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HIGH TEMPERATURE ANNEALS OF
DEFECTS QUENCHED IN METALS

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Technical Progress Report

Because of lack of graduate assistants, several of the projects proposed have not yet been initiated. The status of each as outlined in the proposal is listed below.

A. High Temperature Anneals.

The construction of a suitable circuit for accurate pulsing has been completed and eight anneals have been made. This number is insufficient to draw any firm conclusions. A description of the feedback and control circuit and a discussion of preliminary results are given in greater detail in Technical Report C00-1456-3, copies of which are enclosed with this proposal.

B. Pressure Effect on the Anneal of Quenched-In Resistance in Aluminum.

Preliminary measurements on aluminum indicated an activation volume of about $\frac{1}{2}$ of an atomic volume. The scatter in these values was about $\pm 30\%$. To reduce this scatter, the annealing bath temperature control system was drastically modified. The bath now controls to such an accuracy that no temperature fluctuations can be detected with a chromel-alumel thermocouple. The quenching apparatus was also improved to reduce the possibility of straining the specimen and to increase the accuracy of the pre-quench temperature measurement. Two determinations of the motional volume ΔV_m^{eff} have been made since these modifications. Quench temperatures were 580°C and 583°C and the annealing temperature was -66.3°C and the annealing pressure $8.6 \text{ kg/cm}^2/\text{atm}$ and $8.9/.45 \text{ kg/cm}^2$. These resulted in values for ΔV_m^{eff} of .17 and .20 atomic volume.

C. Effect of Mechanical Strains on High-Temperature Annealing.

This research will not be started until the basic high temperature annealing study is near completion.

D. Application of High Temperature Pulse Technique to the Study of Guinier-Preston Zone Formation in Alloys.

At the time this proposal was made, a metallurgy graduate student was available to perform the measurements. Delays in

developing the pulsing apparatus prevented the use of his services, as he left the University shortly after the working control was completed. This problem can now be undertaken whenever a graduate student with a suitable metallurgical background becomes available.

E. High Temperature Pulsing in Liquid Gases.

Again, the delay in completing the pulsing circuitry prevented initiation of this phase of the project.

PUBLICATIONS

The following publication resulted from the partial support of this contract:

Roy M. Emrick and Brent J. Buescher, Pressure Effect on Quenched-In Defect Migration in Aluminum, Bul. Am. Phys. Soc. 11, 184 (1966).

Technical Report
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Apparatus for Producing High Temperature
Pulses in Metal Wires

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Introduction

The study of point defects in metals by observing the quenching in and the subsequent annealing of excess resistivity in small diameter wires was conceived and developed by Kauffman¹ in 1952. The basic assumption in this technique is that the excess resistance quenched in is directly proportional to the concentration of point defects quenched in. Numerous theoretical and experimental determinations yield a typical proportionality constant of $1.5 \mu\Omega \text{ cm/atomic } \%$ of vacancies. Quenching and resistance measuring techniques have been refined and extended considerably since that time. For a detailed description of quenching methods and resistance measuring techniques, see the technical report by Emrick.²

The DC measuring techniques require that the total annealing time be very much greater than the time for making a resistance measurement (about two minutes) or that the specimen be transferred from a cold measuring bath to the annealing bath, held there for the required time and returned to the cold bath for measurement. The latter technique runs the risk of deforming the specimen by repeated handling and in inaccurate annealing time measurements when the annealing time becomes small. Thus, in gold, annealing temperatures have been limited to about 100°C and in aluminum to about 30°C .

Coupled with this experimental limitation is the discovery that the annealing process in the fcc metals is quite complex. It is generally accepted that the dominant defect quenched in is the vacancy. Single vacancies can reduce the strain energy of the crystal by combining to form divacancies. Higher order clusters are also possible. Thus, in general, not only single vacancies, but divacancies as well will be moving in the lattice. (The tri-vacancy is believed to have very low mobility.) If we denote the single vacancy motional energy by E_m^1 , divacancy motional energy by E_m^2 and the divacancy binding energy by E_B^2 , the effective motional energy E_m^{eff} will be $E_m^{\text{eff}} = E_m^{\text{eff}}(E_m^1, E_m^2, E_B^2)$, even in a simple approximation.³

Measurements of the vacancy formation energy E_f^1 are not so complicated. Calculations⁴ show that the resistance of a divacancy is within a few percent of the resistance of two single vacancies. At the elevated pre-quench temperature, thermal energy will keep the vacancies separated. Even if they combine during the quench, the resistance measurement will still give the pre-quench vacancy concentration assuming no vacancies are lost during the quench.

Also of interest in the study of point defects is the kinetics of the annealing reaction. Sinks for vacancies can be dislocations, grain boundaries, surfaces and clusters of vacancies. The original model of single vacancies annealing to a fixed concentration of sinks⁵ is generally not valid. Such a simple model leads to first order annealing kinetics. In practice, a wide range of annealing kinetics, second-order and even more complex

have been observed.⁶ Experimentally, because of all these complexities, enough boundary conditions cannot be imposed to infer an unambiguous value of E_m^1 , E_m^2 and E_p^2 individually.

If anneals were carried out at temperatures high enough to keep diyacancies dissociated into singles, one could then determine E_m^1 by any one of the standard techniques.⁷ Unfortunately, the anneals progress too rapidly by nearly two orders of magnitude to be measured by the standard DC techniques. For example, in gold wires having the same initial defect concentration, half will anneal out in about 12 hours at 60°C. At 400°C, this half time has dropped to about 100 ms. Enrick⁸ has reported preliminary measurements by a high-temperature pulsing technique of E_m for gold. Flynn, Bass and Lazarus⁹ have reported a method for inferring a high temperature value of E_m^1 , but it does not permit a determination of the high temperature annealing kinetics.

This report will describe apparatus and an experimental procedure capable of making high-temperature isothermal anneals of small diameter wires. Preliminary data from experiments performed on gold will also be given.

CONTROL CIRCUIT

Operating Principle

The feasibility of high-temperature anneals was demonstrated by the use of a programed constant current pulsing device.⁸ This device produced only a fair approximation to a rectangular temperature pulse and the temperature reproducibility from pulse to pulse

was very poor. Large correction factors, in which it was necessary to assume E_m , were necessary to reduce the results to those for a single annealing temperature.

Since resistance varies with temperature, it is possible to use the specimen as its own temperature sensing device. Over a reasonably wide temperature range, this variation is linear. The excess resistance introduced by quenching the specimen even from the highest temperatures is negligible (less than 0.01%) compared with the thermal contribution. Thus, the measurement of the specimen resistance is an accurate measure of its own temperature. For example, a temperature of 385°C can be maintained in a .005 in. diameter gold wire in a 30°C oil bath by a current of about 10 A. Using the approximation $R = R_0(1 + \alpha T)$, $\Delta R/\Delta T \approx \alpha R_0$. For gold, α is about $.0035 \text{ } ^\circ\text{C}^{-1}$. A typical specimen has an R_0 of about 0.018Ω . Thus, for the present specimen design, $\Delta R/\Delta T \approx 6.3 \times 10^{-5} \text{ } \Omega/^\circ\text{C}$. For a constant current of 10 A, a change in specimen temperature of $1 \text{ } ^\circ\text{C}$ would cause a change in voltage across the specimen of about 0.6 mV. Millivolt signals are large enough to provide a sufficiently sensitive temperature error signal.

Preliminary Design

The feedback signal must be a measure of the deviation of the specimen resistance from the resistance at the desired operating temperature. The availability of logarithmic circuit elements made it seem that a reasonable approach would be to use a signal $\ln R_s = \ln V_s - \ln I_s$ and compare it with a reference signal $\ln R_0$. This would give an error signal which was non-linear in the temperature error. Exponential converters were then added to give a

specimen current output of

$$I_o = I_o^1 - k[\exp(\log V - \log I) - R_o] = I_o^1 - k(R - R_o)$$

where k is positive and depends on the overall gain of the control circuit. This feedback network produced a considerably better approximation to a square temperature pulse than the programmed constant current device. However, the logarithmic and exponential circuit elements had a long term drift which changed the pulse temperature by as much as 50 C° from one pulse to the next.

Present Design

Originally, a resistance bridge circuit had been rejected because it was desired to measure only the resistance of the central portion of the specimen. The use of stable, linear high-gain operational amplifiers makes a modified resistance bridge measurement possible. The bridge presently used was designed in collaboration with D. E. McDonald, electronics technician supported in part by this contract. A schematic diagram of the circuit is shown in Fig. 1.

Amplifier 1 is operated differentially at unity gain to give an output $Y-X$. (X, Y, Z are the voltages at the points indicated on the figure. Note direction of current flow.) This particular arrangement permits the most stable operation of the amplifier. The feedback resistors are 100 k ten-turn potentiometers. The input to amplifier 4 is therefore $G_V(X-Y) - G_I Z$, where G_V and G_I

Fig. 1. Schematic Diagram of Resistance Bridge

Fig. 2. Schematic Diagram of Current Control Section

are the gains of amplifiers 2 and 3 respectively. The same current flows through both the specimen and R_I , a 10.0000 m Ω , 100 A meter shunt. Thus, the bridge will be in balance when $G_V(X-Y) - G_I Z = 0$ or

$$\frac{X-Y}{Z} = \frac{G_I}{G_V} = \frac{(X-Y)I}{ZI} = \frac{R_s}{R_I}.$$

The gain ratio G_I/G_V therefore determines the operating point of the bridge. The absolute values of G_I and G_V determine the total gain of the section. The total gain of the control section can be adjusted by the feedback resistor on amplifier 4. The four amplifiers were Burr-Brown Research Corporation Model 1506 DC operational amplifiers.

The pulsing current is supplied by two 6V automotive storage batteries. Peak heating currents exceed 50 A, so that batteries are the most convenient source of current.

The current control section (as well as the original logarithmic and exponential converters) was designed and built by Optical Electronics, Inc. The current control (Fig. 2) has a capacity of 100 A, a rated sensitivity of 100 mV input for full 100 A output current and a rise time to 90% of full current of less than 30 μ s. Measurements in the low current (< 10 A) region indicated a sensitivity of 1.0 A/mV and for currents above 10 A of 1.3 A/mV. The current control is biased so that when the bridge is balanced, a current of about 10 A will flow through the specimen. The bias must be adjusted if appreciable changes in

circuit resistance or in specimen operating temperature are made. Otherwise, the bridge will be forced to operate far from its balance point.

The general circuit (Fig. 3) is similar to the original programed current device in that the same SCR's are used to turn the current on and off and monostable multivibrators provide the timing pulses.

Operation Characteristics

When a trigger pulse is applied to the control circuit, it fires both SCR #1 and the monostable multivibrator in the timing circuit. Since the specimen is at bath temperature, the bridge is far from balance and a maximum error signal is applied to the control transistor. The heating current is limited only by the battery voltage and the total circuit resistance (which increases as the specimen heats). During the heating pulse in a .005 in. diameter gold specimen in stirred 30°C oil, the heating rate is $1 - 2 \times 10^5 \text{ }^\circ\text{C}/\text{sec}$. This rate is constant from room temperature to at least 400°C. Thus, the operating temperature is reached in 2 to 3 ms.

When the operating temperature is reached, the controller cuts the current back drastically. Since the surrounding oil is still cool, the specimen temperature does not overshoot but does drop 10 C° or so below the operating temperature during this current cutback. The ensuing oscillations damp out in another 2 to 4 ms and the current and voltage decrease monotonically thereafter. This decrease is due to a gradual warming of the surrounding oil and thus a decreased heat loss rate.

Fig. 3. Schematic Diagram of Pulsing Circuit

A typical plot, taken from a Polaroid oscilloscope trace, of specimen voltage $V_s(t)$, i.e., the voltage X-Y of Fig. 1, and specimen current $I_s(t)$ as measured by the voltage drop across the meter shunt R_I is shown in Fig. 4. From $V_s(t)$ and $I_s(t)$, the specimen temperature, $T_s(t)$, can be calculated. The results of this calculation for the data of Fig. 4 are plotted in Fig. 5.

At the end of a time predetermined by the multivibrator time constant, the multivibrator emits a pulse which fires the second SCR, turning off the current. The stirred oil then rapidly cools the specimen.

Control Accuracy and Reproducibility

The circuit diagram (Fig. 1) shows that the controller senses the voltage across only the central portion of the specimen. This was intended to be the case, for the resistance measurements are made in this region of uniform temperature. However, there is a time lag between the time the controller senses a temperature error and the time the specimen responds. When only the central portion was used, two thirds of the specimen, which also has an active response to changes in current, was not directly controlled by the feedback circuit. Oscillations of large amplitude and 70 kc in specimen current resulted. Surprisingly, however, the envelope of the oscillations had a shape indicating a response to temperature variations.

Earlier measurements had shown that the temperature along the wire was quite uniform except for a few millimeters near the ends. Moreover, the gradients were reproducible.

Fig. 4. Polaroid Traces of Specimen Voltage
and Current, Pulse #5, Run B 7

Fig. 5. Specimen Temperature Calculated from
Data of Fig. 4.

Thus, the controller senses the voltage drop across the specimen current leads outside the bath. Measurements of temperature during pulse and excess resistance measurements are, of course, still made on the central 2 cm of the specimen.

Data for the twelve pulses of run B 7 are superposed on Fig. 6. These data indicate a maximum temperature variation of $\pm 5^{\circ}\text{C}$. Fluctuations of this magnitude are typical. In addition to a real temperature variation, there is an error in the temperature measurement. V_g and I_g are recorded simultaneously by a Tektronix 555 Dual Beam oscilloscope. Typical deflections are 2 to 3 cm. Accuracy of reading the deflection from the Polaroid film is at best $\pm 0.1\text{mm}$. The oscilloscope sensitivity varies along the sweep. After each run, the oscilloscope is calibrated by reference voltages from a Leeds and Northrup K3 potentiometer. All of the measured deflections are corrected by these calibrations. The uncertainty in calibration results in another error of at least $\pm 0.1\text{mm}$. These errors alone can account for a temperature error of $\pm 5^{\circ}\text{C}$. It is therefore assumed that the mean value of all the pulses during a single run is a reasonable value for the temperature.

Occasionally, a change in specimen geometry causes a monotonic drift in temperature. This apparently is a result of a non-uniform cooling rate and the fact that the controller senses the resistance of the entire specimen, not just the central portion. Such a drift disappears after several pulses and can usually be prevented by pulsing the specimen before quenching.

Fig. 6. Superposition of Temperature Profiles
for the Twelve Pulses of Anneal B 7

In summary, the measured relative temperature fluctuations during a series of pulses are about $\pm 7^\circ\text{C}$.

The absolute value of the temperature, which depends on the absolute oscilloscope calibration, the shunt resistor calibration and the degree to which our specimen matches the resistivity vs. temperature scale^{10,11} may be off by $\pm 10^\circ\text{C}$. Since the temperature appears in an E_m calculation as a difference of the inverse temperatures, the resulting value is relatively insensitive to absolute temperature errors.

Pulse temperatures used to date have ranged up to 440°C . Higher temperatures are possible, but at such temperatures the annealing rate would be so high that the pulse duration would have to be less than 30 ms. The 3 ms warmup time would then constitute more than 10% of the total annealing time. Whereas a correction can be made for the warmup period, it would become too large for the desired accuracy at the higher annealing temperatures.

EXPERIMENTAL PROCEDURE

The specimen is given a pre-quench heat treatment which returns the resistance to a stable value.⁹ This treatment consists of a 5-10 min. anneal at 880°C , 15 min. at 550°C and 30 min. at 300°C , all in air. The specimen is then placed in a stirred oil bath controlled to $\pm 0.03^\circ\text{C}$ at about 30°C . The base resistance measurement is then made. The differential measuring bridge and measuring procedure have been described elsewhere.² The specimen is then pulsed several times, as described in the preceeding

section. This does not change the pre-quench resistance base level. In fact, thirty pulses have produced a change which is within the range of thermal fluctuations. The specimen is then removed from the oil bath, quenched and returned to the oil bath, where it remains for the duration of the experiment.

The excess resistance introduced by the quench is measured. The specimen is then pulsed and a resistance measurement made after the pulse. About four of these pulse-measure cycles can be made in an hour. The specimen voltage and current are recorded on Polaroid film for each pulse.

For anneals using the slope-intercept method,⁷ a series of pulses is made at one temperature followed by a series at a second temperature. For anneals using the Palmer method,⁷ the pulses are alternated between two temperatures. (Rather than try to adjust the 10 turn potentiometers for the Palmer method, a series resistance is switched in and out of one of the bridge arms. The temperature is then as reproducible ($\pm 7^\circ \text{C}$) as if a series of pulses were made without touching the potentiometer settings.)

EXPERIMENTAL RESULTS

To date eight runs have been completed with the new control system. The first three used the slope ratio method and the last five were done by the Palmer method. The data were all plotted on rectangular graph paper.

Slope Intercept Results

Fig. 7 shows the results of run B 2. The curve has been visually fitted through the point of intersection and the slope line drawn in visually. A plot of the average slope values between data points (open circles) was used to extrapolate a slope value at the point of intersection. Within the accuracy of the estimate, the two methods give consistent results.

Palmer Method

Fig. 8 shows the results of anneal B 7. Both the resistance remaining after each pulse (closed circles) plotted as a function of time and average slope between pulses (open points) plotted as a function of average resistance between pulses are shown. The high temperature slopes (open triangles) and the low temperature slopes (open circles) were each fitted with a curve. At a given resistance value, two slope values could be read off to determine a slope ratio and thus E_m . In all but one of the anneals the best fit of the slope vs. resistivity data was a straight line (or else parallel curves) so that E_m was independent of the defect concentration over the measured range.

It should be noted that on three occasions, the first pulse did not control properly. An excessively high temperature resulted, annealing out half of the quenched-in excess resistance on the single pulse.

The results of the eight runs are summarized in Table I. T_Q is the temperature from which the specimen was quenched. ΔR_0 is the actual resistance quenched in. $\Delta R_0/R_0$ is the ratio of

Fig. 7. Slope Ratio Method - Annealing Curves

Fig. 8. Palmer Method - Annealing Curves and Slopes

quenched-in resistance to total specimen resistance at 0°C. T_A^1 and T_A^2 are the two annealing temperatures, T_A^1 being the one used in the first portion of the slope ratio method or the first one of the cycle in the Palmer method. E_m is the effective energy of motion inferred from the data.

TABLE I

Run	T_Q (°C)	ΔR_0 ($\mu\Omega\text{cm}$)	$\Delta R/R_0$ %	T_A^1 (°C)	T_A^2 (°C)	E_m^{eff} (eV)
B 1	884	211.4	.745	323	382	.74 ^a .79 ^b
B 2	786	100.1	.352	297	365	.43 ^a .49 ^b
B 3	784	92.8	.326	315	362	.64 ^a .73 ^b
B 4	750	78.1	.275	310	357	.65
B 5	787	97.3	.342	353	400	.67 ^c .4 ^d
B 6	920	120.6	.430	367	410	.60
B 7	920	184.3	.669	344	384	.77
B 8	965	172.1	.627	389	438	.63 ^c .53 ^d

^aFrom visually estimated tangent on annealing curve

^bFrom extrapolated values of plotted slopes

^cFor $\Delta R/\Delta R_0 > 55\%$

^dFor $\Delta R/\Delta R_0 \sim 40\%$

ACCURACY OF RESULTS

The scatter in measuring ΔR , the excess resistance, is $\pm 0.1 \mu\Omega$. This is a result of thermal fluctuations in the bath and change in contact resistance of various switches in the measuring circuit. Under ideal thermal conditions, the bridge is capable of reproducing to $\pm 0.02 \mu\Omega$, so that the thermal fluctuations are the major source of error in ΔR .

The error in temperature has been discussed in the control system section. The variation of $\pm 7^\circ\text{C}$ is a maximum variation when all runs are plotted on the same graph. The Palmer method, in particular, tends to average out these fluctuations, so that the relative temperature fluctuation is closer to $\pm 4^\circ\text{C}$ on the average for all runs.

An absolute temperature error (for no relative temperature error) of as much as 20°C would introduce a 6% error in $(1/T_1 - 1/T_2)$ for $T_1 = 360^\circ\text{C}$ and $T_2 = 400^\circ\text{C}$. The time measurement accuracy depends on the time base generator of the Tektronix 555 oscilloscope. The oscilloscope was recently serviced and calibrated by Tektronix and certified better than the guaranteed 3% accuracy ratings.

Another source of error is the possible background anneal in the bath at 30°C . The rate of anneal near room temperature is strongly affected by both quench temperature and pre-quench heat treatment. The lower the quench temperature and the longer and higher the pre-quench heat treatment, the slower the anneal for a

given annealing temperature. To determine the rate of anneal in the 29.7°C oil bath, a specimen was given the standard pre-quench heat treatment and quenched from about 870°C. During the first four hours of the anneal, 3% of the quenched-in excess resistance annealed out. The time for half the resistance to anneal out was about 76 hours.

The amount of resistance annealing out during the measurement between pulses would be a maximum of 0.5% of the amount annealed during a pulse. Explicitly, therefore, the effect of annealing between pulses is negligible compared with the other errors.

Until a further study of the absolute temperature error is made, it is not possible to quote a figure for the accuracy of the measured slopes. The best statement that can be made is that the results are reproducible to within 15 percent.

In the Palmer method, large amounts annealed out per pulse during the first portion of the anneal. The average slope was taken as the change in resistance $\delta(AR)$ divided by the pulse duration. When $\delta(AR)$ is large, this is a poor approximation. However, as the anneal progresses, the approximation becomes very good. Thus, E_m measured for the high defect concentrations is less certain than that for the low concentrations.

DISCUSSION

Control System

The control system provides an accurate and reproducible rectangular temperature pulse in small diameter wires. The

temperature range is up to 400°C and the time range is down to 30 ms at the maximum temperature. By increasing the heating current (by increasing the battery voltage) along with some circuit modifications, these restrictions could be relaxed by +100°C and -10 ms.

The control system appears to be meeting all of the desired performance characteristics. It can be modified to perform over a wide variety of ranges of specimen resistance (temperature).

E_m^{eff} by slope-ratio Method

Unless the annealing kinetics are known a priori and the curve is fitted to the data points, the slope ratio method tends to weigh the point at which the temperature was changed far more heavily than the other data points. The scatter of values for runs B 1 to B 3 tends to bear this out. The unjustified assumption of first order kinetics by plotting the data on semi-log paper and fitting the points with the best straight line generally gives an E_m value which is too high.⁹ Plotting the average slope between each set of data points and extrapolating to the point of temperature change is a somewhat better way to use all of the data. By this method many anneals are necessary to get a significant average value. The average value of E_m from the three runs is about .6 eV, which is in agreement with the values of the five runs by the Palmer method.

No isothermal anneal was made over a wide resistance range. It is therefore difficult to determine the annealing kinetics. During the first portion of the anneal, which in these runs were

performed at 300 to 320°C, the data are reasonably well fitted by a straight line on semi-log paper. On two of them, however, there is a distinct deviation on the first two or three pulses. During the second portion of the anneal, the kinetics are definitely not first order, although, again, the deviations are in the first few points after the temperature change. These could be transients, but there is insufficient data to be sure.

E_m^{eff} by the Palmer Method

These anneals yield fairly consistent results, with $E_m = 0.66 \pm 0.07$ eV during the portion of the anneal for $\Delta R/\Delta R_0 > 50\%$. The accuracy of the measurements for values below this decreases somewhat. This is in the middle of the range of values for comparable defect concentrations reported by Ytterhus et al.¹² The latter measurements were performed by conventional techniques near room temperature.

CONCLUSIONS

It therefore appears that the pulsing technique, even at temperatures in excess of 400°C, gives effective E_m values directly comparable with values obtained by conventional methods. Without further experimentation, one may only speculate about the explanation for these results.

One possible reason for these results is that the divacancy binding energy in gold is extremely high. Then, even at elevated temperatures the vacancies would spend the majority of their time as divacancies.

There is another, and possibly more likely, explanation. After the initial quench, the divacancy concentration is relatively high. This is due to the fact that association can occur during the finite quench time. During the short temperature pulse, the divacancies would have time to dissociate a relatively small distance. During the 15 or so minutes between pulses when a resistance measurement was being made, reassociation of the recently dissociated divacancies could occur. There is experimental evidence¹² that even more singles could associate during the measuring period.

Since di's have essentially the same resistance as two singles, the reassociation of singles into divacancies would not change the value of the resistance reading. Thus, the negligible net anneal of resistance between pulses does not imply that interactions between the existing defects does not occur. This means that despite the fact that di's begin to break up during a pulse, they could reassociate between pulses. In that event, the vacancies would still spend the major portion of their annealing time as divacancies.

In order to prevent reassociation of vacancies between pulses, the specimen could be cooled to liquid nitrogen or possibly even lower temperatures. The present pulsing circuitry presents no difficulty in controlling the specimen in liquid nitrogen. The only problem might be the failure of the specimen to cool rapidly enough after the pulse. This problem will be investigated in the next phase of the experiment.

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