This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Concentration Dependence of Frenkel Pair

Defect Annealing in Electron Irradiated Ta and Nb*

Sverre Myhra[†] and John W. DeFord

Department of Physics, University of Utah
Salt Lake City, Utah

The present work is an investigation into the dose dependence of the annealing spectra of Ta and Nb from 15 to 285°K after bombardment with 2.2 MeV electrons. Ta and Nb samples were irradiated to different defect concentrations, then simultaneously annealed isochronally from 14.7 up to 285°K. In Ta recovery peaks were observed at 23, 51, 65, 123, and 170°K. The position of the peak near 170°K is dose dependent and shows the characteristics of a second-order reaction. In Nb recovery peaks were observed at 26.5, 58, 95 and 135°K. The peak at 95°K is dose dependent and follows second-order kinetics initially with an apparent shift to higher order toward the end of the stage. A two-interstitial model seems to give the best fit to the data.

* Work supported by AEC under contract No. AT(11-1)-1494.

[†] Based upon the thesis submitted by Sverre Myhra to the Graduate School of the University of Utah in partial fulfillment of the requirements for the Ph.D. degree.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

I. INTRODUCTION

While a great effort has been made over the last fifteen years to understand the effects of electron irradiation on the fcc metals, comparatively little has been done on the bcc metals. The group V metals: V, Nb, and Ta, in particular have been given little consideration. Recent work by G. Burger et al.¹ and by El-Salam and DeFord² have outlined the annealing spectra for Ta and Nb, but have been inconclusive as far as determining the character of the observed substages. Some very recent work by the Stuttgart group³ has provided further information and led to the proposal of a two-interstitial model for Ta. The available information concerning activation energies and order of kinetics for the observed annealing spectra, however, is preliminary or simply nonexistent. Therefore, the present work was undertaken in order to investigate the effect of concentration on the substages from 15 up to 285°K in Nb and Ta.

II. EXPERIMENTAL

A. Cryostat

A cross-sectional view of this assembled cryostat is shown in Fig. 1. The liquid helium vessel, the heat switch, and the sample holder are enclosed by a heat shield soldered to the liquid nitrogen vessel, to reduce liquid helium losses due to thermal radiation. A vacuum of 6×10^{-6} Torr or better was maintained. The heat switch, similar to one described by Magnuson, Palmer, and Koehler,⁴ constitutes a variable heat leak, such that the temperature of the sample holder can be raised without causing a prohibitive helium loss. The sample

holder, shown in Fig. 2, was machined from a solid copper rod and soldered to the bottom of the heat switch. It was designed to provide good thermal contact between the samples and the helium reservoir via the heat switch and, in addition, to form a well-defined beam window. A resistance heater coil was wound around the stem and controlled with a variable output, constant current supply. Electrical leads were brought from the sample holder region to liquid helium temperature, then to liquid nitrogen temperature, and finally through a serum cap seal to a connector plug on top of the cryostat.

B. Sample Preparation and Mounting

Nb and Ta sample material in the form of 0.002 in. diameter wires having a stated nominal purity of 40 parts per million (ppm) were obtained from Materials Research Corporation. The estimate for nominal purity proved to be somewhat optimistic in that the resistance ratios $R_{295^\circ\text{K}}/R_{11^\circ\text{K}}$ for the received sample material were about 15 for both Nb and Ta.

To avoid cold working the samples after they were annealed, potential leads of the same material as the respective sample were spot-welded in place at the start of the preparation process. These leads were spaced about 3/16 in. apart, defining the active sample length.

The samples were then outgassed and annealed for several hours in a 1×10^{-10} Torr vacuum just below the melting points of the samples.

Resistance ratios, $R_{295^\circ\text{K}}/R_{11.5^\circ\text{K}}$, after this procedure and mounting are shown in Table I for the samples used in the present experiment. The improvement is significant for Ta while only slight for Nb.

The procedure for mounting the samples is shown in Fig. 3. Six

samples were mounted in two layers on separate "sandwiches", such that a Nb sample was almost directly above a Ta sample or vice versa along the beam direction. This facilitated the irradiation procedure in that a pair of Nb and Ta samples could be bombarded independently of the two other pairs until the desired dose was achieved. The first step in the mounting procedure for a "sandwich" consisted of laying the three samples out on a 0.001 in. stainless steel foil cut to the shape of the sample block. Two current and two potential leads per sample were then spot-welded to the foil. Subsequently the foil was glued onto a bakelite backing with Eastman 910 glue and left to dry. Strips of foil were then cut away such that one current and two potential leads per sample were electrically insulated while one current lead per sample was left as a common lead. Next, copper leads were soldered to the appropriate pieces of foil and later connected to electrical leads running to the outside of the cryostat. The two "sandwiches" were glued together and slipped in between the two sample blocks. G. E. varnish 7031 was used to improve the thermal contact between the sample package and the sample holder. Finally, the common current leads were soldered directly on to the sample block to further improve the thermal contact.

C. Resistance and Temperature Measurements

The resistance measurements were made with the standard Rubicon six-dial potentiometer arrangement. The six samples were connected to six independent constant current supplies which were regulated to 1×10^{-4} . All data were taken using a 10 mA measuring current.

Temperature was monitored with a platinum resistance thermometer, calibrated by the National Bureau of Standards, embedded in the stem

of the sample holder. The thermometer current, 10 mA, was also provided by a constant current supply. Because of the large thermal inertia of the sample holder and a time dependent temperature lag between the sample holder and the sample package during heating and cooling the platinum resistance thermometer was supplemented with a dummy sample. The dummy sample, calibrated at thermal equilibrium with the platinum resistance thermometer, was used to measure the temperature of the sample package during nonequilibrium conditions such as annealing.

D. Irradiation

The irradiation was done in the University of Utah 2.5 MeV Van de Graaff electron accelerator facility. From a consideration of previous work on threshold displacement energies for W^5 and Mo^6 , it was estimated that the threshold energies for Nb and Ta would be about 40 eV. For Ta this corresponds to a beam energy of roughly 1.5 MeV for head-on electrons. To ensure reasonable damage rates, the samples were bombarded with 2.2 MeV electrons. The beam current, $0.7-1.2 \mu A/cm^2$, caused a heating of the samples of about $2^\circ K$ such that the irradiation was performed at approximately $14^\circ K$.

E. Isochronal Annealing

Isochronal anneals were made from 14.7 to $80.3^\circ K$ in temperature increments that, on the average, were equal to 5% of the annealing temperature. After each anneal, resistance measurements were made at a base temperature of $11.9^\circ K$. The annealing pulse length was 10 minutes and heating and cooling times were generally less than one minute for the last degree Kelvin. Temperature instability ($\Delta T_{\text{Peak-to-Peak}}$) during the annealing pulses was usually less than $0.08^\circ K$. From 82.3 up to

285°K liquid nitrogen was substituted for liquid helium and a new base temperature was established at 78.0°K.

III. DAMAGE PRODUCTION

Damage production is discussed in paper II of this series, "Threshold Displacement Energy Measurements on Ta and Nb".⁷ The results are shown in Table II.

IV. RECOVERY

Curves showing the normalized recovery from 14.7 to 285°K for the five irradiated samples are presented in Fig. 4a - 4e and in derivative form in Fig. 5a - 5e. Due to the limited experimental accuracy of the medium and low dose data and of the liquid nitrogen measurements, the temperature increments were increased in the analysis of this data in order to reduce the experimental scatter. Where the rate of annealing was below the experimental accuracy, the slope has been averaged over large temperature intervals. This is shown by straight horizontal lines in the derivative curves.

The slope diagrams for Ta reveal five substages centered at 23, 51, 65, 123, and 167°K. These are labelled as $II_A(Ta)$, $II_B(Ta)$, $II_C(Ta)$, $III_A(Ta)$, and $III_B(Ta)$, respectively. The peak $II_A(Ta)$ is resolvable only in the high dose sample data while $II_B(Ta)$ and $II_C(Ta)$ are present in the high, medium, and low dose data; $III_A(Ta)$ again is only resolvable for the high dose while $III_B(Ta)$ appears in both high and medium dose. The position of $III_B(Ta)$, furthermore, is strongly dose dependent and the temperature quoted above for this peak is for high dose.

The Nb slope diagrams exhibit four substages centered at 26, 58, 94, and 134°K. These are labelled $II_A(Nb)$, $III_A(Nb)$, $III_B(Nb)$ and $III'_A(Nb)$, respectively. $II_A(Nb)$, $III_A(Nb)$, and $III_B(Nb)$ are all resolvable in both the high and medium dose data, while $III'_A(Nb)$ only can be seen for high dose. The position of $III_B(Nb)$ is dose dependent and the temperature quoted again refers to high dose.

The following peaks show first-order annealing characteristics insofar as their position appears to be dose independent: $II_A(Ta)$, $II_B(Ta)$, $II_C(Ta)$, $II_A(Nb)$, and $III_A(Nb)$. The activation energies can be calculated from the following equation:

$$E = kT_c \left(1 - \frac{\Delta}{2T_c} \right) \left[\log \nu t - \log \left(\exp \frac{E\Delta}{kT_c^2} - 1 \right) \right] \quad (1)$$

where k = Boltzmann's constant, T_c is the temperature at the center of the peak on the derivative plots, ν is the frequency factor, t is the annealing time. The above equation follows from the assumptions that Δ , the temperature interval between successive anneals, is constant over the peak and small compared to the width of the peak, and that t is small enough that $\Delta\rho/\rho$ for any one anneal is much less than 1. $\Delta\rho$ is the amount annealed out in the particular anneal and ρ is the damage remaining at the beginning of the anneal. Under the conditions of the experiment the second term is rather small and we have

$$E \approx kT_c \log \nu t \quad (2)$$

The frequency factor, ν , has been calculated from the expression

$$h\nu = kT \quad (3)$$

where h is Planck's constant and T is the temperature. Activation energies for the peaks listed above together with the percent of damage that has annealed out within each particular substage, are shown in Table III. $III_A(Ta)$ is tentatively identified as a first-order substage but, because it is only resolved in the high dose data and is relatively weak, no further analysis will be attempted for $III_A(Ta)$.

Two peaks, $III_B(Ta)$ and $III_B(Nb)$, show higher order characteristics. An exact second-order kinetics was assumed, and the annealing data was plotted for the high and medium doses on an Arrhenius diagram. The results for Ta high and medium doses are shown in Fig. 6 a-b while Nb high and medium doses are shown in Fig. 7 a-b. The linearity is reasonably good over almost three decades in all cases. Activation energies can readily be calculated from the slopes. Furthermore, the intercept of the curve with the horizontal axis can be used to find an approximate value of ν/ρ_F where ρ_F is the specific resistivity of Frenkel defects. These results are tabulated in Table IV together with T_0 , the temperature corresponding to the intercept, and the percent annealing within the substages. A check on the consistency of assuming second-order kinetics can now be made by solving for the order from the expression for the dose dependent peaks shift. The following expression for the activation energy of a general process obeying any order greater than one can readily be derived along the same lines as for first order.

$$E = kT_c \left(1 - \frac{\Delta}{2T_c}\right) \left[\log \left(\nu t \left(\frac{\rho_c}{\rho_F} \right)^{(\gamma-1)} \right) - \log \left(\exp \frac{E\Delta}{kT_c^2} - 1 \right) \right] \quad (4)$$

where ρ_c is the damage remaining at the beginning of the anneal at T_c ,

γ is the order of the reaction, and all other parameters are as defined in the first-order case above. As before the second term is small and we find:

$$E \approx kT_c \log \left\{ \gamma t \left(\frac{\rho_c}{\rho_F} \right)^{(\gamma-1)} \right\} \quad (5)$$

Considering two similar processes with different defect concentrations, an expression for the temperature shift can be derived using Eq. (5).

$$\Delta T_{12} \approx (\gamma-1) \frac{k}{E} T_{c1} T_{c2} \ln \left(\frac{\Delta \rho_{c2}}{\Delta \rho_{c1}} \right) \quad (6)$$

where ΔT_{12} is the temperature shift and subscripts 1 and 2 refer to the two doses. Experimental values for T_{1c} , T_{2c} , ΔT_{12} , $\Delta \rho_{c2}/\Delta \rho_{c1}$ and calculated values for γ are listed in Table V. Thus substages III_B(Ta) and III_B(Nb) apparently obey second-order kinetics within experimental error. Finally, Eq. (5) with $\gamma = 2$ can be used to recalculate the activation energies. These values are shown in Table IV in brackets.

V. DISCUSSION

A. First-Order Stages in Ta and Nb

Recent work by Burger et al.¹ and by the Stuttgart group,³ already mentioned above, shows annealing substages at 9 and 15°K for Ta and at 15°K for Nb. These substages are not present for the work reported in this article because the irradiation was carried out at approximately 14°K. They will however be taken into account in this discussion.

From the reported characteristics of the low temperature peaks at 9 and 15°K in Ta it is plausible to assign the recovery of an interstitial defect, which we shall label as Type A, to these substages. Doping experiments on Ta^{1,3} show that oxygen in concentration of

1×10^{-3} clearly depresses the peak at 15°K while the annealing in Ta between 20 and 120°K is strongly enhanced. It is proposed that a similar model explains the recovery of Nb for the 15°K substage and the annealing between 20 and 50°K . Thus the substages $\text{II}_\text{A}(\text{Ta})$, $\text{II}_\text{B}(\text{Ta})$, and $\text{II}_\text{C}(\text{Ta})$ are interpreted as showing the release of Type A interstitials from impurity sinks and subsequent annihilation with vacancies. Some retrapping and cluster formation may also take place. For Nb the peak $\text{II}_\text{A}(\text{Nb})$ is given the same interpretation. As supporting evidence it may be noted that these stages are virtually nonexistent in pure Ta³ but become dominant upon doping. Furthermore, all of these stages are much too broad to signify single processes for both Ta and Nb. This is reasonable since one would expect a range of binding energies for different impurities and different interstitial-impurity configurations. Since the rate equations for an impurity release stage are governed by the rate at which interstitials are released from traps, the processes are clearly first order as is observed experimentally.

It remains to be explained why the damage production curves in the present work and those presented in the earlier work by El Salaam and DeFord² show linearity to a fractional defect concentration of roughly 1×10^{-4} , even though the irradiation was performed at a temperature which in both metals was approximately equal to that of a free migration peak. Irradiation at a temperature above a close-pair peak clearly will not interfere with the linearity in the damage production since close-pair annihilation is independent of defect and impurity concentration. Furthermore, if the impurity concentration at all times during the damage production is much larger than the concentration of

free vacancies, the damage production will be approximately linear because the free migration stage will be totally suppressed. (Thus the nonlinearity should be small for the present impure samples.) This interpretation is supported by the fact that doping pure Ta with oxygen to a concentration of 1×10^{-3} depresses the recovery below 20°K from 40 to 5%.³

There are two more first-order peaks to be considered, $III_A(Ta)$ and $III_A(Nb)$. In accordance with a two-interstitial model, $III_A(Ta)$ is tentatively identified as the close-pair recovery peak for a second type of interstitial which we shall call Type B. This peak has been observed in previous experiments^{1,2,3} and shows little dependence on impurity concentration. $III_A(Nb)$ is harder to identify. It appears in the annealing spectrum as a broad substage with possibly multiple superimposed spikes. It could either be the Type B interstitial close-pair recovery substages forming a broad peak, or the Type A interstitials being released from traps. Further studies comparing pure and doped samples are necessary to make a decision.

B. Second Order Stages in Ta and Nb

In Section IV it was shown that substages $III_B(Ta)$ and $III_B(Nb)$ were strongly dose dependent and that $III_B(Ta)$ obeyed second-order kinetics for the whole substage. $III_B(Nb)$ obeys second order for a little more than the lower one-half. Then there is a large high temperature tail that may signify either a competing process or a tendency toward higher orders of kinetics. About twenty percent of the total damage remains in the high dose samples after these second-order peaks. Several mechanisms that could account for these features

were examined. The most plausible model points toward free migration of the Type B interstitial with subsequent interstitial-vacancy annihilation and impurity trapping as the processes giving rise to these substages.

This model was considered in some detail. The processes can be described by a set of differential rate equations⁵ that can readily be combined to give

$$\frac{dv}{dt} = K_i \left[1 - \frac{K_{iT}}{K_i v} \ln \left(\frac{v_0}{v} \right) \right]^2 v^2 \quad (7)$$

where v and T are the concentrations of free vacancies and traps, respectively. v_0 is the initial concentration of free vacancies at the start of the stage. K_i and K_{iT} are the rate constants for free migration resulting in annihilation and trapping, respectively. Eq. (7) can be interpreted as a second-order rate equation with a variable rate constant. Initially, when $v_0 \approx v$, the second term in the brackets is much smaller than one and the kinetics is second order. Then, as v becomes smaller, the second term in the rate constant may become comparable to one and the order will steadily rise above two. This clearly depends on the magnitude of $K_{iT}/K_i \times T/v$ versus $\ln v_0/v$. At the end of the stage, $dv/dt = 0$, so that

$$1 - \frac{K_{iT}}{K_i} \cdot \frac{T}{v_F} \ln \frac{v_0}{v_F} = 0 \quad (8)$$

where v_F is the final concentration of free vacancies. From the annealing curves we may estimate v_0/v_F for Ta and Nb if the simplifying assumption is made that the resistivity contribution of a trapped interstitial is the same as that of a free one.

$$\frac{v_o}{v_F}(\text{Ta}) = 2.25$$

$$\frac{v_o}{v_F}(\text{Nb}) = 1.25$$

Assuming that $K_{IT}/K_i \approx 1$, we can calculate T in terms of v_o for both materials

$$T(\text{Ta}) = 0.55 v_o(\text{Ta})$$

$$T(\text{Nb}) = 3.6 v_o(\text{Nb})$$

Assuming that $\rho_F(\text{Ta}) \approx \rho_F(\text{Nb})$ and that $v_o(\text{Ta}) \approx v_o(\text{Nb})$, one may conclude that the concentration of impurity traps is about seven times greater for Nb than for Ta. Referring back to the resistance ratios for the samples in question, it is striking to note that the residual resistivity is approximately seven times greater for Nb than for Ta ($\rho_{295^\circ\text{K}}(\text{Ta}) \approx \rho_{295^\circ\text{K}}(\text{Nb})$). Using preliminary values⁷ for $\rho_F(\text{Ta})$ and $\rho_F(\text{Nb})$, we can estimate v_o for the Ta and Nb high dose samples

$$v_o(\text{Ta}) = \frac{\Delta\rho_o(\text{Ta high dose})}{\rho_F(\text{Ta})} = 140 \times 10^{-6}$$

$$v_o(\text{Nb}) = \frac{\Delta\rho_o(\text{Nb high dose})}{\rho_F(\text{Nb})} = 175 \times 10^{-6}$$

where $\Delta\rho_o$ is the damage remaining at the start of the Type B interstitial free migration peak. The effective concentrations of impurity traps are then equal to

$$T(\text{Ta}) = 80 \times 10^{-6}$$

$$T(\text{Nb}) = 630 \times 10^{-6}$$

From the resistivity ratio measurements we have for the high dose

samples that

$$\frac{\Delta\rho_o(\text{Ta})}{\rho_o(\text{Ta})} = 0.7$$

$$\frac{\Delta\rho_o(\text{Nb})}{\rho_o(\text{Nb})} = 0.1$$

where ρ_o is the residual preirradiation resistivity at 11.5°K. From these ratios one can readily compute the average specific resistivity due to the impurity sinks, where ρ_{Imp} is the specific resistivity due to impurities since

$$\rho_F v_o = \frac{\Delta\rho_o}{\rho_o} \rho_{\text{Imp}} T$$

The results are that

$$\bar{\rho}_{\text{Imp}}(\text{Ta}) = 10.4 \mu\Omega - \text{cm/at. \%}$$

$$\bar{\rho}_{\text{Imp}}(\text{Nb}) = 10.0 \mu\Omega - \text{cm/at. \%}$$

These values are somewhat higher than typical values noted in the literature (2-3 $\mu\Omega - \text{cm/at. \%}$). The reason could be that only a fraction of the total impurity concentration is effective as sinks.

It should be noted that Nb having the greatest concentration of impurities shows more suppression of the free migration stage, III_B(Nb). This stage also features a high temperature tail indicating that the last one-third of the recovery within this stage obeys an order of kinetics greater than two, in agreement with Eq. (7).

The annealing curve for the Nb high dose sample indicates a

peak at about 134°K. This substage, III_A¹ (Nb), is tentatively identified as Type B interstitial release from impurity sinks. The data, however, is too uncertain to warrant any further discussion of this peak.

Finally we may speculate as to the identity of the two types of interstitials believed to be responsible for the annealing spectra of Ta and Nb. Extensive computer calculations on interstitials and vacancies in α -Fe have been done by R. A. Johnson.⁸ He notes, together with Erginsoy et al.,⁹ that the $\langle 110 \rangle$ -split interstitial has the minimum energy configuration. The $\langle 111 \rangle$ -split interstitial, or crowdion, on the other hand, was found to have a very shallow local minimum energy configuration. Finally, the $\langle 100 \rangle$ -split interstitial was found to be unstable. Migration energies for the $\langle 110 \rangle$ and the $\langle 111 \rangle$ interstitials were estimated to be 0.33 and 0.04 eV, respectively. From Table IV the activation energy for free migration of the Type B interstitial in Ta is 0.32 eV. Furthermore, the energy for free migration of the Type A interstitial at 15°K in Ta may be estimated as about 0.03 eV from simple temperature scaling considerations. The close agreement may be fortuitous since we are comparing two different metals, but it is plausible to adopt the $\langle 111 \rangle$ -split interstitial as Type A and the $\langle 110 \rangle$ -split interstitial as the Type B.

VI. CONCLUSION

a) The annealing spectra for Ta and Nb show impurity dependent substages for Ta in the temperature range 20-120°K and for Nb in the temperature range 20-50°K. The positions of these substages do not depend on defect density. These substages are interpreted as release

of $\langle 111 \rangle$ -split interstitials from traps.

b) A second type of interstitial, tentatively identified as the $\langle 110 \rangle$ -split interstitial, migrates at 95°K in Nb and at 170°K in Ta, and gives rise to substages obeying apparent second-order kinetics. There may be associated close-pair recovery peaks centered at 123°K in Ta and 60°K in Nb. The temperature positions of the free migration stages are dose-dependent.

c) Detailed calculations, based on an interstitial trapping model for Stage III free migration, give at least qualitative agreement with the observed characteristics.

d) There is evidence for the $\langle 110 \rangle$ -split interstitial release from traps in Nb giving rise to a substage at 135°K .

e) The above assignment and the associated activation energies in Ta are consistent with computer calculations of Johnson in Fe.

The proposed model for recovery up to 285°K in electron-irradiated Nb and Ta is summarized in Table VI.

ACKNOWLEDGMENTS

The authors wish to express their gratitude to all members of the Radiation Damage Group at the University of Utah for their help on the project. In particular, they are indebted to Gerald Youngblood for his assistance during the run. In addition, a special thanks goes to Dr. Abraham Sosin for his critical reading of the manuscript and many helpful discussions.

BIBLIOGRAPHY

1. G. Burger, K. Isebeck, R. Kerler, J. Völkl, H. Wenzel, H. H. Kuhlmann, and H. Schultz, Phys. Letters 20, 470 and 472 (1966).
2. Ehab M. Abd El-Salam and J. W. DeFord, Phys. Rev. 161, 600 (1967).
3. H. Hemmerich, D. Meissner, H. Schultz, and F. Walz, Proceedings of Conference on Interstitials and Vacancies at Jülich, Germany, Sept. (1968).
4. G. D. Magnuson, W. Palmer, and J. S. Koehler, Phys. Rev. 109, 1990 (1958).
5. H. H. Neely, D. W. Keefer, and A. Sosin, Phys. Stat. Sol. 28, 675 (1968).
6. P. G. Lucasson and R. M. Walker, Phys. Rev. 127, 485 (1962).
7. G. Youngblood, S. Myhra, and J. W. DeFord (to be published).
8. R. A. Johnson, Phys. Rev. 134, A1329 (1964).
9. C. Erginsoy, G. H. Vineyard, and A. Englert, Phys. Rev. 133, A595 (1964).

TABLE I

TABLE I

Sample Data

Sample	No. of hrs. outgassed	$R_{295^{\circ}\text{K}}/R_{11.5^{\circ}\text{K}}$
Sample VII Ta 12	6	258
Sample IV Ta 13	3	142
Sample VI Ta 14	3.5	206
Sample II Ta 15 ^a	4 < t < 9	174
Sample I Nb 13	3	26.3
Sample III Nb 14	3.5	29.1
Sample V Nb 15 ^a	4 < t < 9	25.6

^a Samples burned out overnight

TABLE II

Damage Rate Data

Sample	Total Damage ($\mu\Omega\text{-cm}$) $\times 10^{-2}$	Damage Rate ($\mu\Omega\text{-cm/elect/cm}^2$) $\times 10^{-26}$
II (Ta)	2.56	
VI (Ta)	8.85	2.95 ± 0.30
VII (Ta)	3.19	
I (Nb)	3.88	3.86 ± 0.40
V (Nb)	12.0	

TABLE III

First Order Substages in Nb and Ta

Peak	$T_c (^{\circ}\text{K})$	$\nu (10^{11} \text{sec}^{-1})$	$E_{\text{Act}} (\text{eV})$	$\Delta\rho_{\text{Peak}}/\Delta\rho_{\text{Tot}} (\%)$
II _A (Ta)	23.0 ± 1.5	4.8	0.065	8.4
II _B (Ta) High Dose	49.0 ± 1.5	10.2	0.145	8.4
II _B (Ta) Med. Dose	49.5 ± 1.5	10.3	0.14	8.7
II _B (Ta) Low Dose	51.5 ± 1.5	10.7	0.14	10.7
II _C (Ta) High Dose	66.0 ± 1.5	13.7	0.19	6.4
II _C (Ta) Med. Dose	66.0 ± 1.5	13.7	0.19	5.8
II _C (Ta) Low Dose	65.0 ± 1.5	13.5	0.19	5.4
II _A (Nb) High Dose	25.5 ± 2.5	5.3	0.075	16.4
II _A (Nb) Med. Dose	29.0 ± 2.5	6.11	0.075	19.7
II _A (Nb) High Dose	59.0 ± 2.5	12.3	0.16	24.1
III _A (Nb) Med. Dose	57.5 ± 2.5	12.0	0.16	27.6

TABLE IV

Arrhenius Plot Analysis of Second-Order Peaks in Ta and Nb

Peak	$E_{\text{Act}}(\text{eV})$	$T_0 (^{\circ}\text{K})$	$\frac{\nu}{\rho_F}(\text{sec}\cdot\mu\Omega\text{-cm})^{-1}$	$\frac{\Delta\rho_{\text{Peak}}(\%)}{\Delta\rho_{\text{Tot}}}$
III _B (Ta) High Dose	0.33 ± 0.02 $[0.32 \pm 0.02]^a$	146	$4.17 \cdot 10^8$	30.8
III _B (Ta) Med. Dose	0.33 ± 0.03 $[0.31 \pm 0.03]^a$	144	$4.17 \cdot 10^8$	34.5
III _B (Nb) High Dose	0.16 ± 0.02 $[0.16 \pm 0.02]^a$	82	$5.28 \cdot 10^7$	13.6
III _B (Nb) Med. Dose	0.18 ± 0.03 $[0.17 \pm 0.03]^a$	80	$5.28 \cdot 10^7$	19.5

^aFrom Eq. (2); other values are derived from the slopes of the lines in Figs. 6 and 7.

TABLE V

Dose Dependence of Second-Order Peaks in Ta and Nb

Substage	T_{1C} ($^{\circ}\text{K}$)	T_{2C} ($^{\circ}\text{K}$)	ΔT_{12} ($^{\circ}\text{K}$)	$\Delta\rho_{c2}$ ($\mu\Omega\text{-cm}$)	$\Delta\rho_{c1}$ ($\mu\Omega\text{-cm}$)	n calculated
III _B (Ta)	174.5	167.0	7.5	$2.90 \cdot 10^{-2}$	$1.10 \cdot 10^{-2}$	2.0 ± 0.15
III _B (Nb)	99.5	94.5	5.0	$1.63 \cdot 10^{-2}$	$0.76 \cdot 10^{-2}$	1.95 ± 0.20

FIGURE CAPTIONS

Fig. 1 Sectional view of cryostat.

- (1) Quick Connections
- (2) Serum Cap Feed-Throughs
- (3) Liquid Nitrogen Can
- (4) Support Tube
- (5) Heat Shield
- (6) Outer Casing
- (7) Liquid Helium Can
- (8) Heat Switch
- (9) Terminal Board
- (10) Entrance Port
- (11) Slit System
- (13) Sample Holder
- (14) Exit Port
- (15) Terminal Board

Fig. 2 The sample holder.

Fig. 3 Sample mounting procedure

Fig. 4 a-c Isochronal annealing curves for Ta.

Fig. 4 d-e Isochronal annealing curves for Nb.

Fig. 5 a-b Slope of isochronal annealing curves for Nb.

Fig. 5 c-e Slope of isochronal annealing curves for Ta.

Fig. 6 a Arrhenius plot for high dose Ta.

Fig. 6 b Arrhenius plot for medium dose Ta.

Fig. 7 a Arrhenius plot for high dose Nb.

Fig. 7 b Arrhenius plot for medium dose Nb.

BOTTOM VIEW

FRONT VIEW

Scale 1:1

DAM. REMAINING (%)