. to obtain carefully made alloys of Aℓ plus from 2 ppm to 60 ppm Au in order to look at salute effects both in grain boundaries and within grains.

11. Dr. G. Miller (Materials Science and Engineering Division and Physics Department) has been participating in theoretical studies within the group over the summer. This is to put us in a better position to analyze current results and also to look ahead to the possibility of angular correlation work and void studies.

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LIST OF FIGURES

Figure 1 Typical appearance of the variance of fit and the primary lifetime of the positron annihilation experiment plotted as a function of the FWHM assumed in the data analysis.

Figure 2 The mean positron lifetime of pure Ni plotted as a function of cyclic deformation of 45 ksi.

Figure 3 The mean positron lifetime of Ni - 66.5% Co plotted as a function of cyclic deformation at 45 ksi.

Figure 4 X-ray particle size $D_{<111>}$ of Ni - 66.5% Co as a function of fatigue life.

Figure 5 X-ray particle size $D_{<100>}$ of Ni - 66.5% Co as a function of fatigue life.

Figure 6 Root mean square average microstrain along the <111> direction at various depths perpendicular to the diffracting planes for the 66.5% Co alloy during fatigue at 45 ksi. From top curve down, the depths are 60, 80, 100 and 120 A, respectively.

Figure 7 The correlation between calculated dislocation density and fatigue damage for Ni - 66.5% Co.

Figure 8 Positron lifetime versus tensile strain for copper specimens of various grain sizes.

Figure 9 Positron lifetime versus hardness for AISI 4340 steel.

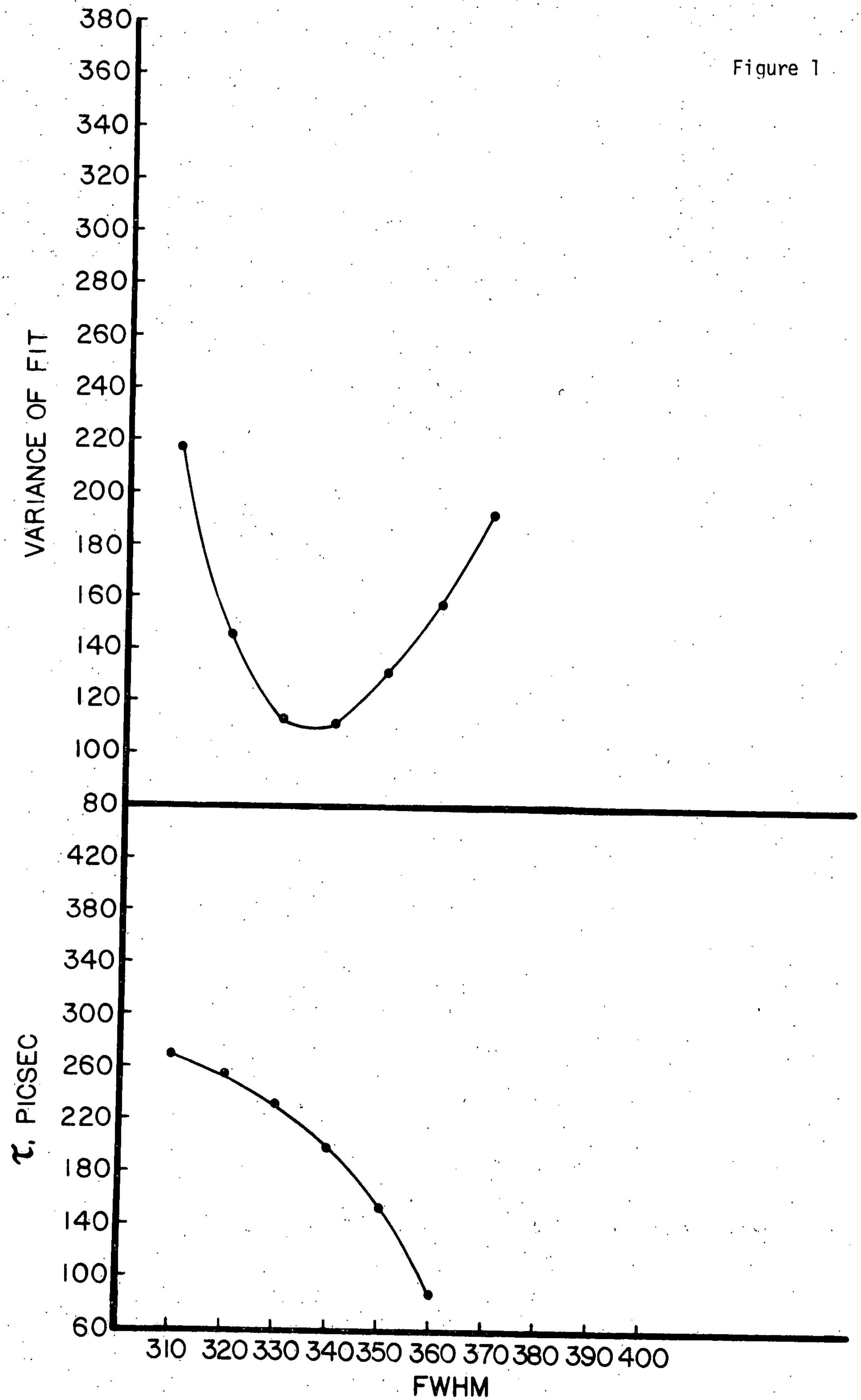
Figure 10 Positron lifetime as a function fo electrochemical processing of 4340 steel.
a. annealed (Hardness = Rc 7)
b. Hardness = Rc 37
c. Hardness = Rc 43
d. Hardness = Rc 49.5/51

Figure 11 Positron lifetime versus tensile strain for 4340 steel for materials of initial hardnesses of Rc 7, Rc 37, Rc 43 and Rc 49.5/51.

III. Time Information on Principal Investigator

The principal investigator, Dr. J. G. Byrne, has spent three months full time during the summer of 1973 on this research. During the 1972-1973 academic year, he spent the equivalent of one month full time on this research while at the Universidad Central de Venezuela. Dr. Roland Ure, acting principal investigator during the 1972-1973 academic year, spent one month full time during that academic year (at no cost to AEC).

Figure 1


380
360
340
320
300
280
260
240
220
200
180
160
140
120
100
80
VARIANCE OF FIT
420
380
340
300
260
220
180
140
100
60
τ, PICSEC
310
320
330
340
350
360
370
380
390
400
FWHM

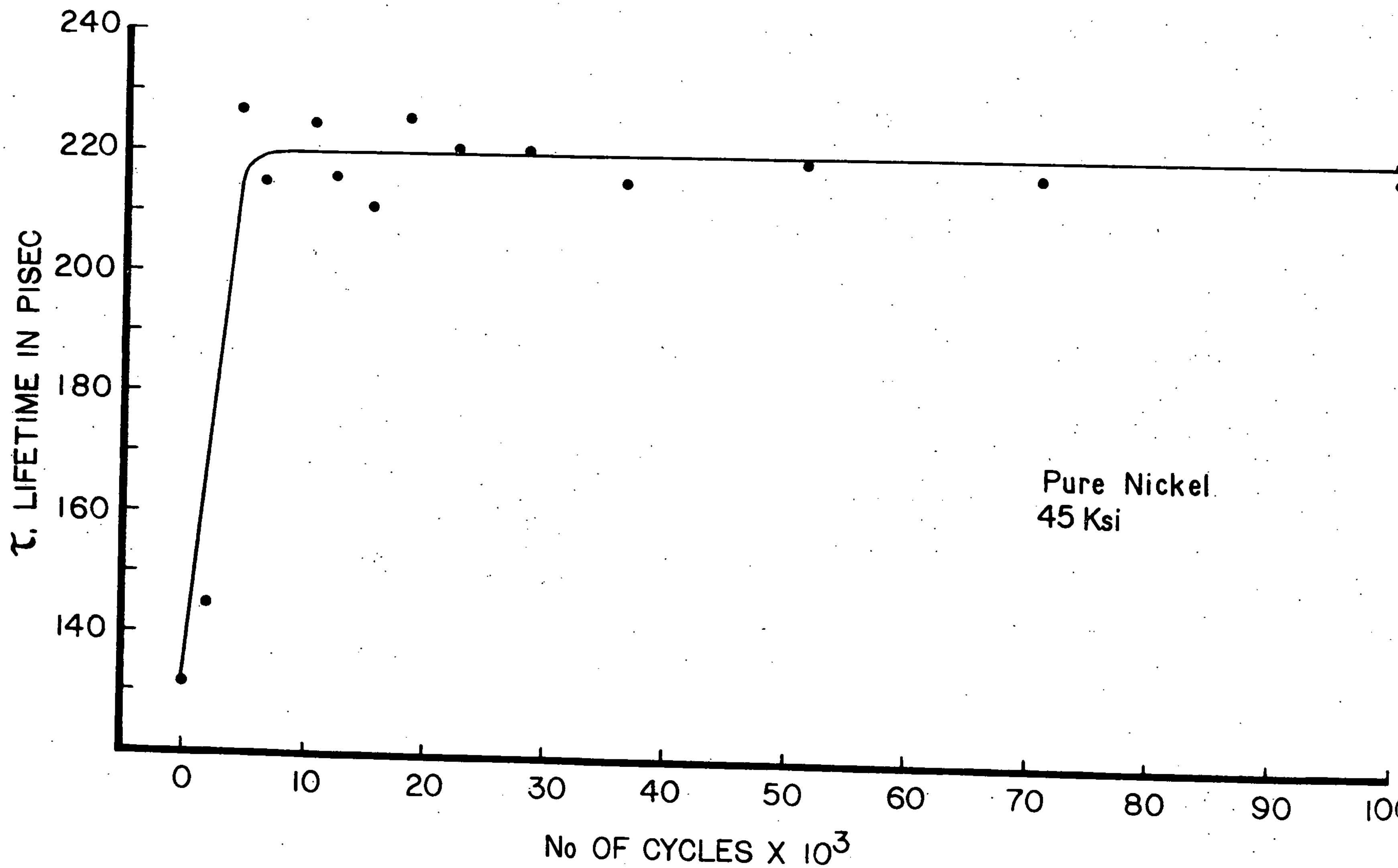
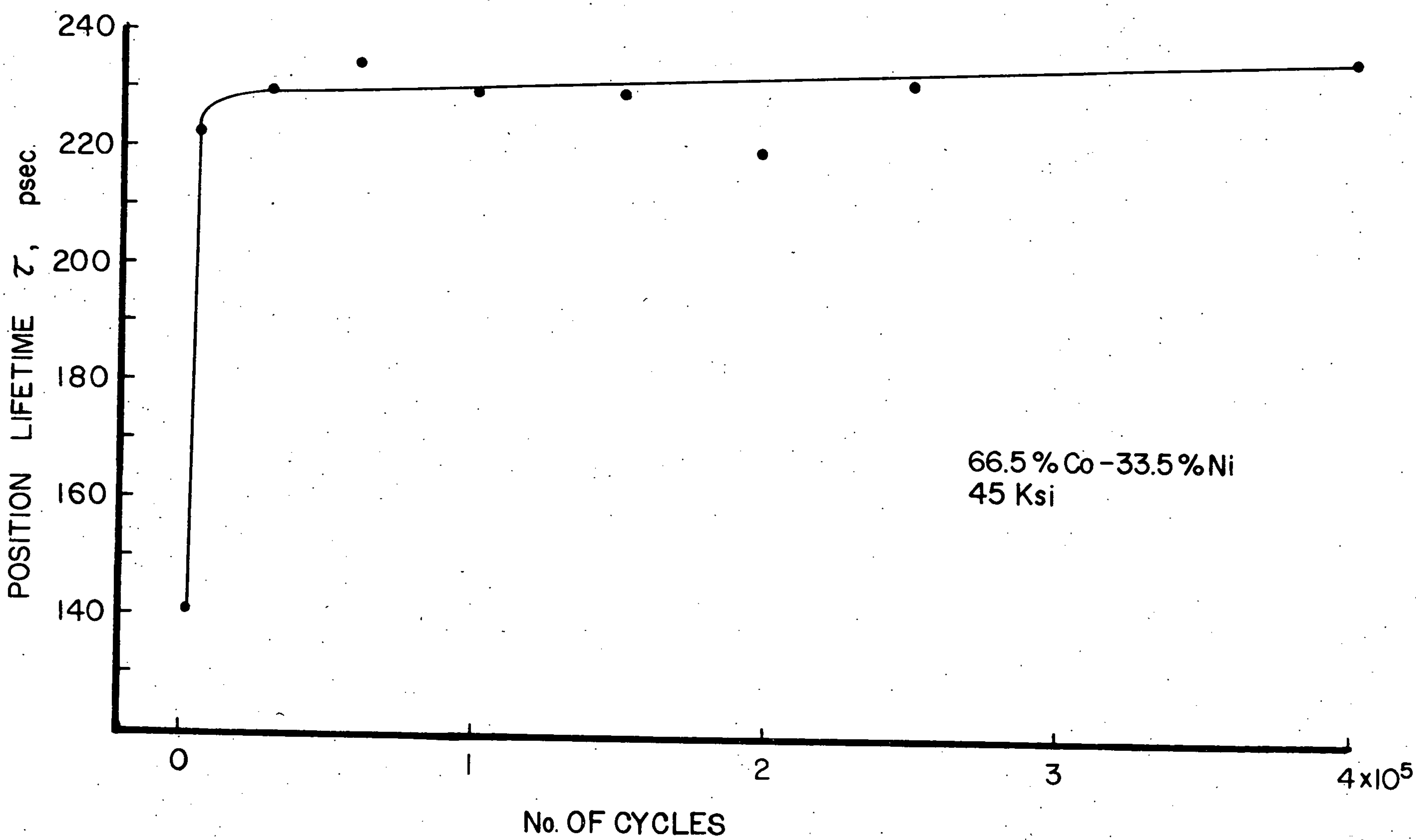

240
220
200
180
160
140
τ, LIFETIME IN PISEC
0
10
20
30
40
50
60
70
80
90
100
No OF CYCLES X 10^3
Pure Nickel
45 Ksi

Figure 2



240
220
200
180
160
140
POSITION LIFETIME τ, psec.
0
1
2
3
4x10^5
No. OF CYCLES
66.5 % Co - 33.5 % Ni
45 Ksi

Figure 3

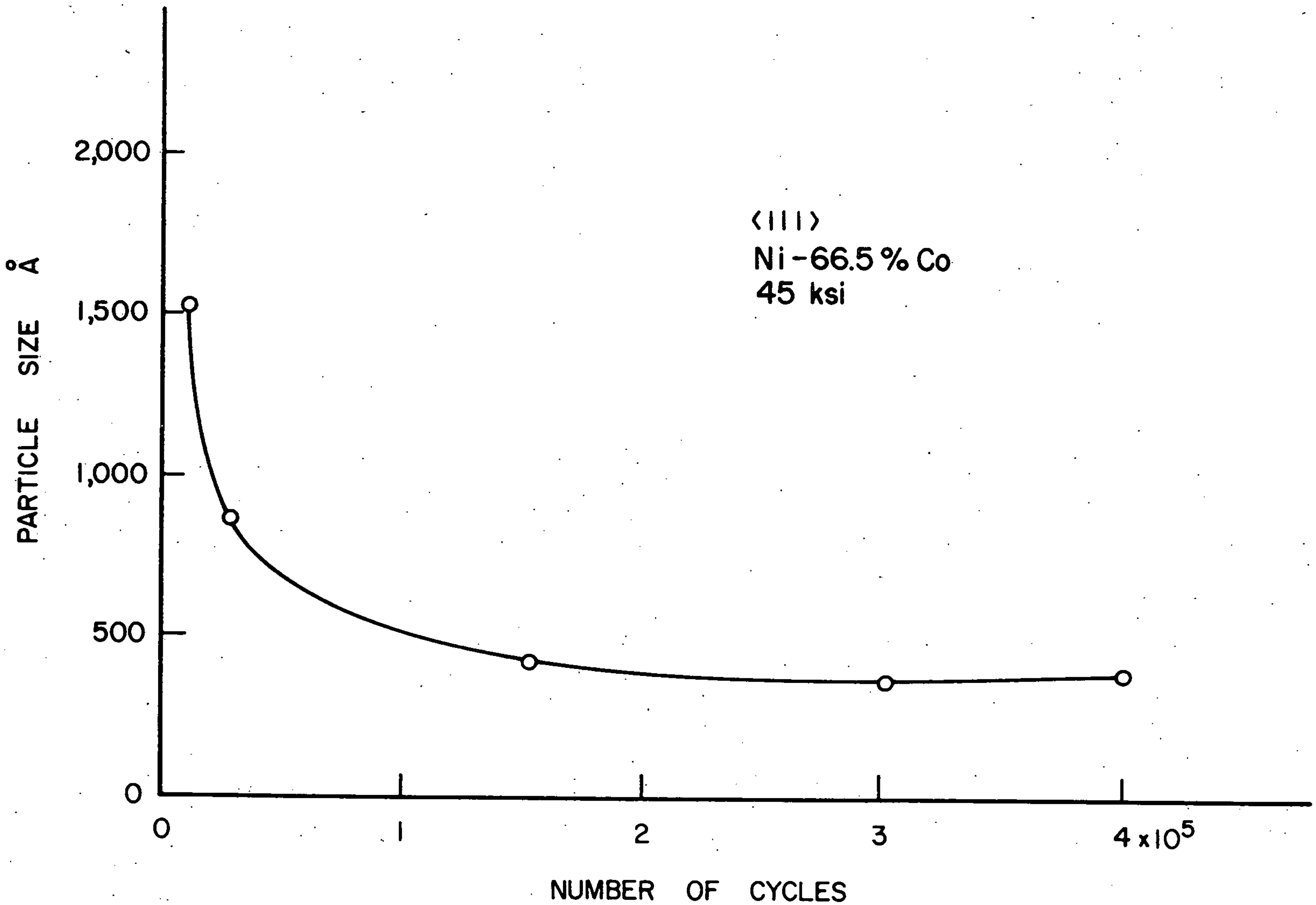

2,000
1,500
1,000
500
0
PARTICLE SIZE Å
⟨111⟩
Ni-66.5 % Co
45 ksi
0
1
2
3
4 x10^5
NUMBER OF CYCLES

Figure 4

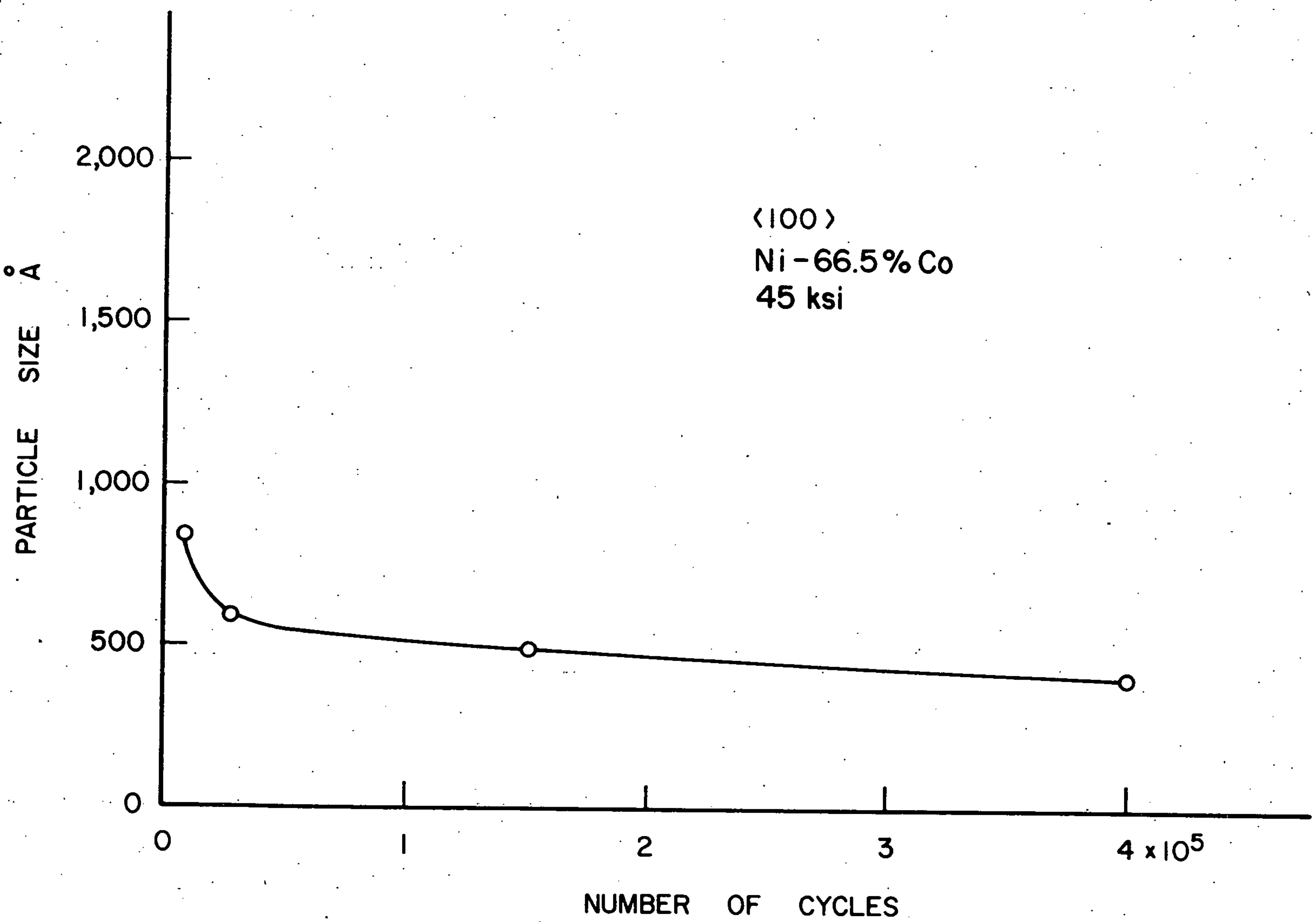

⟨100⟩
Ni-66.5% Co
45 ksi
PARTICLE SIZE Å
2,000
1,500
1,000
500
0
0
1
2
3
4 x 10^5
NUMBER OF CYCLES

Figure 5

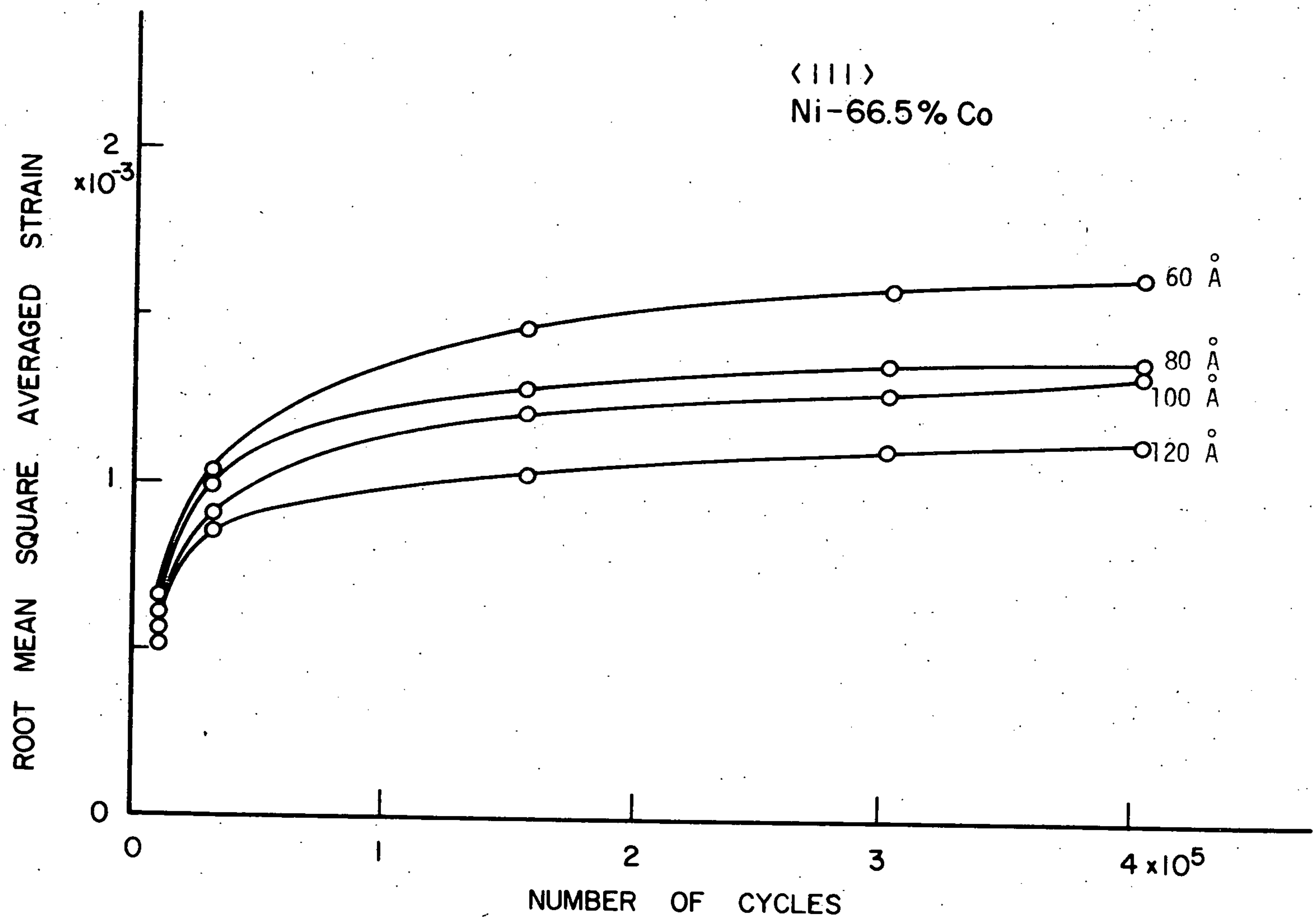
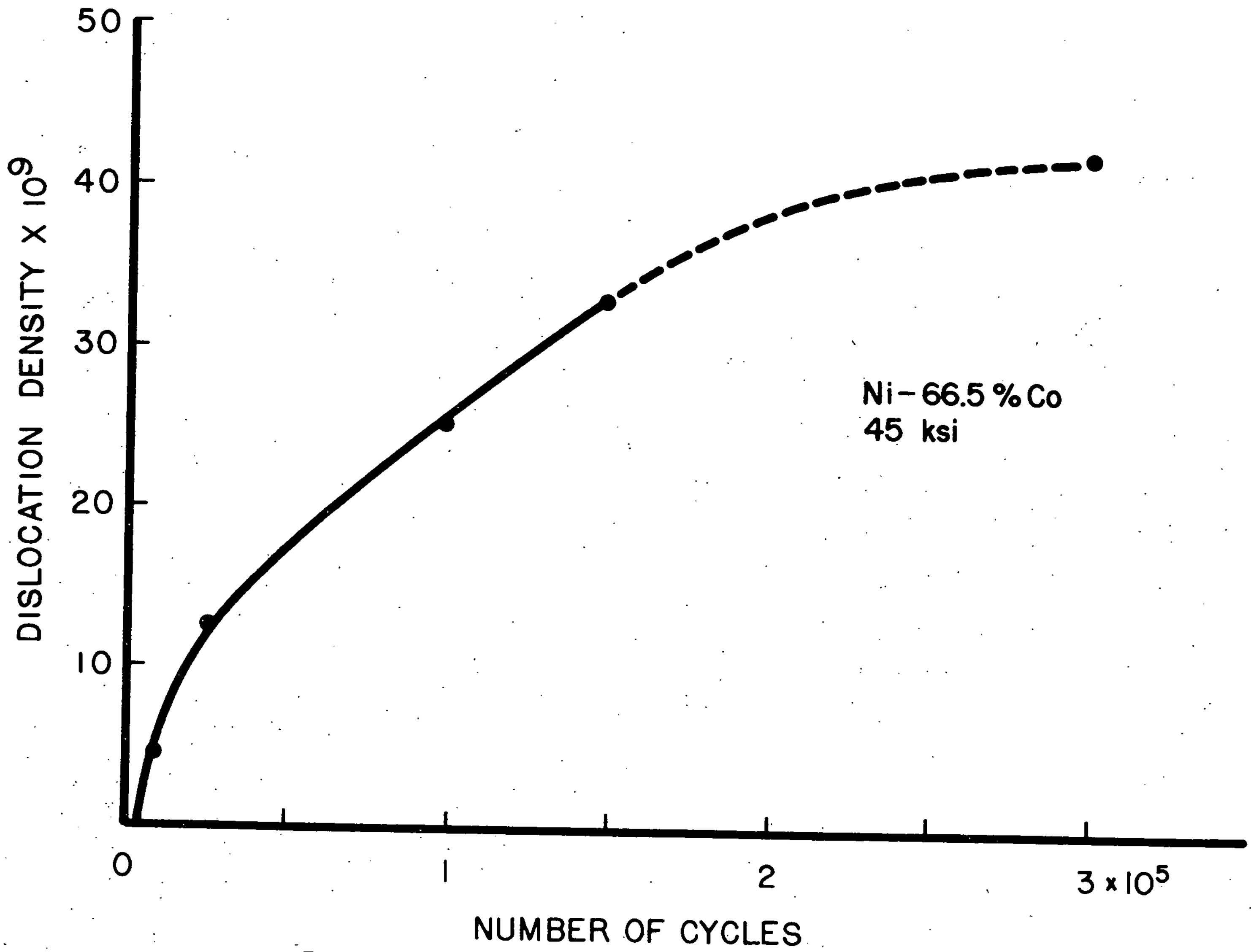
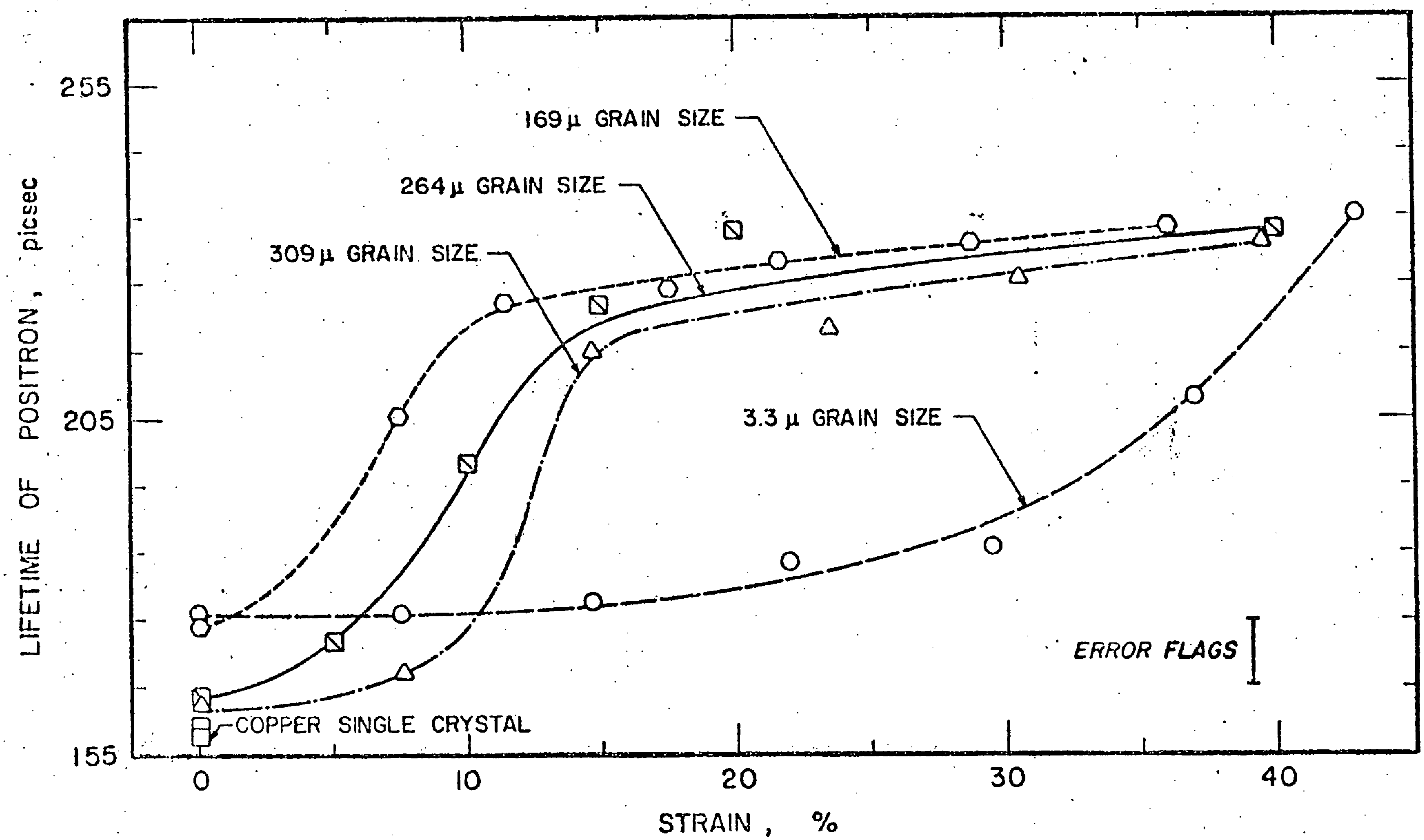

⟨111⟩
Ni-66.5% Co
ROOT MEAN SQUARE AVERAGED STRAIN
2
x10^-3
1
0
60 Å
80 Å
100 Å
120 Å
0
1
2
3
4 x10^5
NUMBER OF CYCLES

Figure 6



50
40
30
20
10
0
1
2
3 x 10^5
DISLOCATION DENSITY X 10^9
NUMBER OF CYCLES
Ni-66.5 % Co
45 ksi

Figure 7



Copper Samples 99.999^{+} % Purity.

Figure 8

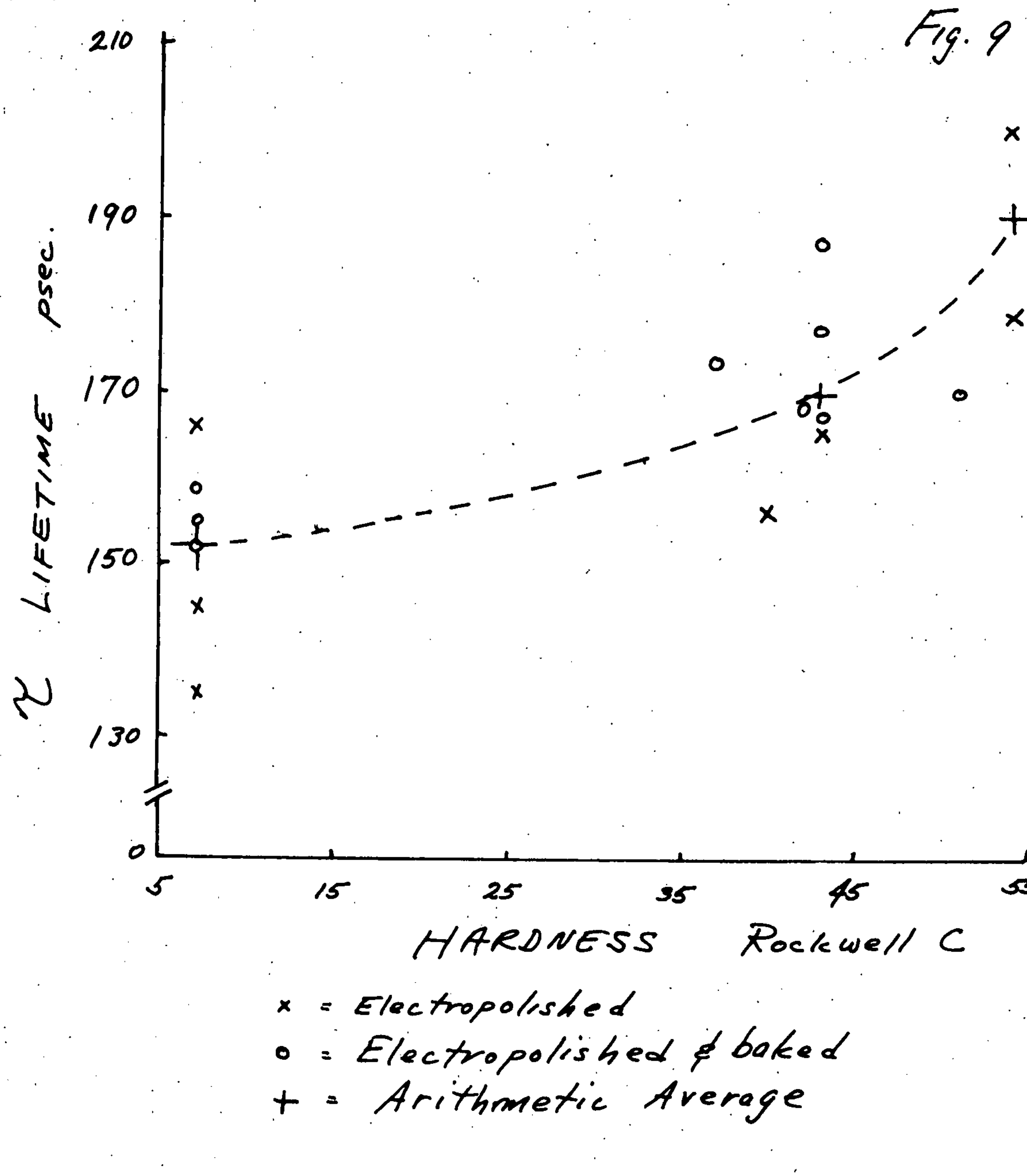


Fig. 9
210
190
170
150
130
0
τ LIFETIME psec.
5
15
25
35
45
55
HARDNESS Rockwell C
x = Electropolished
o = Electropolished & baked
+ = Arithmetic Average

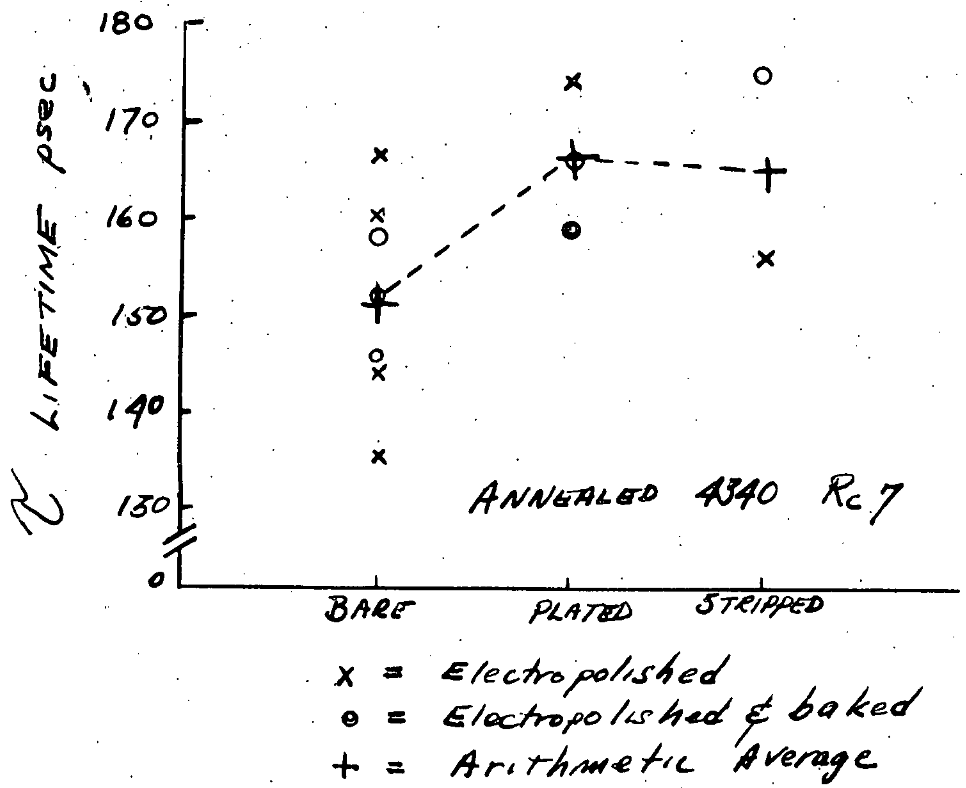


Fig. 10a

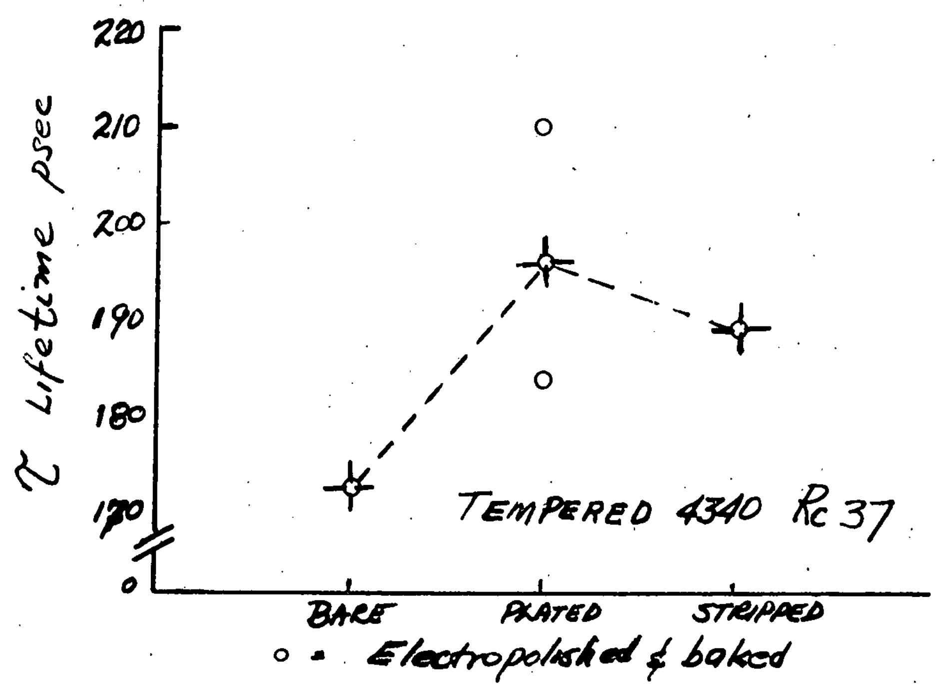


Fig 10b

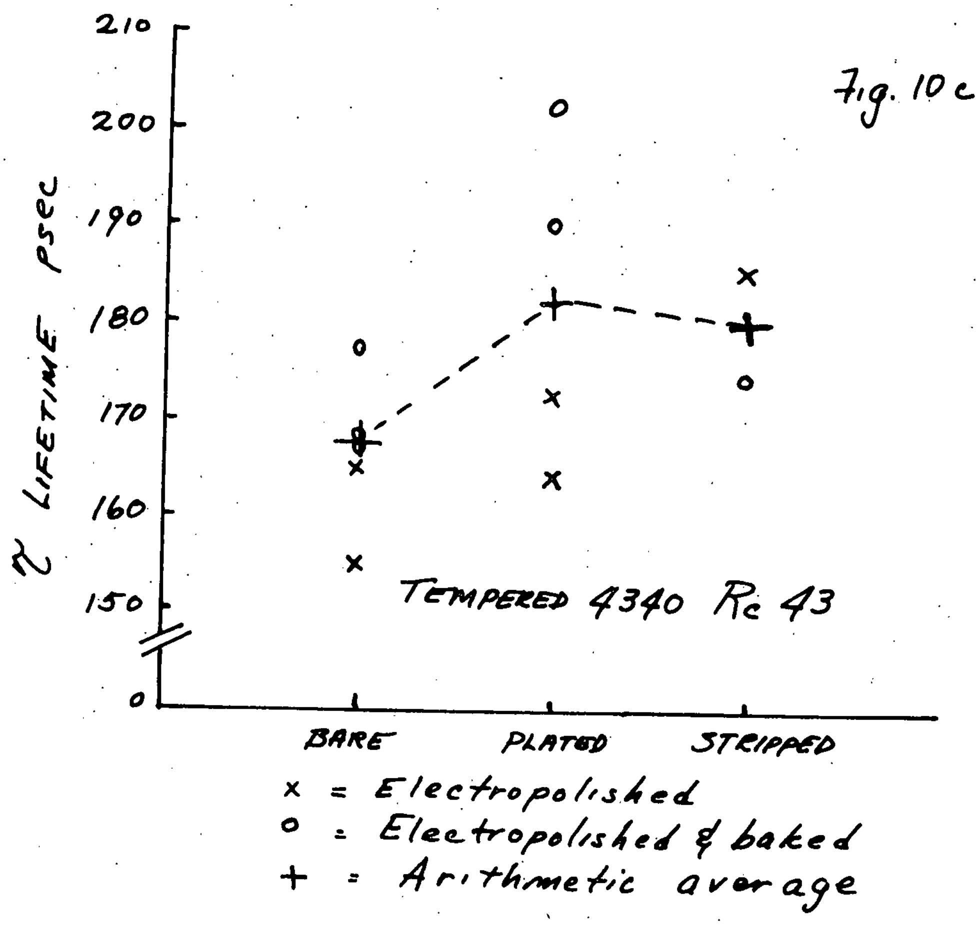


Fig. 10c
210
200
190
180
170
160
150
0
τ LIFETIME psec
TEMPERED 4340 Rc 43
BARE
PLATED
STRIPPED
x = Electropolished
o = Electropolished & baked
+ = Arithmetic average

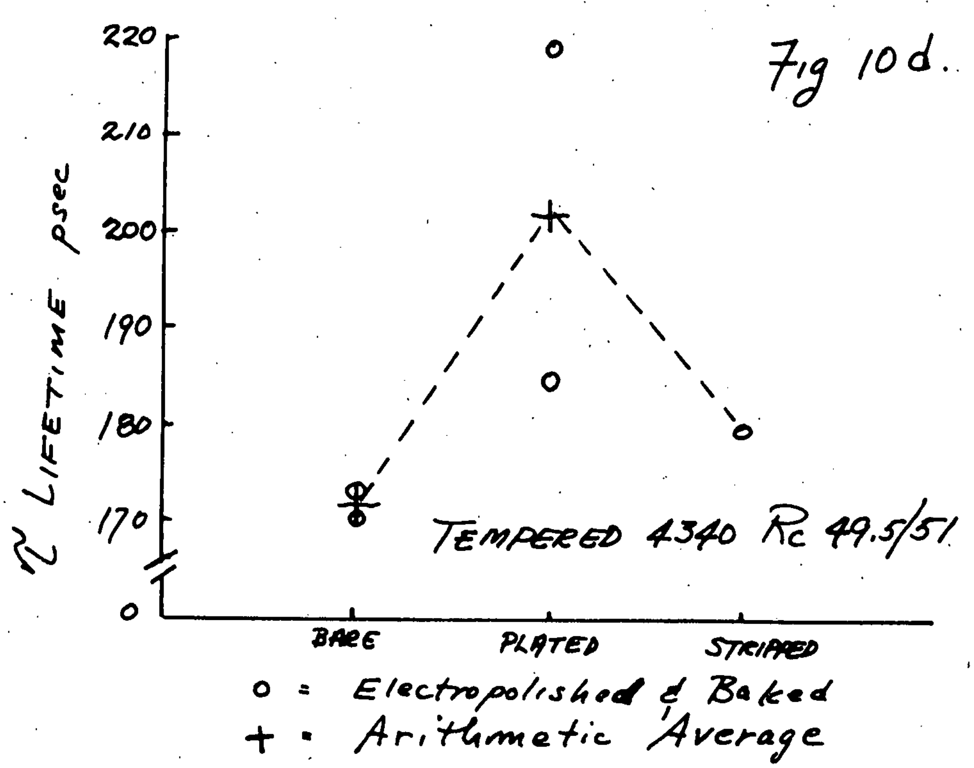


Fig 10d.
220
210
200
190
180
170
0
τ LIFETIME psec
TEMPERED 4340 Rc 49.5/51
BARE
PLATED
STRIPPED
o = Electropolished & Baked
+ = Arithmetic Average

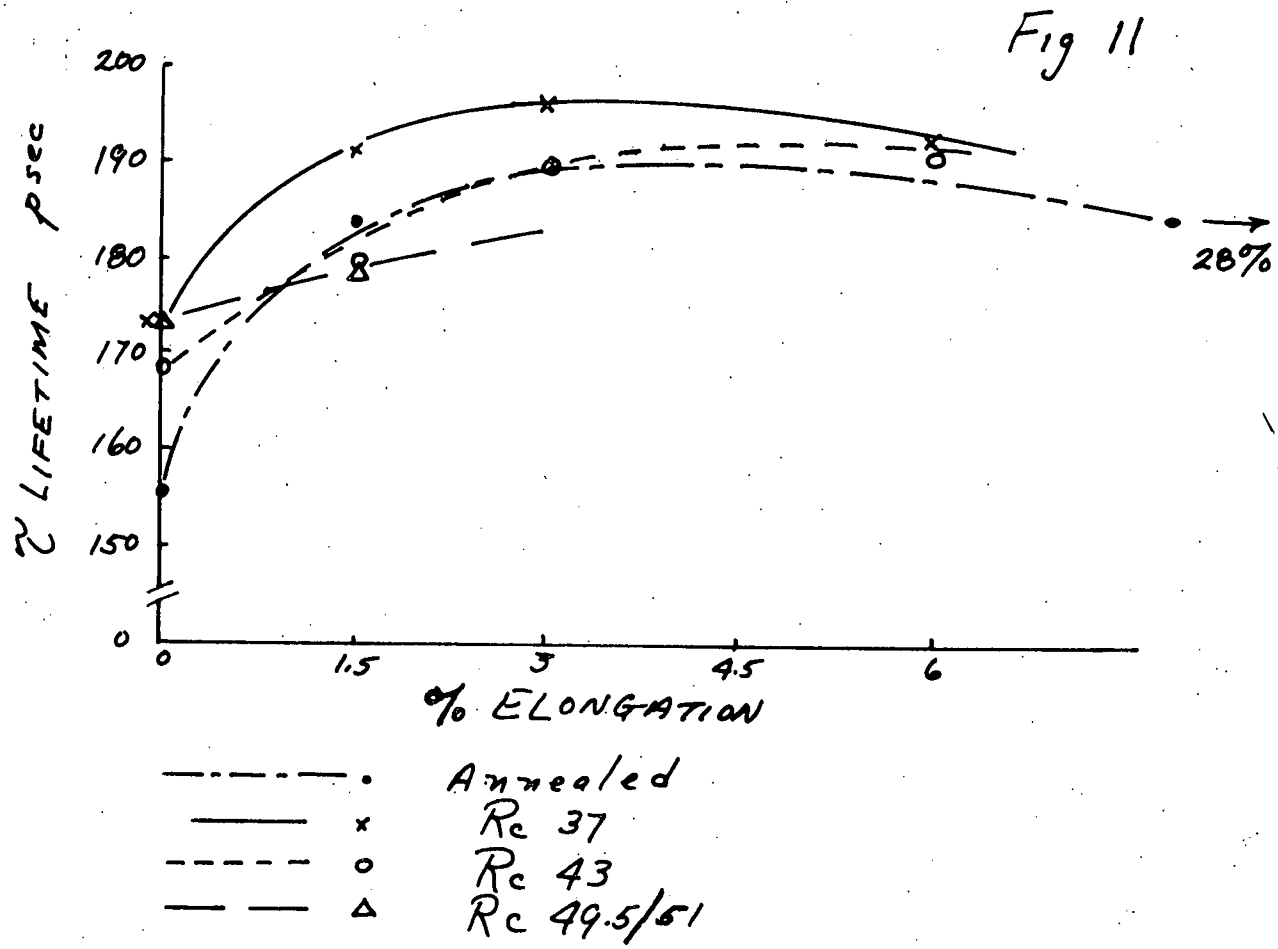


Fig 11
200
190
180
170
160
150
0
τ LIFETIME psec
0
1.5
3
4.5
6
% ELONGATION
28%
Annealed
Rc 37
Rc 43
Rc 49.5/51