TECHNICAL PROGRESS REPORT
 CONTRACT No. AT(11-1)-1456
 MARCH 1, 1970 THROUGH SEPTEMBER 15, 1970

HIGH TEMPERATURE ANNEALS OF DEFECTS
 QUENCHED IN METALS

MASTER

RECEIVED BY DTIC FEB 1 3 1970

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The topics are described in the order of the 1969 Proposal.

A. High Temperature Anneals

and E. High Temperature Pulsing in Cryogenic Liquids.

No further development of this line of approach was made since it was set aside in last year's proposal in favor of more productive studies.

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

This portion of the project has been completed. Mr. Buescher received his Ph.D. in August 1969 and currently has a post-doctoral position at Rensselaer Polytechnic Institute. The paper resulting from this research has been submitted for publication. A preprint of this paper, C00-1456-12 accompanies this proposal and will serve as a completed report. Briefly, it was found that the motional volume determined during -65°C anneals after quenches from 580°C was $3.0 \pm 0.3 \times 10^{-24} \text{ cm}^3$ and during 0°C anneals after 331°C quenches was $2.8 \pm 0.3 \times 10^{-24} \text{ cm}^3$. The former is attributed to the motion of divacancies. The latter could not unambiguously be assigned to any particular atomic species. The value could be explained either by a large divacancy contribution to self-diffusion or by a temperature dependence of ΔV_m .

C. The Effect of Mechanical Strains on High Temperature Annealing. Personnel have not been available to begin this portion of the proposal.

D. Has been deleted.

E. High Temperature Pulsing in Cryogenic Liquids.

This was discussed under part A.

F. Study of the Effect of Pressure on the Mössbauer Spectrum of $\text{Co}^{57\text{m}}$ in NaCl .

A vapor deposition system has been constructed to prepare specimens with greater activity. The pressure windows absorb about 70% of the incident radiation, so that it has been difficult to attain sufficiently good counting statistics. Too

large an isotope concentration has resulted in the formation of precipitates rather than a homogeneous distribution of the isotope. The specimen preparation apparatus has been completely redesigned and rebuilt with the result that highly improved specimens can be produced. The activity must still be increased somewhat for ideal results, but the major problems have been eliminated. Problems were also encountered with the 400 channel analyser and logic circuitry. Considerable time was spent in trouble-shooting and repair procedures. Data acquisition will be rapid once suitable specimens have been prepared.

G. The Effects of Pressure and of Quench Rate on Quenched-In Resistance.

This portion of the research has been completed for aluminum and gold. It constituted a portion of the Ph.D. thesis of P. B. McArdle, who is now on the faculty at Western Carolina College. The paper has been accepted for publication in the December 15, 1969 issue of Vol. 3 of the *Physical Review*. Preprints of this article have been submitted previously under the number C00-1456-11. Formation volumes were found to be $6.8 \pm 0.4 \text{ cm}^3/\text{mole}$ in gold and $6.38 \pm 0.15 \text{ cm}^3/\text{mole}$ in aluminum. The gold value is in good agreement with previous measurements when these are corrected for the pressure coefficient of resistance. The value in aluminum is the first to be made. Evidence that dislocations are not sinks for the majority of the vacancies during quench was also found.

It has been found that in platinum, vacancy clustering does not occur after quench. As a check of the analysis of the loss mechanism in gold and aluminum, platinum is currently being quenched. Clustering occurs in gold and aluminum after quench, so that it is not unreasonable that it is also an important mechanism during quench. Since clustering is not observed in platinum after quench, it is less likely to be an important mechanism during quench. Specimens have been prepared and preliminary quenches have been made. The pressure

effect is readily observed. However, there are indications that the specimens are being contaminated during a series of quenches. Various procedures are now being tested in an effort to eliminate the impurities.

The platinum wires are being quenched from 1200°C and the controller is keeping the temperature remarkably stable for such high temperatures. The evidence for impurities is a large baseline resistance shift following the post-quench anneal. Various annealing procedures and pressure gases are being tested in order to obtain a reproducible resistance baseline.

H. The Effect of Pressure on Ferroelectrics.

This portion of the research was approved after one of the Mössbauer studies proved to be unfeasible. The material NaNO_3 exhibits anomalous behavior of its dielectric constant ϵ and loss factor $\tan \delta$ at about 3.5 kbar at 22°C. An AC capacitance bridge set-up was available for making such measurements. A special specimen plug for making capacitance measurements at pressure was designed and constructed. Specimen temperature is controlled primarily by a furnace in the pressure vessel and secondarily by an oil bath surrounding the vessel. Temperature control problems and stray capacitance problems have been encountered and solved. Also a modified Sawyer-Tower circuit for measuring polarization P vs. field E hysteresis loops has been built and tested. Specimen preparation techniques have also been developed and found satisfactory. Preliminary measurements have been made on KNO_3 which show the apparatus to be functioning as designed.

PUBLICATIONS

The following publications have resulted from the partial support of this contract;

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

Roy M. Emrick, High Temperature Anneals of Defects Quenched in Gold, Bull. Am. Phys. Soc. 12, 389 (1967).

R. M. Emrick, Pulse Annealing of Gold, Physics Letters 25A, 249 (1967).

Brent J. Buescher and Roy M. Emrick, Motional Volume of Defects Quenched in Aluminum, Bull. Am. Phys. Soc. 13, 418 (1968).

R. M. Emrick and D. E. McDonald, Programmable Temperature Controller for Small Diameter Wires, Review of Scientific Instruments, 40, 82 (1969).

R. M. Emrick and P. B. McArdle, Effect of Pressure on Quenched-In Resistance in Gold and Aluminum, Physical Review, Series 3, December 15, 1969, in press.