SEMI-ANNUAL STATUS REPORT

For the Period

1 June 1966 to 31 December 1966

DEFECT PRODUCTION IN SINGLE CRYSTALS RESULTING FROM ION BOMBARDMENT

by

Lawrence B. Shaffer

Prepared for

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio

13 January 1967

GRANT NGR 36-019-001

X RAY PHYSICS LABORATORY
Hiram College
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TABLE OF CONTENTS

I. INTRODUCTION AND SUMMARY

Contained in this report is a summary of the progress made during the period of time from June through December, 1966 on work supported through the Lewis Research Center of NASA. This work begun in June, 1966 constitutes an area of surface physics involving a study of the damage produced on the surface of metal single crystals during ion bombardment using x ray methods. The technique to be developed as part of this study will be useful in supporting the investigation of metal single crystals using low-energy electron diffraction methods as well as other areas of investigation where information as to the dislocation density and interstitial content of a surface under ultra high vacuum is useful. Other such areas might be ion implantation studies, rolling contact studies,[1] etc.

Another area of interest is that of applying standard x ray techniques to the determination of certain thermodynamic properties of cesium gas, e.g., isothermal compressibility, and possibly the ion - ion interaction potential. The methods to be used are fairly direct and straightforward,[2,3] however the latter parameter may be quite sensitive to experimental conditions.

The work reviewed above and as outlined in the proposal will be accomplished using two major apparatus facilities. The first facility is a double crystal x ray spectrometer in conjunction with an ultrahigh vacuum (10^{-10}Torr) chamber and ion gun for the ion bombardment damage studies. The second facility is the 4-slit scattering geometry[2,3] with a rotating anode x ray tube and appropriate power supply for the cesium gas studies.

II. SINGLE CRYSTAL DAMAGE STUDIES

Theoretical and Problem Evaluation Studies

Much attention has been given to the design of the experiment and to the necessarily controlled parameters. Two questions of major concern have been: A. What is the sensitivity of the x ray method to detecting small changes in the surface condition due to ion bombardment since the volume of material damaged by the ions is quite small compared to the volume seen by the x rays? and B. What is the necessity for a vacuum environment around the crystal since the x ray penetration depth is such that surface contaminations should not be seen?

Answers to question A have been discussed with many researchers (Univ. of Wisc., Ohio State, MIT, Mellon Institute, ORNL) and no definitive answer has been found. One positive reaction has been that the strain fields introduced by dislocations extend some distance from the dislocation center[4] and hence that dislocations at the surface set up strain fields penetrating into the crystal so that x ray detection is possible.[5,6] However the sensitivity question has not yet been resolved.

A preliminary experiment was to be run at NASA Lewis by J. Ferrante, J. Singer, and L. B. Shaffer in which a crystal would be ion bombarded in the LEED System, then mounted in a standard x ray diffractometer, and the rocking curve obtained for various bombardment times. This experiment may have indicated the sensitivity of the method at least to damage produced by ion bombardment and hence given a partial answer to question A. But whether the damage seen would be due to the ion bombardment induced damage or to surface contamination induced damage would not be determined since the crystal is necessarily not kept in a controlled vacuum environment. However for numerous reasons, but particularly due to relocation of equipment at Lewis, the above experiment has not yet been carried out.

Answers to question B are about as nebulous as those to question A except that surface contamination in electron microscopy due to "dirty" vacuum systems has been a real problem in introducing damage into the sample.[7] Evidence is also available that thin films deposited on single crystal Si wafers significantly strain the wafers as observed by x ray techniques.[8]

Thus although preliminary definitive information as to the above questions has not been obtained, this is, after all, the purpose of the grant; i.e. to investigate the possibility of this technique as an important tool in the study of single crystal surface damage. We have, therefore, forged ahead in the design and development of an appropriate instrument to make the indicated measurements.

It is anticipated that the apparatus as designed will not only allow measurements of the rocking curve widths as proposed but also will allow topographic pictures after the method of Deslattes[9] and possibly some extinction measurements. The sensitivity of the method definitely depends on the amount of damage present in the crystal, e.g. rocking curve methods are not sensitive up to dislocation densities of $10^4/cm^2$.[10] Thus depending on the particular crystal, one method of observing the damage may not be adequate and other methods will need to be investigated. It is anticipated that two kinds of Cu crystals can be analyzed - one with less than 10^3 dislocations/cm^2 and one with 10^6 to 10^8 dislocations/cm^2.

Experimental Procedures and Apparatus Requirements

Vacuum System and Double Crystal Spectrometer

The design of a double crystal x ray spectrometer with enough flexibility for all the vacuum requirements, crystal motion requirements, ion gun and electron gun geometry, etc. is quite a demanding task. Design ideas began with a "vacuum-oriented" system in which both crystals were mounted on precision rotary feedthroughs in the same 18" diameter vacuum chamber. This design has the advantage of future flexibility with the vacuum system and convenient interchange of ion bombardment, electron bombardment, and x ray analysis procedures. However, not only was the placement of the x ray exit and entrance windows a problem so as to have access to all the desired Bragg reflections, but also the requirements on the rotary motion feedthrough devices were quite stringent due to the crystal alignment requirements. The crystal faces must remain parallel to a given (usually vertical) line to within ± 5 seconds and for complete analysis must be rotated through angles as small as 1 second about a vertical (usually) axis.

Two bids were received on the above system each approximating $28,000, with one (Varian) being submitted only after a design contract to consider the precision rotary feedthrough requirements. The receiving of bids has been one of the hindrances to the grant program to date. A bid on a slightly modified system to that above was received from a third company but their bid was quite high due to their limited capability.

The next design idea was an "x ray-oriented" system which features an 8" vacuum chamber, which can be precisely located on a standard double crystal spectrometer for x ray analysis of a crystal mounted inside and then can be attached to a vacuum system through a valve arrangement so that ion bombardment and rough pumping is accomplished through the valves. This design has the advantage that the necessary tilt and translation adjustments on the crystal can be made external to the vacuum chamber by positioning the entire chamber on a precision spindle in air. However, the 8" vacuum chamber is limited to approximately 72 pounds due to load limitations of the precision spindle. Thus the additional vacuum system is necessary and provides space for the large ion pump, roughing pumps, ion gun, partial pressure gauge tube, etc. This means that the ion bombardment takes place with the ion beam traveling through the 2 straight-through valves. Also, the crystal must be rotated 180° to face the ion beam and then returned to the same position under the requirements for crystal location given above for the x ray analysis.

After considering the advantages and disadvantages of the two systems described above a contract was let on 29 December, 1966 to Varian Associates to construct the special chamber, provide a standard 12" vacuum system, and supply the partial pressure analyzer. No decision has been made on the double crystal spectrometer but it most certainly will be similar to one at Ohio State.

X Ray Equipment

In addition to the vacuum system and spectrometer an x ray source, power supply, and detector are required. An x ray power supply has been built at Hiram and is now about 90% complete, lacking only several slow-delivery items. The circuits have all been checked out as far as possible and if all goes well, the 50KV, 200MA power supply, regulated to 0.1% on both voltage and current and interlocked with the x ray tube, should be operational in February. The water-cooled rotating anode x ray tube is in operating condition after being leak checked and pumped down to 2×10^{-6} Torr for the past several months. However this tube will probably not be used with the spectrometer since it is not very easily moved. A new multi-target x ray tube may have to be designed or commercial diffraction tubes (4 are needed) purchased for use with the spectrometer. The rotating anode tube will be used with the 4-slit scattering geometry for the cesium gas scattering to be described below. The power supply will be used for both experimental setups as long as concurrent use is feasible. It may be necessary to acquire another power supply (50KV, 50MA) for use with the spectrometer if machine-time scheduling becomes crowded.

A scintillation detector in conjunction with a 400 channel analyzer will be used to analyze the spectrometer data. Complete rocking curves will be stored in 100 channel wide blocks and intercompared before read-out. Also, the spectral distribution can be easily monitored with the 400 channel analyzer.

A versatile recording system is being set up with a teletypewriter and paper tape punch so that either the 400 channel analyzer or the single channel system used with the 4-slit geometry will generate hard copy or punch paper tape through the teletype. Certain problems of interfacing the teletype with the x ray detector electronics and IBM computors are about solved. This system will be easily converted for use with any automation equipment decided upon to use with the 4-slit geometry or the spectrometer.

Assuming that a decision is made soon on the double crystal spectrometer and that delivery of the vacuum system is in early April, the system should be completely set up and preliminary data available by June, which schedule is in accordance with that proposed.

III. CESIUM GAS SCATTERING STUDIES

Theoretical and Problem Evaluation Studies

To date not much thought has been given to the theoretical problems involved with relating certain thermodynamic properties of gases to their x ray scattering curve. For certain parameters, e.g. isothermal compressibility, density, etc., such relations are available and some experimental technique developed.[2,3] However, in a discussion meeting at NASA Lewis last October, interest was apparent in the possibility of being able to experimentally determine the ion - ion interaction and atom - atom interaction in cesium gas. More work will be done in this area during the next 12 months.

Experimental Procedures and Apparatus Requirements

Since the isothermal compressibility, density, etc. are useful parameters obtained from x ray scattering data, work has begun on the experimental equipment necessary. Actually, most of the equipment needed for these measurements was already available at Hiram due to a previous research grant from Research Corp. Certain modifications were recently made in the 4-slit scattering geometry so now all that is needed is a properly-designed Cs gas sample holder.

Since these parameter measurements depend on knowing the ratio of the power in the beam incident on the sample to that scattered from the sample (so-called "absolute intensity" measurements), some preliminary effort will be necessary to check our beam calibration procedures and to investigate certain problems in these measurements. Work has begun in this direction as the principal investigator is giving an invited review paper on this topic next month in New York City.

IV. CONCLUSIONS, RECOMMENDATIONS, AND PROBLEMS

The initial time period discussed in this report has been a difficult one in that attempting to set up a research group here at Hiram we are not only setting up the equipment and program to do the grant work but also attempting to reorganize and create certain support facilities which are necessary for research but which are not presently available at Hiram. These problems were not unanticipated but attempting to solve them has still involved a certain amount of time. Apparently, these facilities will become available after some more effort and will greatly enhance the NASA-supported research program.

The reorganization of research groups at Lewis directly concerned with the grant work as well as the change in the liaison man at Lewis have all contributed delays and uncertainties into the work schedule. The reorganization may also involve a budget problem since the proposal work was part of an in-house research program at Lewis in which close collaboration with Lewis Research Center was anticipated as well as loan and transfer of certain items of equipment not requested directly in the proposal. Since the interest of the Lewis Research Center has shifted along with the reorganization, certain additional items of equipment may be necessary. Also, certain items were underestimated ($15,000 instead of $28,000 for the vacuum double crystal spectrometer) and omitted, such as the 400 channel analyzer. Thus, in order to maintain the spending rate in accordance with the financial operating plan a separate request will be made to transfer to Hiram in support of the grant certain items of equipment which may be available at Lewis in residual inventory.

V. REFERENCES

1. M. F. Amateau and J. W. Spretnak, "Plastic Deformation in MgO Crystals Subjected to Rolling Contact Stresses," J. Appl. Phys. 34, 2340(1963).

2. W. W. Beeman, et al., "Size of Particles and Lattice Defects," Handbuch der Physik 32, 321(1957).

3. L. B. Shaffer, "Absolute X Ray Scattering Cross Sections of Liquids and Solutions," Ph. D. Thesis (University Microfilms, Ann Arbor, 1964).

4. W. T. Read, Dislocations in Crystals (McGraw-Hill, Inc., New York, 1953) pp. 114.

5. D. W. Pashley, "The Direct Observations of Imperfections in Crystals," Reports on Prog. in Physics 28, 291(1965).

6. E. Menzel, "Nearly Perfect Metal Crystals," Rep. Prog. Phys. 26, 48(1963).

7. L. Thomas, Ph. D. Thesis (Cornell, Dept. of Materials Science, 1966)

8. H. A. Blech and E. S. Meieran, "High Temperature X Ray Topography of Si Wafers Strained by Thin Surface Films," Appl. Phys. Letters 9, 245(1966).

9. R. D. Deslattes, et al., "Observations of Dislocations in ADP: Production of Dislocation Free Crystals," J. Appl. Phys. 37, 541(1966).

10. T. O. Baldwin and F. W. Young, "The Use of Borrman X Ray Topography and Absolute Intensity Measurements for the Characterization of Defects in Nearly Perfect Crystals," Paper A2 presented at Pittsburgh Diffraction Conference, Mellon Institute, November 9-11, 1966.