FINAL REPORT

for project entitled

ANISOTROPIC DIFFUSION AND ELECTROMIGRATION

under

CH Contract No. AT(11-1) - 3020

Prepared by

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I History of Project Development.

The work of this project began under this contract in 1950. The theme of the work at its inception was to investigate, as systematically as possible, diffusion in non-cubic crystals where the phenomenon is essentially anisotropic. In such systems the diffusion coefficient is a tensor rather than a scalar with two (or possibly three) diagonal terms in the principal axis frame of reference. It was hoped that from the extra information of the different terms at every temperature one could make deductions concerning the nature of the diffusion mechanism(s). It turned out that to some extent this was possible, especially in the first work reported (A 1 - designation refers to the listing in Appendix I of publications generated under this contract), which happened to be on zinc. Later the work on other metals, cadmium, thallium, indium and antimony (A 4 - A 7, A 13) provided data that was in general consistent with the vacancy mechanisms but anisotropy alone could not give an unequivocal answer as to a unique choice of mechanism. In zinc and cadmium also there was some study of grain boundary diffusion (A 3, 4) following the Fisher analysis.

The next step in the development was to turn to the diffusion of impurities in these metals following a program closely analogous to that pursued elsewhere for the noble metal solvents. The results for zinc and cadmium (A 8, 11, 12, 15 and 19) showed that indeed the predictions of the "shielded ion model", so successful for the noble metals, had appreciable validity for the divalent metals also. The similarity broke down when it came to the investigation of impurity enhancement of self-diffusion; it turned out that the alloying of zinc with a percent or so of silver, a slow diffuser, enhanced rather than decreased the self-diffusivity (A 18). This result pointed out the need to bring the Friedel oscillation into consideration for interatomic potentials in non-monovalent metals. As might be expected such

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oscillations became even more important in working with metals of even higher valence such as tin (A 20; App. II C 3). The consequence of bringing such oscillations into the model has been to explain a general trend of the activation energies with valence that runs in the reverse direction from that established with the monovalent matrices, i.e. the solutes with valence less than the solvent tend to diffuse somewhat more rapidly than the solvent atoms, etc. While there are still several systems that would be interesting to investigate, this phase of the work will lapse - at least for the time being.

During the academic year 1954-55 the principal investigator worked as a liaison officer with Office of Naval Research London and in course of travels made contact with the laboratory of W. Seith at Münster who at that time with Wever was developing a quite new insight into the nature of electromigration in metals by the use of surface markers and by systematic studies of simple alloy systems. Shortly thereafter the interest in electromigration which developed from contact with Seith became the dominant theme of the project. The specialization in this area put this laboratory in a sense in a unique position, at least for this country, in carrying out a sustained, basic research program into the nature of self-electromigration in pure metals. Measurements were made on gold^(B1), copper^(B2, B12), silver^(B9, B20, B21), sodium^(B11), platinum^(B4, B8), cobalt^(B10), indium^(B7), zirconium^(B16), uranium^(B17), zinc^(B14), and cadmium^(B22). However, the work of the project has been more than a series of routine measurements on various metals, as the following paragraphs should show.

Up to now the electromigration measurements in this laboratory have all been made by the so-called "vacancy flux method" particularly suitable for self-electromigration. The method consists of observing the dimensional changes of the specimen in rod form through measurement of surface markers and specimen diameter as a function of time. Originally there was considerable

scepticism of the validity of the method of surface scratches. The value of the method for obtaining results of intermediate accuracy now seems justified, particularly if the scratch measurements are coupled with measurements of specimen diameter. The activation energy of the observed motion is always in the range of values appropriate to bulk rather than surface motion. Since the current sweeps around the scratches there is less chance for vacancy pile-up here than at inert markers. The ratio by which the dilatation is divided between radial swelling and longitudinal extension was studied in the case of aluminum^(B5) and shown to vary in an understandable way with the ratio of length to radius.

With the first paper^(B1) from this project on electromigration, a theoretical treatment of the wind effect along ballistic lines was presented and this approach was later modified in a summary report^(B4) given at the 1962 International Conference on Lattice Defects at Kyoto, Japan. The papers have been copiously cited and the nomenclature of the analysis widely followed.

It was with the electromigration of platinum^(B4) that we first became aware of the coexistence of the conjugate phenomenon of thermomigration^(B8) as caused by the symmetrical thermal gradients in the specimen. Later, similar effects were observed in sodium, cobalt, zirconium, and titanium. We were surprised by the size of the Soret coefficients but later developed an approximate theory which explained why, for the transition metals at least^(B13), the action of the charge carriers through the thermoelectric coupling should make an anomalously large contribution in these materials.

The simple theory predicts that the sign of the electromigration should depend on the sign of the charge carriers, negative for electrons and positive for holes. However, the sign of the Hall effect and the sign of electromigration do not show the expected correlation for small values of the

Hall effect. Feeling that the correlation should be reliable for large Hall effect, we chose the metal with the largest normal effect known, cobalt, and found the predicted direction mass motion to the cathode.

In the course of investigating mass motion in zirconium⁽¹⁶⁾ and uranium^(B17), metals with a phase change in the temperature region under study, we observed discontinuities of the marker displacement function at the phase boundaries, as well as overall changes in length. The discontinuities could be explained on the basis of negligible atomic mobility in the low temperature phase. The changes in length were attributed to the fact that dimensional changes at the phase boundaries were constrained to be only longitudinal since radial changes were prevented by the static lower phase. This meant that dimensional changes induced by thermomigration at the center of the specimens were not compensated by the same changes in reverse in the cooler portions. Hence the overall length of the specimens would change. The thermomigration in zirconium also had another interesting feature in that there was no curvature apparent, within the accuracy of the data, of the analogous Arrhenius plot, in contrast with the well-known situation for self-diffusion in this metal. The activation energy observed in thermomigration agreed quite well with that which characterizes the low temperature part of the self-diffusion Arrhenius plot. We were led to hypothesize that a common mechanism to account for both phenomena might be dislocation transport since correlation consideration considerations would make it relatively less effective for straight diffusion than for forced motion.

Studies in electromigration in single crystals of zinc^(B14) and cadmium^(B22) have been performed to determine the anisotropy of the electromigration drive. For both metals the story was much the same. Although diffusivity for each has its largest component for motion along the c-axis, mass flow under the influence of the electric current was greater in the basal

plane. Clearly the driving force of electromigration for the basal plane must exceed that along the c-axis quite appreciably -- in fact by a factor of the order of two. A theoretical understanding of this anisotropy is one of the more challenging problems posed by electromigration studies^(B23). Like the question of the sign of the atom flow, the problem involves, in an intimate way, the metallic Fermi surface and the structure of the Brillouin Zones. Considerable progress has been made in this direction by theoretical studies in another project supported by the National Science Foundation (Grant No. GP-23865).

There is one other very interesting facet of the work on the hexagonal metals which has bearing on the way that vacancies are created and destroyed. With non-cubic metals the studies of vacancy creation with rising temperature, such as the Simmons-Balluffi measurements, can reveal not only the number of vacancies formed per degree rise, but also to what degree the dilatation is produced by expansion along the c-axis or by swelling in the basal plane. Just such measurements have been made by Nowick and Feder⁽¹⁾ on cadmium where they find expansion along the c-axis dominating. They draw the conclusion that the prismoidal dislocations are the principal vacancy source in this metal. This result is also borne out by Alexander's study^(B22) of electromigration in cadmium, only of course in this experiment the creation (and annihilation) of vacancies comes about from the divergence of the vacancy flux rather than adjustment to increasing temperature. Interestingly enough the situation is reversed for zinc where the measurements of Gilder and his students⁽²⁾ have shown that there is practically no vacancy expansion along the c-axis with increasing temperature. This again checks with Routhbort's electromigration work^(B14) which pointed to edge dislocations in the basal plane as the vacancy sources.

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1. A. S. Nowick and R. Feder, Phys. Rev. B 5, 1238 (1972); R. Feder and A. S. Nowick, Phys. Rev. B 5, 1244 (1972)
 2. H. M. Gilder and G. N. Wallmark, Phys. Rev. 182, 771 (1969); "X-ray Thermal Expansion Measurements in Zinc", M.S. Thesis of S. F. Apostolou, RPI (1970).

In addition to the work on self-electromigration there was a strong interest in starting some studies of impurity electromigration. The first effort in this direction attempted to follow radioisotopes at a weld interface in zinc but after several frustrating failures the attempts to secure suitable welds were discontinued. A new and very promising line of investigation was opened up dealing with the heavy interstitials in polyvalent metals, specifically as a starter gold in lead. Because of the high mobility of such a solute the electromigration steady state can be established in a relatively short time after introducing the solute - by plating on one surface for example. In addition to its direct simplicity this technique has the added advantage that the analysis of the steady state profile gives the strength of the driving force for electromigration directly, nearly independent of the diffusivity. As a result the temperature dependence of the driving force shows up clearly and is not obscured by the high temperature dependence of the diffusion coefficient. Most of the thinking has been that the fast motion of monovalent solutes in polyvalent solvents must be by the interstitial mechanism but there have been some suggestions of other possibilities. There is reason to believe that electromigration may prove to be useful probe to distinguish between mechanisms. A preliminary report of this work has been submitted as a contributed paper for the March meeting at San Diego (App II B).

In general the situation presented by the data on the electromigration of the light interstitials (H, Ni, O, and C) in a large number of solvents appears to be more systematic and easier understood in terms of a simple overall picture than that for any other system. The single most puzzling aspect is the existence of an isotope effect for the driving force of hydrogen and deuterium in Fe and Ni, apparently a purely quantum effect. We have recently completed a study of these two isotopes in silver, measuring both electromigration and permeation over a fairly wide range of temperature

and pressure. The dependence of both effects on pressure and temperature were highly consistent with a diffusion-limited process. The permeabilities were in the ratio $N_H/N_D \approx 1.25$ which seemed reasonable but the ratio of the electromigration driving forces was $Z_D/Z_H \approx 2.5$ which was a good deal larger than isotope^{effects} reported earlier. In addition the values of the driving forces themselves were also unexpectedly large, leading to the suggestion that perhaps the values reported earlier for hydrogen in transition metals were for atomic hydrogen rather than protons. The experiments indicate that new theoretical models are needed to account for the unexpected results even qualitatively. The work on hydrogen in silver will be reported at the Annual Meeting of the APS in New York (Bull, APS II 18, 54 (1973)).

There have also been some studies of electromigration in thin gold foils, about 1μ in thickness. Even at high temperatures the marker motions seem to be appreciably higher than in the bulk. Further work is planned in this area with the thought that results may have some bearing on the problems in thin film electromigration. Fortunately the NSF will be providing some support so that it will be possible to continue a modest program in the electromigration area.

II Significance of the Contributions from the Project.

In the field of straight diffusion studies the project has made a significant contribution in emphasizing the role of anisotropic studies in understanding diffusion mechanisms. At a rough guess nearly half the current values for anisotropic diffusion coefficients in pure, non-cubic metals were obtained in this project.

Considerably more unique has been the work on electromigration where this laboratory has been practically alone in this country maintaining a long-term general commitment to the study of this area, comparable to that found in France, Germany, Sweden and Russia. There has been an active

theoretical effort and an attempt to systemize concepts and nomenclature. An experimental technique has been developed and refined; it has been widely applied to some dozen pure metals. Interesting side effects have been explained. In particular some large thermomigration effects have come to light and a qualitative theory proposed for their explanation. The use of electromigration as a basic probe to explore the defect-electron interaction has been emphasized. The basic anisotropy of the driving force in non-cubic single crystals is under investigation. The motion of both "light" and "heavy" interstitials can be fruitfully explored by electromigration; in fact some new and unexpected questions have arisen in connection with forces felt by the hydrogen isotopes in a nearly free-electron environment. Although the main interest of the project has been primarily in bulk phenomena, the effect of dislocations, grain boundaries, and free surfaces are coming into focus where the technological interest is higher. In summary the work of the project has underlined the importance of electromigration both as a probe and a basic study in its own right and, on the applied side, has interfaced with the problem of forced motion in defected regions which is of such importance in establishing device stability.

It is perhaps appropriate to indicate at this point that there has been considerable general interest in this work. The principal investigator gives on the average two or three colloquiums a year on the general area. He has given three invited talks at AIME meetings and addressed two ASM seminars, with the more recent talk coming up last October. There have been three invited addresses to the American Physical Society, the latest being March 1971. There have been three encyclopedia articles. Two review articles have just been finished (App II A). In the planning stage, there may be a story for the American Scientist and eventually a long article for the Solid State Physics series, both on invitation.

III Educational Contributions from the Project.

Over the last twenty-two years many young potential scientists have profited from the educational provision of the Atomic Energy Commission contract support. The area lent itself to a series of stimulating and challenging investigations that could be individually and profitably pursued by second and third year graduate students. The list of those earning advanced degrees is given in Appendix II B and C for Ph.D. and M.S. respectively. Not to be overlooked also are the very great mutual advantages that accrued from the more mature postdoctoral students both from this country and from overseas; they are listed in Appendix III A.

APPENDIX I LIST OF ARTICLES PUBLISHED UNDER USAEC SUPPORT
CONTRACT NO. AT(11-1)-3020
(Abstracts of 10 minute papers excluded)

A. DIFFUSION STUDIES

- A1. Self-Diffusion in Zinc, H. B. Huntington, E. S. Wajda and G. A. Shirn, Acta. Met., 1, 513 (1953).
- A2. A Simple Automatic Counter, E. S. Wajda and G. A. Shirn, Nucleonics, 10, (8) 30 (1952).
- A3. Grain-Boundary Self-Diffusion in Zinc, E. S. Wajda, Acta. Met., 2, 184 (1954).
- A4. Lattice and Grain-Boundary Diffusion in Cadmium, E. S. Wajda, G. A. Shirn and H. B. Huntington, Acta. Met., 3, 39 (1955).
- A5. Self-Diffusion in Thallium, G. A. Shirn, Acta. Met., 3, 87 (1955).
- A6. Calculations of the Entropies of Lattice Defects, H. B. Huntington, G. A. Shirn and E. S. Wajda, Phys. Rev., 99, 1085 (1955).
- A7. Self-Diffusion in Indium, J. E. Dickey, Acta. Met., 7, 350 (1959).
- A8. Anisotropic Diffusion of Silver and Indium Tracers in Zinc Single Crystals, Joseph H. Rosolowski, Phys. Rev., 124, 1828 (1961).
- A9. Self-Diffusion in 50-50 Gold-Cadmium, H. B. Huntington, N. C. Miller and V. Werses, Acta. Met., 9, 749 (1961).
- A10. New Technique for Calculating Correlation for Vacancy Diffusion, H. B. Huntington and P. B. Ghatge, Phys. Rev. Lett., 8, 421 (1962).
- A11. Anisotropic Diffusion of Cadmium and Gold Tracers in Zinc Single Crystals, P. B. Ghatge, Phys. Rev., 131, 174 (1963).
- A12. Screened Model for Impurity Diffusion in Zinc, P. B. Ghatge, Phys. Rev., 133, A1167 (1964).
- A13. Self-Diffusion in Antimony, H. B. Huntington, P. B. Ghatge and J. H. Rosolowski, J. Appl. Phys., 35, 3027 (1964).
- A14. Interstitial Diffusion Mechanism in Rutile, H. B. Huntington and G. A. Sullivan, Phys. Rev. Lett., 14, 177 (1965).
- A15. Diffusion of Copper and Gallium in Single Crystals of Zinc, A. P. Batra and H. B. Huntington, Phys. Rev., 145, 542 (1966).
- A16. Anisotropic Diffusion of Mercury in Zinc, A. P. Batra and H. B. Huntington, Phys. Rev., 154, 569 (1967).
- A17. Atom Movements in Metals, H. B. Huntington, in "Atomic and Electronic Structure of Metals", American Society of Metals Seminar 1967.
- A18. Effect of Silver on Diffusion in Zinc, C. J. Santoro, Phys. Rev., 179, 593 (1969).

- A19. Diffusion of Tin into Zinc, J. Stanley Warford and H. B. Huntington, Phys. Rev., B1, 1867 (1970).
- A20. The Diffusion of Antimony into Single Crystal Tin, F. H. Huang and H. B. Huntington, Scripta Met., 5, 705 (1971).
- A21. Impurity Diffusion in Cadmium, Chih-Wen Mao, Phys. Rev. B5, 4693 (1972).

B. STUDIES IN ELECTROMIGRATION

- B1. Current-Induced Marker Motion in Gold Wires, H. B. Huntington and A. R. Grone, J. Phys. Chem. Solids, 20, 76 (1961).
- B2. Current-Induced Markers in Copper, A. R. Grone, J. Phys. Chem. Solids, 20, 88 (1961).
- B3. Sensitive Pyrometer with High Relative Reproducibility, Theodor Hehenkamp, Rev. Sci. Instr., 33, 229 (1962).
- B4. Electromigration in Metals, H. B. Huntington and S. C. Ho, J. Phys. Soc. Japan, 18, Suppl. II, 202 (1963).
- B5. Current-Induced Mass Transport in Aluminum, R. V. Penney, J. Phys. Chem. Solids, 25, 335 (1964).
- B6. Electromigration in Liquid Metals, Alexander Lodding, pp. 491-496, Electromigration in Solid Metals and Alloys, H. B. Huntington in Encyclopedia of Electrochemistry. Edited by C. A. Hampel, Reinhold Publishing Corp., New York (1964).
- B7. Current-Induced Motion of Lattice Defects in Indium, A. Lodding, J. Phys. Chem. Solids, 26, 143 (1965).
- B8. Thermal Diffusion in Platinum, S. C. Ho, Th. Hehenkamp and H. B. Huntington, J. Phys. Chem. Solids, 26, 251 (1965).
- B9. Electromigration and Void Formation in Silver, P. S. Ho and H. B. Huntington, J. Phys. Chem. Solids, 27, 1319 (1966).
- B10. Electromigration and Soret Effect in Cobalt, P. S. Ho, J. Phys. Chem. Solids, 27, 1331 (1966).
- B11. Electromigration and Thermal Transport in Sodium Metal, G. A. Sullivan, Phys. Rev., 154, 605 (1967).
- B12. Search for Reversal in Copper Electromigration, G. A. Sullivan, J. Phys. Chem. Solids, 28, 347 (1967).
- B13. Driving Forces for Thermal Mass Transport, H. B. Huntington, J. Phys. Chem. Solids, 29, 1641 (1968).
- B14. Electromigration in Zinc Single Crystals, J. L. Routbort, Phys. Rev., 176, 796 (1968).

- B15. Electromigration in Liquid Na-K Alloys, J. C. Jousset and H. B. Huntington, Phys. Stat. Sol., 31, 775 (1969).
- B16. Electromigration and Thermomigration in Zirconium, D. R. Campbell and H. B. Huntington, Phys. Rev., 179, 601 (1969).
- B17. Electromigration and Thermomigration in Gamma-Uranium, J. F. D'Amico and H. B. Huntington, J. Phys. Chem. Solids, 30, 2607 (1969).
- B18. Current Basic Problems in Electromigration in Metals, H. B. Huntington, TMS-AIME, 245, 2971 (1969).
- B19. Dynamic Studies of Vacancy Motion, H. B. Huntington, M. D. Feit and D. Lortz, Crystal Lat. Defects, 1, 193 (1970).
- B20. Electromigration and Associated Void Formation in Silver, H. R. Patil and H. B. Huntington, J. Phys. Chem. Solids, 31, 463 (1970).
- B21. Kinetics of Gas-Bubble Swelling in Silver, H. R. Patil and H. B. Huntington, J. Appl. Phys., 42, 3916 (1971).
- B22. Electromigration Single Crystal Cadmium, W. B. Alexander, Z. f. Naturf., 26a, 18 (1971).
- B23. Anisotropy in Electromigration, H. B. Huntington, W. B. Alexander, M. D. Feit and J. L. Routbort in "Atom Transport in Solids and Liquid Metals". Edited by A. Lodding and T. Lagerwall, Verlag Zeitschrift für Naturforschung, Tübingen (1971).
- B24. Electromigration in Encyclopedia of Chemical Technology Suppl. Vol. 2nd ed. pp. 278-294, J. Wiley and Sons, Inc., New York, (1971).

APPENDIX II LIST OF PAPERS AND WORK IN PROGRESS

A. REVIEW ARTICLES ACCEPTED FOR PUBLICATION

1. Electro- and Thermomigration in Metals, H. B. Huntington, contribution to ASM Seminar on "Diffusion" - Cleveland 1972.
2. "Electromigration in Metals" - contribution to "Diffusion in Solids: Recent Developments", edited by A. S. Nowick and J. J. Burton.

B. CONTRIBUTED PAPERS FOR FORTHCOMING MEETING

1. Electromigration and Permeation of Hydrogen in Silver R. E. Einziger and H. B. Huntington, APS Bull II 18, 94 (1973).
2. Electromigration of Gold in Lead by R. N. Jeffery and H. B. Huntington to be given at the March meeting, 1973 at San Diego.

C. RESEARCH IN PROGRESS

1. Electromigration of Magnesium in Single Crystals by John Wohlgemuth.
2. Anisotropy of Electromigration in Single Crystal Tin by A. Khosla.
3. Diffusion in Tin by F. H. Huang and H. B. Huntington.

APPENDIX III LIST OF INDIVIDUALS PARTICIPATING IN PROJECT EFFORT

A. NAMES OF POSTDOCTORAL STUDENTS AND POSITIONS

Th. Henenkamp	1960-61	Professor at Goettingen, Germany
A. Lodding	1962-63	Professor at Chalmers University, Gothenburg, Sweden
G. A. Sullivan	1964-66	(1) Clevite Technical Center (2) Air Force Institute of Technology (3) Chalmers University of Technology (4) State of Pennsylvania, Harrisburg
N. V. Doan	1966-67	Scientist at Saclay, France
J. C. Jourset	1967-68	Scientist at Fontenay-aux-Roses, France
J. L. Routbort	1966-68	Scientist at Argonne National Laboratory
W. B. Alexander	1965-67	Deceased
R. W. Jeffery	1970-73	

B. NAMES OF THOSE EARNING PH.D. AND PRESENT POSITIONS

<u>Name</u>	<u>Year of Degree</u>	<u>Position</u>
E. S. Wajda	1953	Manager with IBM - Fishkill, N. Y.
G. A. Shirn	1954	Scientist with Sprague Electric - North Adams, Massachusetts
J. E. Dickey	1957	Schenectady, New York
J. H. Rosolowski	1961	Scientist with General Electric Co. Schenectady, New York
P. B. Ghate	1962	Section Chief-Texas Instruments Dallas, Texas
R. V. Penney	1962	Scientist with Ford Motor Company Dearborn, Michigan
P. S. Ho	1964	Sprague Electric Cornell, Postdoctoral, Assistant Pro- fessor, Associate Professor on leave to IBM
A. P. Batra	1966	Research Assistant Professor-University of North Carolina, Chapel Hill, N. C.

<u>Name</u>	<u>Year of Degree</u>	<u>Position</u>
C. J. Santoro	1968	Manager, Motorola Company - Phoenix, Arizona
D. R. Campbell	1967	Scientist with IBM - Yorktown, N. Y.
J. F. D'Amico	1968	Western Electric - Princeton, N. J.
H. R. Patil	1969	Postdoctoral - Cornell Postdoctoral - MIT
C. W. Mao	1971	Oneonta College
F-H Huang	1972	Postdoctoral student - Cornell
R. E. Einziger	1973	

C. NAMES OF THOSE EARNING MS DEGREES AND PRESENT POSITIONS

<u>Name</u>	<u>Year of Degree</u>	<u>Position</u>
A. R. Grone	1960	Physics Chairman - Hudson Valley Community College
V. Nerses	1959	New England Nuclear Company, AEC.
N. C. Miller	1960 Later Ph.D. in NMR research	Sprague Electric Company
J. Stanley Warford	1968	unknown
L. W. Bryant	1971	U. S. Air Force