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Second Semiannual Report

A STUDY OF DEFECT STRUCTURES WITH THE FIELD ION MICROSCOPE

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ABSTRACT

The research effort in field ion microscopy to date is briefly reviewed. Significant advances include: (a) development of considerably improved image resolution through the use of fiber optics, (b) an improved method for indexing field ion micrographs, (c) accurate computer simulation of the image using new criteria, (d) computer simulation of the "appearance" of a screw dislocation intersecting several crystallographic planes on a field ion tip, (e) computer simulation of a variety of dislocation images in FCC crystals, and (f) and analysis of short- and long-range order in Ni-Mo alloys.

Future research proposed is: (a) an extension of computer simulation studies of defects in BCC and HCP crystals, (b) computer simulation of partial dislocations and stacking faults in FCC crystals, (c) combined experimental and computer simulation studies of quenched-in defects in platinum.

I. Introduction

The author feels that the research program undertaken with the present grant from NASA has been very fruitful to date. All of the suggested studies cited in the original proposal have not been attempted nor have all of the ideas expressed there been substantiated. A good portion of the proposed research has, however, either been completed or is underway at present. In addition some new areas have been added that were not originally anticipated.

For the sake of clarity and conciseness this report will be written under the topics suggested by the following four questions:

1. What was originally proposed?
2. What has actually been done?
3. What is the current status of the research program?
4. Where should we now proceed?

II. The Original Proposal

The principal aim of the initial proposal, under which this grant was obtained, was to study defect structures in ion emission images of metals and to investigate the stress distribution in a specimen under imaging conditions. It was suggested that ball models and/or computer models could be used to simulate defects and these compared with experimental images. Various types of point, line, and planar defects could be experimentally obtained by suitable specimen treatment, and these images analyzed in detail.

The stress distribution was to be studied, to the first approximation, by using the Moiré method on two dimensional models with a force distribution corresponding approximately to that encountered in imaging.

In addition, an improved image recording device (a fiber optic screen) was suggested. A liquid-helium-cooled cryostat was to be included in the microscope in this laboratory.

III. The Research to Date

We will not repeat in detail the results given in our first semiannual progress report nor the three publication preprints which have already been forwarded.[1-4] Three further preprints,[5-7] of as yet unreported work, are being submitted along with this report. A general summary of the significance of these results, however, is probably useful.

A. Fiber Optics and Image Recording

A fiber optic window similar to the one originally proposed, but without the curved inside face, was obtained from University of Florida funds after having been deleted from the present grant budget. Because it was made to order it was not delivered until August, 1966. Results obtained with it were very striking (see references 1 and 2) not simply because of the good photographic technique, but because it provided resolution of atoms in areas of the image that was not otherwise obtainable. Thus, information about defects, relative intensity, kinds of atoms imaging, etc. can now be obtained where it could not be previously. This recording technique is now commonly employed here on all experimental images.

B. Computer Simulation of the Image

1. The Perfect Crystal

In order to predict how images of defects should appear, or conversely to analyze those images obtained experimentally, the imaging conditions in a perfect crystal must be understood. A. J. W. Moore[8] had earlier proposed a "shell model" as a criterion for image simulation with a computer, but he has not published further.

The result he obtained were verified in the present program. The "shell" model was tested under varying conditions in an attempt to understand the physical basis for the obviously good qualitative agreement with experiment. It was finally concluded that exact agreement (i.e., image point for image point) between experiment and the computer results was not attainable unless the environment of each surface atom was considered. Using a model based on the number and kind of neighbors (up through sixth) of each surface atom, it was shown that every image point could be simulated, at least over a small region of the surface. The results of this study to date are described in detail in references 1 and 4. This model has been dubbed the "neighbor" model for brevity and to distinguish it from Moore's "shell" model.

2. The Imperfect Crystal

Since the shell model gives good qualitative results and is easily applied, even to an image containing defects, it was used as a test of a theory proposed by Pashley[9] and Ranganathan.[10] This theory proposes criteria for the results of the intersection of any crystallographic plane of a field ion specimen by a dislocation of arbitrary Burgers vector. The component of the Burgers vector normal to the plane in question should give rise to a spiral of some integral number of leaves that can be computed. Application of the "shell" model to the case of a screw dislocation intersecting a larger number of different crystallographic planes has completely verified the hypotheses and expanded their utility. This work is the subject matter of reference 6 enclosed. The computer simulation was then carried further to analyze dislocation configuration of various kinds at a number of different crystallographic orientations in FCC materials. Several experimental images of defects were analyzed in detail with nearly one to one agreement. This work is the subject matter of reference 7, enclosed. It seems quite evident that the long-range effects of dislocations in the image can now be accurately predicted and, conversely, dislocations may be identified from their images.

C. Atomic Order in Ni-Mo Alloys

More by chance than by design, a study of short- and long-range order (SRO and LRO) in Ni-Mo alloys was initiated. Originally, a specimen of Ni - 14 a/o Mo was imaged only to aid the research of the doctoral program of B. G. LeFevre. This dissertation was concerned with the "K-effect" (see reference 7) in the Ni-Mo system. It was hoped that some direct evidence for SRO could be detected. In fact, the alloy yielded extremely interesting results. The development of SRO in the FCC α-phase apparently does not proceed as was thought--by small domains of LRO nucleating and growing--but rather occurs as a homogeneous process. Stoichiometric Ni_4Mo (β) was also studied as a standard by which to recognize order in the α-phase. It produced images in the fully ordered (LRO) state that are among the best so far obtained (see references 1, 2 and 7). Although it was not intended to study this alloy extensively, it has proven so informative that a fairly extensive study has been made.

D. Indexing of Images

In the course of studies on platinum and ordered Ni-Mo alloys it became clear that the orientation relationships of distorted and/or incomplete images were rather difficult to determine. After giving the subject some study, the procedure described in reference 3 was developed and has since been successfully employed in many images. It is a simple and straightforward method, but has not been previously reported and offers considerably more accuracy than other methods in use.

E. The Microscope

The originally intended liquid-helium-cooled microscope was abandoned when it was found possible to use a Joule-Thompson (gaseous-expansion) device capable of attaining 16°K. This device has been incorporated into a stainless-steel microscope body, which was described in reference 1. There is virtually no risk in using liquid-hydrogen cooling with this arrangement, since the hydrogen is in a closed circuit. There are many other conveniences associated with the use of this device, which are now well known. Except for very special purposes, there seems to be no pressing reason to go to any other method for FIM work.

F. Short Course on Field Ion Microscopy

At the beginning of this grant (March 1966) a nine day short course was held under the author's direction here at the University of Florida. Although this did not directly concern the present grant, its effect was indirectly very significant.

Lecturers who attended the course were as follows:

Prof. E. W. Mueller
Dr. S. S. Brenner
Dr. A. J. W. Moore
Dr. B. Ralph
Dr. M. J. Southon
Dr. A. J. Melmed
Dr. S. Ranganathan
Prof. J. Galligan
Dr. D. G. Brandon

In addition to some twenty-five students in attendance from various laboratories throughout the United States, nearly all of the lecturers spent the entire period here. Dr. Moore remained 6 weeks. In general there was a very excellent interchange of ideas and information during the informal laboratories and evening sessions among all participants. It is no exaggeration, I think, to say that the lecturers derived as much benefit from the course as the students. A very direct effect of the discussions among the lecturers was some excellent criticism of the various research efforts in the field as a whole. It is difficult to assess the total tangible influence of this on our own research effort, but it is probably equivalent to a man year of misdirected effort spared. The lectures, including my own, are in the final stages of preparation for publication as an introductory book on field ion microscopy. It is to be published by Plenum Press.

IV. Current and Future Research

Because of the success of the computer simulation technique to date, it is being pushed very hard at present. Specifically, semi-quantitative images of partial dislocations and stacking faults emerging at various crystallographic surfaces in FCC are being studied. A general pattern seems to be emerging, starting from

Pashley's and Ranganathan's original proposals.[9,10] The BCC and HCP structures are also being studied, but this work has just begun.

Ball models are now used only sparingly, as useful means of analyzing perfect crystallographic surfaces--especially high index planes. In a word, ball models cannot compete for efficiency with a computer program properly devised and executed.

The study of long-range order is being pursued both in Ni_4Mo and other alloy systems. Although it was not originally planned to do this, the splendid image quality of Ni_4Mo offers a unique opportunity to make a major contribution to the understanding of atomic order. It also appears that the atomic species may be distinguishable by difference. An interesting result that is already quite apparent, is that the crystal facets which develop on the end form of a FIM specimen are highly sensitive to the crystalline symmetry. That is, in going from disorder to full LRO, the crystal structure goes from a FCC solid solution to a BCT structure. This is accomplished by only a slight contraction (1% in the c direction and 1/2% in the two a directions) relative to the original FCC structure. Yet the indexing of the crystallographic planes is drastically altered. For example, certain planes are present in the BCT that are not in FCC etc . This appears also to be the case for CoPt, for which a good deal of literature has appeared of late (11-15), but it has not been widely recognized as yet. We are reanalyzing the published data on CoPt as well as our own data on Ni-Mo and hope to have a preprint available shortly on the crystallography of both alloys.

We have not done any stress analysis at all to date and it does not appear that we will be able to begin in the immediate future.

Work on secondary defects in platinum has proceeded to the stage where most of the transmission electron microscopy survey has been completed, but no results are available yet with the FIM. After quenching from near the MP and reageing at 300° - 600°C, a wide variety and density of secondary defects is produced in Pt. Before proceeding to the FIM, however, we wish to obtain a reasonable density of defect clusters and to be able to say with some certainty what these are. That is, we wish to make the FIM interpretational problem as straight forward as possible so that the interpretational work [5,6] may be verified. We then hope to extend the analysis to defects that cannot be unambiguously identified with the electron microscope (e.g., small point defect clusters, tetrahedra and dislocation loops).

SUMMARY

The major objectives of the original research proposal have been and are being pursued with good success. The interpretation of defect images in FCC materials is well advanced and is now being thoroughly tested in platinum. The computer simulation program is being expanded to include BCC and HCP materials and stacking faults in FCC crystals.

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