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AEC Final Report on Contract No. COO-1439-2

RADIATION INTERACTIONS IN SOLIDS:
SURFACE AND HIGH-PRESSURE EFFECTS

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This document constitutes the final report on Contract No. COO-1439-2 entitled, "Radiation Interactions in Solids: Surface and High-Pressure Effects." This contract extends over a three-year period ending December 31, 1967. It is divided into three general areas of interest: the first, "Neutron Diffraction," the second, "Radiation Damage and Interactions at High Pressures," and the third, "Radiation Effects on the Surface Properties of Solids." The report is broken down into sections describing each of these areas. The first, entitled "Neutron Diffraction," was primarily the responsibility of Professor William F. Vogelsang, and the second and third were primarily the responsibility of Professor Charles W. Maynard.

A. NEUTRON DIFFRACTION

This section of the report will cover the work done on the development of a system for the investigation of the structure of solids at high pressures using neutron diffraction techniques.

The motivation for this work is straightforward. The use of neutron diffraction techniques to complement x-ray techniques for the investigation of solids is well-known.¹ With the increasing interest in the investigation of high pressure phases of solids using x-ray techniques,² the use of neutron diffraction is a natural extension. Consequently, after an initial study had been made, it was proposed that two areas of work be entered into: the design and construction of pressure vessels suitable for use in neutron diffraction, and the design and construction of a neutron diffractometer, the initial work to be done on the pressure vessel problem.

Authorization for the diffractometer was received subsequently and the design of the diffractometer was started. The initial operation of the diffractometer as a simple neutron spectrometer took place in 1967. The amount of operating time available was limited by the reactor, both in power level (500 kwh/week) and in reactor operating personnel for extended low power operations. Consequently, operation of the diffractometer has been limited to the spectrometer mode and has been aimed toward measurements designed to confirm the operating parameters of the system, establish operating procedures and

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correction factors and eliminate minor design shortcomings which become apparent during operation. The reactor was shut down early in October, 1967 for modification and refueling for operation at 1 MW steady state.

During this period, minor modification has been made to the diffractometer in light of the experience gained through operation. As of the present time (January, 1968), the reactor has been returned to operation and work has commenced on realignment of the diffractometer. Operation is still limited by a lack of licensed operators, but this should be corrected by early February, 1968.

Pressure Vessel Development

The initial studies performed were aimed toward the development of a pressure vessel suitable for use with the diffractometer. It was felt that the first step should be the development of a vessel adequate for the purpose with subsequent development of more refined vessels to be performed either in parallel with the diffractometer construction or following completion of the diffractometer as time and manpower would allow. The type of vessel is determined by the requirements of a large sample volume ($3/8$ - $1/2$ " diameter 2" length) and a wall relatively transparent to thermal neutrons. The requirement of large size is best fulfilled by a piston and cylinder device. All developments have been based on this arrangement. A number of materials were considered for the vessel. Most of the high strength materials previously used in high pressure work cannot be used in this application, either because of too large a cross-section either scattering or absorption or because of induced radioactivity from neutron absorption. The material chosen for these studies was 7075T6 aluminum. This alloy has a relatively low neutron cross-section, little residual activity and a reasonably high yield strength (73,000 psi). The choice is a natural one and has been used by the MTR group for their vessels.³

The cylinder is of the double-ended type with pistons of crucible AA tool steel. Two types of seals have been developed. The first was of the standard Bridgeman type using "O" rings as the compressible member. Difficulty with air entrapment and lack of ease of fabrication lead to a unique technique involving the use of a crushable insert. The device is shown in Figure 1. The cylinder is bored out to accept the tool steel pistons. An insert of aluminum is machined to the same diameter as the cylinder bore and then counter-bored almost full depth. A $1/32$ " hole is drilled in the blind end and counter-bored to accept a small steel ball. The sample is placed inside the aluminum insert and the insert and fixed piston assembled in the vessel bore as shown in the figure. The pressure transmitting medium, if one is used, is inserted through the hole in the insert, the steel ball put in place and the upper piston inserted. As pressure is applied via a ram on the

PISTON AND CYLINDER PRESSURE VESSEL WITH CRUSHABLE INSERT SEAL

Figure 1

movable piston, the ball is forced into the insert forming a seal, and as the pressure is further increased, the insert itself seals around the pistons. Measurements indicate that pressures in the range of 20-25 kilobars may be obtained. At higher pressures the vessel yield maintaining the pressure at the 20-25 kilobar level. The pressure so generated may be held indefinitely. If a clamp is added to the cell to retain the piston, the device may be removed from the ram for use in the diffractometer. Either type of seal is satisfactory and the devices are considered adequate for the initial work contemplated. Further work will be aimed at multi-section devices to take full advantage of the fixed angle feature of the diffractometer with the object of attaining higher pressures.

Diffractometer Design

The diffractometer design chosen for construction is of the fixed angle type. A review of neutron diffraction systems in use at the time the design was first contemplated showed that almost without exception, the systems in use were of the fixed angle type. In this type, the beam of thermal neutrons is monochromatized using a single crystal and the resulting monochromatic beam is allowed to impinge on the sample under investigation. The sample may be a powder or a single crystal, and the details of the design vary with the nature of the sample (powder or single crystal) and the resolution required and intensity of beam available. This sort of spectrometer is the type described in Bacon and elsewhere. A number of papers have appeared dealing with the analysis of the optics of the system so that its characteristics are fairly well understood.^{4, 5, 6, 7, 8} However, it still does not appear that any procedure exists for predicting both the intensity and resolution to be expected for an arbitrary design.

In considering the design of a diffractometer especially suited for samples under high pressure, the primary complication that enters is the effect of the vessel used to contain the sample. Since neutron diffraction requires, in general, large samples of the types of pressure generating devices that have been developed, only the piston and cylinder device has adequate sample volume. As indicated earlier, a piston and cylinder device suitable for use up to 20 kilobars using aluminum for the cylinder is relatively easy to develop. The thermal neutron cross-section of aluminum is relatively low, and it is a reasonable choice for a pressure vessel. To obtain higher pressures, some material other than aluminum must be used; however, materials with better mechanical properties, e. g., steel, have neutron cross-sections which make them unacceptable. The fixed angle spectrometer offers a possible solution to this problem.

In the fixed angle diffractometer, a powder sample is placed in a collimated thermal neutron beam and observations made at some fixed angle θ of the wavelength distribution of the scattered beam. Peaks in

the distribution will occur at wavelengths where the Bragg condition is satisfied. In the conventional diffractometer, λ is held constant in the Bragg equation while θ is varied, whereas in the fixed angle diffractometer, θ is held constant and λ is varied. A diffractometer of this type was first reported by Buras⁹ and called to our attention by Dr. Brugger at MTR. The energy analysis may be performed in two ways: by a time of flight technique using a chopper or a pulsed fast reactor, or by the use of a single crystal as a wavelength analyzer. A review of several such systems is given in reference 3. The advantage of the fixed angle diffractometer for high pressure work comes about because neutrons are brought into the sample at a fixed angle and only those neutrons diffracted in a particular direction are selected. Thus a vessel may be made with windows of high transmission material backed up by massive support of high strength material. In view of these advantages, the decision was made to build a fixed angle diffractometer with the wavelength analysis being performed by a single crystal. This method of wavelength analysis was chosen since it was felt to be simpler than a chopper-time of flight system. In addition, this system offers more flexibility since it may be easily converted to a fixed wavelength diffractometer for either powder or single crystal samples. The simplicity and flexibility outweigh the potential disadvantages which include distortion of the spectrum by second order diffraction and simultaneous parasitic reflections at the analyzing crystal.

As mentioned earlier, several papers have appeared on the analysis of the fixed wavelength diffractometer. Sailor⁴ calculated the relative luminosity and resolution of a single axis spectrometer. This work was extended by Caglioti^{5, 6, 7} and co-workers in a series of papers covering powder and single crystal diffractometers and comparing their results with measurements made on the ISPRA reactor. Willis⁸ made a similar calculation for a single crystal diffractometer, assuming a trapezoidal response function for the collimators rather than Gaussian as in the work of Caglioti and that of Sailor.

In these calculations, however, both spatial effects and the reflectivity of the monochromatizing crystal have been suppressed, their results yielding only the relative luminosity. Werner and Arrot have studied the spatial problem and shown that it introduces an asymmetry in the reflected beam, thus modifying the single crystal reflectivity as summarized by Bacon.¹ The result is that while the calculations of Caglioti seem to predict relative luminosity and resolution, a systematic method which may be used to calculate the absolute intensity has not been presented.

In the case of the fixed angle diffractometer, the problem is somewhat complicated. While in the fixed wavelength case, conditions at the single crystal (monochromatizer) remain fixed independent of the angle of observation; in the fixed angle case, the conditions at the single crystal (analyzer) are constantly changing so that to analyze

the system, to even obtain relative luminosities, the wavelength dependence of the crystal reflectivity must be known.

Part of Caglioti's model may be directly applied, however. He writes the luminosity or resolution of a fixed wave diffractometer as

$$G = G(\alpha_1, \alpha_2, \alpha_3, \quad a = \frac{d_1 \sin \theta_1}{d_2 \sin \theta_2})$$

where G stands for either the resolution or luminosity function and α_1 is the horizontal angular divergence of the i th collimator, the mosaic spread of the monochromatizing crystal, d_1 the spacing of the planes of the monochromatizing crystal, θ the Bragg angle at the monochromatizing crystal, d_2 the spacing of the planes of the sample, and θ_2 the Bragg angle of the reflection from the sample. The fixed angle diffractometer at a given setting is just the fixed wavelength diffractometer operating in reverse order. Thus the formula for the fixed wavelength diffractometer may be transformed into that appropriate for the fixed angle case by changing the order of the collimators and inverting "a" since the order is now powder sample, single crystal, i. e., let $\alpha_1 \rightarrow \alpha_3$, $\alpha_3 \rightarrow \alpha_1$, $a \rightarrow 1/a$. This form is good to within Caglioti's approximations for predicting changes made at any fixed angle.

The more difficult problem of calculating the crystal reflectivity as a function of wavelength remains. Bacon¹ has summarized the basic calculations for an extended single crystal in reflection. This calculation is not adequate if the effects of finite crystal and collimator size are to be accounted for. To this end, work was initiated on an analysis of the diffractometer trying to include the spatial distribution of the neutron beam. This work, which resulted in a Ph. D. thesis¹¹ of which the abstract is appended, showed that the work of Caglioti incorporated certain approximations. Implicit among the assumptions is that the characteristics of the system may be defined in terms of the mosaic spread of the single crystal (analyzer or monochromatizer). The more detailed analysis shows that their result is obtained only if an effective mosaic spread is assumed which depends not only on the actual mosaic spread of the crystal but the remaining parameters of the system. Only when the effective mosaic spread is independent of the other parameters of the diffractometer are the results of Caglioti obtained. Using this revised procedure, it was possible to predict the observed intensity of the diffracted beam in the ISPRA diffractometer within a factor of two. It is planned to perform experiments on the University of Wisconsin diffractometer to provide additional experimental verification of this analysis when time permits.

The neutron diffractometer is shown in Figure 2. The mechanical design of this diffractometer is straightforward and efforts have been made to keep the design simple but flexible. The central post is anchored to the reactor lab floor and serves as a support for the

GENERAL LAYOUT OF THE NEUTRON DIFFRACTOMETER

Figure 2

sample and pressure-generating apparatus and is the primary reference point for the system. The second collimator and the analyzing crystal table, which serve to determine the scattering angle, move together and pivot about the central post. Scattering angles from -12° to 72° may be obtained.

The shield surrounding the sample consists of a two-inch layer of Pb, a layer of cadmium enclosed in a neutron shield formed from two-inch thick layers of masonite. A large slot is left in the shield for the second collimator to pass through, and that portion of the slot not taken up by the collimator is filled with wedges of shielding material so that the scattering angle may be changed in increments of a few degrees. The interior of the cavity in the shield is 11 inches in diameter and large enough to include a pressure vessel and press. Access to the cavity is through removable plugs in the top and sides of the shield. The analyzing crystal is mounted on the crystal table in line with the second collimator and the unknown sample being investigated. The neutron detector is mounted on an arm pivoting about the crystal. The crystal table and the arm are coupled together with the counter moving at twice the angular rate of the crystal table to maintain the Bragg relationship. Both the counter-arm and the crystal-table are driven by Slo-syn stepping motors, and coupling is obtained by pulsing the crystal-table motor at half the rate of the counter-arm motor. Coleman digitizers connected to the shafts of the stepping motors provide digital readout of the position of both the crystal table and the counter. The neutron detector is a ceramic end-window BF_3 counter and is mounted in a shield of a mixture of paraffin and B_4C on the detector arm. Provision has been made for a third collimator between the analyzing crystal and the detector. The counting cycle is set by a lightly loaded transmission fission counter mounted on the end of the second collimator. The fission counter drives a preset count scaler. The output of the preset count scaler controls a record-printing scaler which registers counts from the BF_3 neutron detector. A signal from the preset count scaler also starts the control system for the Slo-syn stepping motors, thus moving the crystal and detector to the next position following completion of a count cycle. This rather simple version of the type of control circuitry commonly employed on most neutron diffractometers provides semi-automatic operation, yet retains the flexibility to allow conversion to a fully automatic system with complete data readout in a form suitable for computer processing at some later date.

While the diffractometer has been designed specifically for high pressure work in the fixed angle mode, enough flexibility has been retained so that conversion to a conventional fixed wavelength diffractometer is quite simple, and in this mode the diffractometer could easily operate with either a powder or single crystal sample. Further, enough room is available in the cavity in the shield for magnets, etc.

The experimental program completed to date has utilized the diffractometer as a single axis neutron spectrometer. The purpose of those experiments has been to 1) check out the operation of the system, 2) establish the parameters of the system, i. e., crystal mosaic spread, effective resolution, 3) determine the neutron spectrum emerging from the reactor, 4) establish procedures for the elimination of second order contamination by the single crystal, 5) verify the positions of the parasitic reflections and make studies to reduce their effect, and 6) make measurements aimed at finding the optimum amount of gamma shielding which may be inserted in the thermal neutron beam. Of the above, all are essentially complete, however 3) must be repeated with the new core in the reactor since it serves as the basic normalization experiment for subsequent work. In addition, further information is required on item 2) with revised collimators now in use. Item 5) is partially complete in that all parasitic reflections have been identified; however, a study of the correlation between the intensity of parasitic reflections and crystal thickness for thick crystal will be a considerable effort, both analytical and experimental,^{12, 13} and only a few preliminary steps in this direction have been made.

Following completion of the above steps, the diffractometer will be switched to the fixed angle mode and measurements started to establish performance with standard samples, e. g., nickel at atmospheric pressure. High pressure work will then be initiated, first with the standard samples, then with a material which undergoes a known phase change at a reasonably low pressure, i. e., RbCl. Upon completion of this work, it is presently planned to start measurements on ND₄F to verify its high pressure phase.

B. RADIATION DAMAGE AND INTERACTIONS AT HIGH PRESSURES

Since ultrahigh pressures cause lattice constants and in some cases the crystal structure of a material to change, it is to be expected that the effects on material properties of bombardment by high energy radiation would change with pressure. For example, on a crude and qualitative basis, since the hard cores of the atoms are closer together under pressure, the barriers to having an atom knocked from a lattice site through the space between two neighboring atoms should be higher. If this is the case, the average number of atoms knocked from their lattice positions will decrease and thus the damage should be reduced. However, there are a number of other possibilities in this general area other than a simple reduction in the amount of damage resulting from the exposure. For example, if one has a situation in which a phase transformation is favored energetically but the barriers to such a transformation are large, it may be possible for the energy deposited by high-energy radiation to trigger the transformation by driving the system over the barrier which prevents the process from occurring readily. In yet another way, a phase transformation may actually occur in the material, and on releasing the pressure the material reverts to its original phase. In this case, if radiation introduces defects into the lattice, the reverse transformation could be blocked. If this occurred, one would be able to quench in high pressure phases much as we quench in metastable phases by lowering the temperature rapidly. To the knowledge of the present investigators, nothing in this general area has been investigated prior to the work described here. In attempting to do radiation damage under high pressure, it was first necessary to build high-pressure devices called cells or bombs which could be used in the presence of highly energetic radiation (primarily neutrons). This means that the material should not be easily made radioactive. This rules out many of the standard materials used in high pressure apparatus, since the alloying elements occurring in steels often are very bad from the standpoint of their becoming radioactive. Further, many high pressure devices require the use of a working fluid which must be connected to a hydraulic ram by a high pressure tube. It would be difficult to introduce this into a reactor so that the material could be irradiated under high pressure. As a consequence of this complication, it was decided that our initial effort would be to produce a cell which could put a sample under pressure and retain it under pressure over a period of time while being completely disconnected from any external apparatus. It was also decided that it would be desirable to be able to see the sample under pressure and make certain measurements while the pressure was applied. To facilitate this we chose to use a diamond anvil device, and the initial cell was purchased as a standard item from a commercial supplier. This cell was only moderately satisfactory, and it soon became obvious that we could develop a better cell for in-reactor studies. Such a cell was developed and used very satisfactorily. This

cell has been described by Kulcinski, Maynard and Walsh in a recent publication.¹⁴ This cell would be described as a special Bridgman opposed diamond anvil apparatus, and it could generate up to 50 kilobars of static pressure at ambient temperatures. It was quite small and was easily fitted into the reactor environment, occupying only a four cubic inch volume. The small size of the sample that could be accommodated between the diamond anvils made it virtually impossible to carry out regular radiation damage studies with this device. As a result, most of the effort was directed toward phase transformation work since we could remove the sample from the anvil and then carry out x-ray studies on it. A series of investigations on a wide variety of materials was carried out and has been reported by Kulcinski and Maynard.¹⁵ The results of this investigation were generally negative within the range of flux exposures and fission fragment exposures that were used. This was believed to be, at least in part, due to the high stress gradient across the sample produced by the diamond anvil-type device. This would be of no consequence in some types of work, but here the defects introduced by stress gradients could exceed those introduced by the radiation damage. At this point, it became clear that there were two very strong reasons for wishing to go to a hydrostatic pressure device of a more conventional type, even if lower pressures were dictated by such equipment. The hydrostatic pressure would not introduce large numbers of defects from working the sample; and in addition would allow much larger sample volumes, which would in turn allow more conventional measurements such as that of resistivity for deduction of the extent of the radiation damage. There are problems associated with such devices in that they cannot be constructed of steel and therefore one needs to work with aluminum or zirconium. We have chosen to work with aluminum. Some of the harder aluminum alloys are satisfactory for pressures up to the neighborhood of 25 kilobars, and an enormous number of investigations can be undertaken in this pressure range. However, the problem mentioned earlier of not wishing to have high pressure oil lines running into the reactor core region has made it desirable to develop a hydrostatic cell which could be disconnected from the pressure-generating ram and could hold a seal for a period of days to weeks. This has resulted in the design of a cell with a new type of seal mechanism which should be highly useful. A thin-walled cylinder of soft aluminum with a cap having a very small hole in the center is used to contain the sample to be studied. This also will contain the oil used as a pressure-transmitting medium. This cylinder fits immediately within the high pressure cylinder used to contain the overall device. The oil is filled up to the very top and the cap pressed down. Any air and excess oil will be extruded through the small hole in the top. After wiping this clean, a ball bearing is placed on top of the hole. A piston is then lowered onto the cylinder, pressing the first small container, which expands against the wall and driving the ball bearing into the hole sealing the small chamber completely. The piston which comes from above is in turn pressed on by a piston from the hydraulic ram

used to generate the pressure. This is fitted with a threaded device with an appropriate shoulder to allow the device to be tightened and the hydraulic ram to be removed. A schematic figure of this device is shown in the accompanying figure. This cell has been tested repeatedly and has been shown to hold pressure to the order of 15 kilobars for a week without reduction in pressure. The full implementation of this device has not been completed, since we are still working on the best pressure measuring method and developing the manganin wire gauges and their application in this instance. When the performance of the cell has been verified a note will be published, probably in the Reviews of Scientific Instruments, on this particular cell and its unusual seal. We would expect our initial studies with this cell to be a fairly conventional radiation damage study of manganin since, until a full understanding of the effects of radiation and pressure on manganin are obtained, it will not be possible to carry out the necessary pressure calibration and measurements with any degree of competence. This work is currently being pursued with university funds.

C. RADIATION EFFECTS ON THE SURFACE PROPERTIES OF SOLIDS

The effects of radiation on surface properties of solids has been subjected to considerably less investigation than the effects on bulk properties. The present effort has been directed toward a study of surface effects, and the properties chosen for the study are the contact potential and the catalytic activity of a surface. The particular material chosen for this investigation was germanium. This choice was dictated by the ready availability of high quality large crystals. An all-metal, ultrahigh vacuum system using a getter-ion pump, was constructed and used to provide the environment for the studies. A 1-1/8" diameter germanium crystal wafer with (1, 0, 0) orientation was ion-bombarded with 300 eV Argon ions. The Argon ions were chosen as a convenient way of introducing radiation damage into the surface. The bombardment cleaned and damaged the germanium surface. Annealing at 500°C removed the damage. The change in contact potential of the germanium with ion bombardment was measured with respect to a tungsten single crystal of (1, 1, 1) orientation, using the Kelvin vibrating capacitor method for the contact potential difference measurements. The ion bombardment produced an increase in the germanium work function of $0.18 \pm .04$ eV for one crystal and -0.14 ± 0.30 eV for a second crystal. The first crystal was of 25 to 45 ohm centimeter resistivity, and the second possessed a 0.3 to 0.8 ohm centimeter resistivity with unknown impurities. Catalytic activity was measured by determining the percent of conversion for the hydrogenation of ethylene reaction. The measurements were made using a mass spectrometer as the detection

instrument. In the specific case studied, the bombarded catalyst crystals produced 9% conversion while annealed or undamaged crystals produced 12% conversion and the background, that is, with no catalyst in the chamber, resulted in 8% conversion. One would infer from this that the bombardment caused a decrease in catalytic activity which might be assignable to a decrease in the ease of ethylene adsorption. The measurements were made over a wide pressure range from 9×10^{-6} mm of mercury to 2×10^{-4} mm, with the higher conversion resulting at lower pressures. While a thesis resulted from this work, it is doubtful that one can place great reliability on these results, primarily because of the large background conversion which occurs. This led us to the idea that we should reconstruct our vacuum system making it a small glass system and possibly having separate chambers for the getter-ion pump and for the bombardment from that used for making the measurements. Thus the glass could be kept much cleaner, and if the system were considerably smaller in extent the sample would have a greater surface area relative to the chamber, resulting in a lower background. Plans were made for carrying out this investigation; however, due to termination of the contract and lack of suitable personnel, the new system has not been constructed.

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