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STUDIES OF NEUTRON IRRADIATION EFFECTS AT IPNS-REF*

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Neutron irradiation effects studies at the Radiation Effects Facility (REF) at the Intense Pulsed Neutron Source (IPNS) located at Argonne National Laboratory (ANL) are reviewed. A brief history of the development of this user facility is followed by an overview of the scientific program. Experiments unique to a spallation neutron source are covered in more detail. Future direction of research at this facility are suggested.

Introduction

Argonne National Laboratory has been a center for neutron irradiation effects studies for over 20 years. The somewhat simple ambient temperature studies of the 1950's at ANL were followed in the 1960's and 1970's by more sophisticated low-temperature studies in the CP5 reactor. This latter effort was led at ANL by Thomas Blewitt. The CP5 irradiation facilities consisted of two liquid helium cooled irradiation cryostats, one for thermal neutron irradiations and the other with a ^{235}U converter for fast neutron irradiations, ambient temperature irradiation positions including a rabbit tube, and high temperature (up to 700^{o}C) irradiation thimbles. These facilities were designed, constructed and characterized by Blewitt and coworkers.[1,2] The CP5 facilities at ANL along with somewhat more limited facilities at Oak Ridge National Laboratory (ORNL) were for 20 years the mainstay of the U.S. program in basic research on neutron irradiation effects. The low-temperature (4.2 K) fast-neutron irradiation facility in CP5 was the only such facility in this hemisphere and one of only 5 in the world during this 20 year period. The situation today is approximately the same, but at ANL, the CP5 reactor has been replaced by IPNS-REF.

Major areas of research during the days of CP5 were the following: Irradiation hardening in Cu, Cu alloys and Nb single crystals was studied at temperatures between 4.2 and 600 K.[3] Simultaneous measurements of resistivity, lattice parameters and anomolous x-ray transmission in fast-neutron irradiated Cu at T < 50 K yielded information on migrating defect species and annihilation and clustering effects.[4] Dislocation loops of interstitial and vacancy natures were detected by changes in length and resistivity during thermal neutron irradiation of uranium.[5] Low-temperature irradiations of superconductors gave information on the effects of crystal structure and metallurgical variables on various superconducting parameters.[6] The most accurate neutron sputtering yields and spatial distribution of sputtered atoms were determined for single crystals of gold.[7] Simultaneous measurements of changes in length and resistivity during low-temperature irradiations of Cu and Ni have revealed aspects of the structure of defect cascades.[8] Measurements of disordering in ordered alloys by magnetization, resitivity, and transmission electron microscopy (TEM) have revealed fundamental mechanisms of neutron damage production.[9] Finally, low-temperature defect production measured by resistivity techniques, along with a state-of-the-art determination of neutron flux and spectrum, has produced the first meaningful comparison with similar data from other reactors, ion accelerators, and fission fragment experiments.[2]

These one-sentence descriptions of some past research at CP5 will give, perhaps, a perspective on any comparison of the usefulness of fission and spallation neutron sources for this area of research.

The Radiation Effects Facility at IPNS

For several years before and immediately following the close-down of CP5 in 1979, the Blewitt group, including R. Birtcher, B. Brown, M. Kirk, B. Loomis and T. Scott, prepared designs, calculations and mock-up experiments[10,11] for the IPNS-REF. Construction took place in 1980-81 and the first experiments at 4.2 K were performed in January, 1982. The facility is operated by ANL as a national user facility for both an in-house irradiation effects program and outside users. A Program Committee reviews all research proposals. The instrument scientist with major responsibility of facility management for users is R. C. Birtcher. About 50% of research at the REF is performed by outside users (non ANL).

The facility itself consists of two irradiation cryostats (5 cm inner dia.) with controllable temperatures between 4.2 K and about 400 C and neutron fluxes of about 1 x 10^{12} n/cm^2 sec ($E_n > 0.1$ MeV, synchrotron current of about 10 μA delivered on a ^{238}U target), and a smaller ambient temperature irradiation position. These irradiation facilities are located adjacent to a separate irradiation effects target and surrounded by lead to produce a fast neutron flux without significant moderation. Figure 1 displays the resultant neutron energy spectrum and compares it with pure and degraded fission spectra. A close similarity between degraded fission and spallation can be noted, with the exception of the high energy "tail" from spallation which accounts for about 3% of the neutron flux and about 15% of the damage in a typical material. These neutron fluxes, energy spectra and flux gradients have been measured to the highest accuracy achievable today (about 10% in the flux defined for neutron energies > 0.1 MeV). A more approximate measure of secondary proton fluxes and energy spectra have been made, and an upper limit on the gamma flux has been determined. More details of these measurements and the facility can be found in references 12-14.

It is important at this point to emphasize those features that are unique to the accelerator based spallation neutron source. The most important for research to date has been the low gamma flux relative to a typical fission source. We have measured the gamma flux to be at least one order of magnitude lower than a fission source with the same neutron flux. Of next importance has been the ability, through the accelerator controls, to turn the neutron flux on or off, to precisely control the total neutron dose (especially very low doses) and the neutron dose rate. Of more minor importance to present research, but of possible future importance, is the high energy "tail" in the neutron spectrum and the pulsed nature of the neutron flux (100 n sec pulses at 1, 5, 10, 15 or 30 Hz repetition rates).

The low gamma flux (especially from a non fissioning spallation target) makes the consideration of a "next generation" REF with the capability of low temperature irradiations in a neutron flux of 10^{14} - 10^{15} n/cm^2 sec very attractive. This would be

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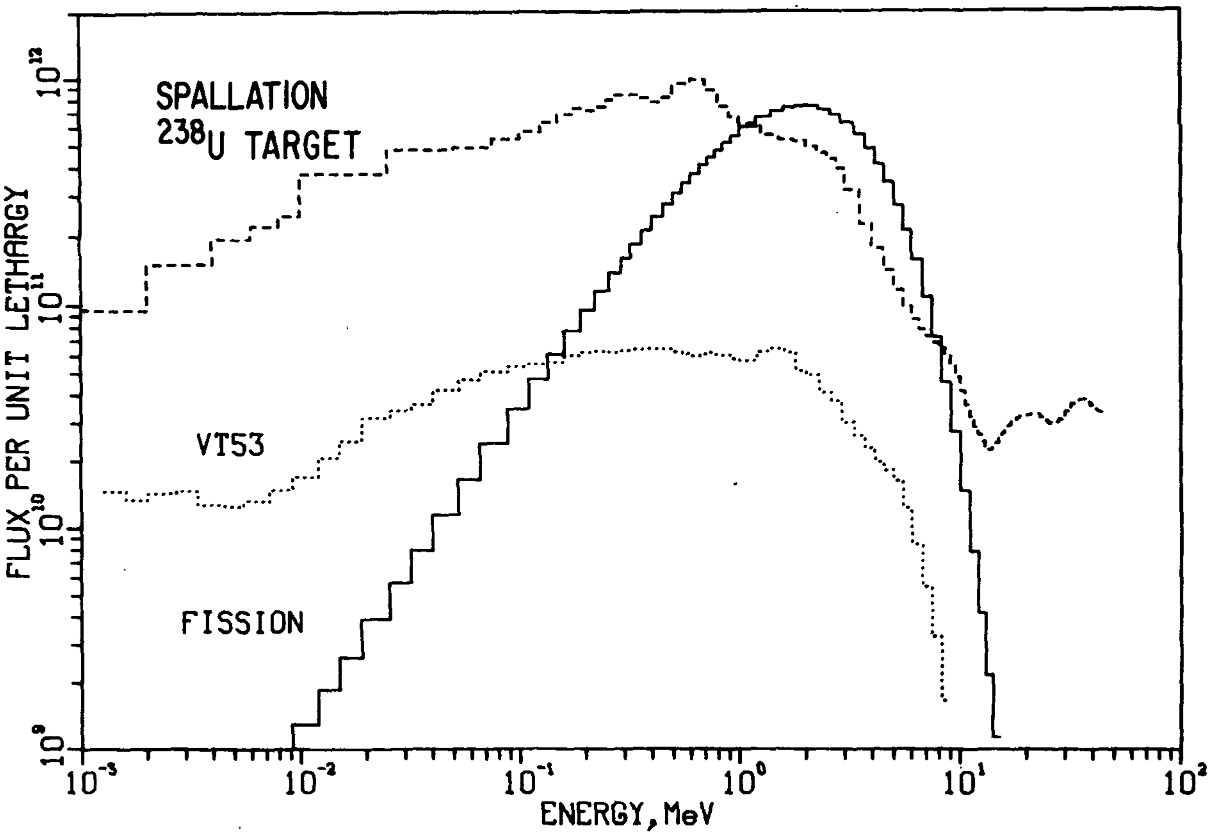


Figure 1. Comparison of neutron spectra (with arbitrary normalization) from pure fission, degraded fission (VT53 facility in CP5 reactor), and spallation from ^{238}U (500 MeV proton energy).

impossible in a fission reactor due to gamma heating. It is for this reason that we consider the REF at IPNS to be an experiment in itself. It is the first and only REF at a spallation source. It is currently the only low-temperature neutron irradiation facility in the USA. It is the only national user facility in this field in the world.

Coming from our long experience with a fission source, we should discuss briefly those features of an accelerator based spallation source that we have found to be disadvantages. We expected and have measured rather severe flux gradients in irradiation positions near the target. This can impose some restrictions, but to now they have been only a minor constraint. Irradiation rig design, sample design and placement, and sample rotation have all been used to minimize the effects of these flux gradients. Inherent in accelerator systems and extensive safety interlocking has been rather numerous beam shutdowns during daily operation. This is of little or no consequence to either low-temperature irradiations or many sample irradiations at ambient temperature. However data can be affected in those in situ experiments at controlled temperatures when defects are mobile. Temperature control itself can be affected, and equilibrium defect concentrations temporarily changed.

Finally, the use of a dedicated target for these studies has both advantages and disadvantages. Total control of the accelerator for precise on, off, dose and dose rate values has tremendous advantages for some in situ experiments. However, the irradiation effects target is programmed to receive the proton beam only 1/4 of total IPNS running time, placing a severe restriction on the length (dose) and number of experiments that can be done. A limited number of small samples can be irradiated at ambient temperature near the neutron scattering target. The flux is lower and the energy spectrum is moderated somewhat.

Overview of Scientific Program

The Radiation Effects Facility at IPNS was designed and constructed (with support from the Basic Energy Sciences Division of the U. S. Department of Energy) to be a basic research tool in this field. In most instances research performed here is designed to contribute to a fundamental understanding of irradiation effects in metals, semiconductors, superconductors, and insulators in support of both the fission and fusion energy programs. The ANL program is designed to investigate the production by neutron irradiation of point defects and defect cascades, defect migration, annihilation, clustering, and interaction of point defects with other defects such as impurities and dislocations. Electron, proton, ion and gamma irradiations are also performed to complement neutron irradiation data in several experiments. The influence of these various defect states upon the physical properties of materials is examined.

The primary emphasis of the ANL program is the use of the unique aspects of this pulsed spallation neutron source. Thus major experimental efforts are in progress studying neutron irradiation effects in organic insulators, semiconductors, and ordered alloys. Irradiation effects due to a gamma flux can be quite important in both organic insulators and semiconductors. This facility is unique in that fast-neutron irradiation effects can be independently studied in these materials. Precise control of dose and dose rate has been essential in the semiconductor and ordered alloy work. It is believed that in the near future the pulsed nature of the source will be utilized in these same two areas of experiment. A more detailed discussion of these unique experimental areas is postponed until the following section while the balance of the experimental program is briefly reviewed.

Spallation and 14 MeV Neutron Irradiation of Stabilized NbTi Superconductors[15]

Experiments are being performed on the irradiation behavior of NbTi, a leading candidate for the superconducting magnets in a fusion energy device. The effect of cyclic neutron irradiation at 5 K and annealing to 300 K is being investigated for various metallurgical states of NbTi. Critical current density (j_c) is measured as a function of applied field up to 9 T at 4.2 K after each cycle of irradiation and annealing. Generally, small decreases have been measured in j_c after 2 cycles and the smallest decrease is for materials with a high degree of final cold work and optimized initial annealing temperature (380 C).

Observations in an HVEM of in situ Tensile Deformation and Fracture in Neutron-Irradiated Niobium[16]

High-purity and O-doped (200 appm) Nb single-crystal tensile specimens were irradiated in IPNS-REF at 300 K. Observations of the in situ tensile deformation in the ANL-High Voltage Electron Microscope (HVEM) have shown the deformation processes that give rise to irradiation induced embrittlement and/or deformation. This process consists of dislocations gliding in channels which are thus cleared of the defect clusters produced by the neutron irradiations. Video tape recordings of details of the process have been made with the HVEM.

Non-ANL User Experiments

Both Los Alamos National Laboratory (LANL) and Oak Ridge National Laboratory (ORNL) has been major outside users of IPNS-REF in programs to study neutron irradiation effects in fusion energy magnet materials. The LANL experiments on organic insulators will be discussed in the next section. The ORNL experiment (R. Coltman and C. Klabunde) is designed to study changes in magnetoresistivity of copper at 5 K under cyclic neutron irradiation and annealing. It now appears that the copper stabilizer in the superconducting magnets for a fusion reactor may be the most irradiation sensitive component.[17] Initial data has been taken on the increase of resistivity and magnetoresistivity in copper after 3 irradiation and anneal cycles. Analysis of results is underway. These results coordinate nicely with the NbTi work described above. To perform these experiments the ORNL group constructed an irradiation rig capable of in situ measurements in applied magnetic fields up to 7 T.

Other experiments in progress include: continuation of the semiconductor work (see next section on silicon) by the University of Missouri group (J. Farmer and J. Meese with R. Birtcher, ANL) with measurements of Deep Level Transient Spectroscopy in irradiated germanium; a study of neutron irradiation effects in the amorphous metal superconductor, Mo-Ru-B at 4.2 K with in situ magnetic fields to 7 T (R. Scanlan, Lawrence Berkeley Laboratory); a study of fatigue in neuron irradiated copper single crystal (D. Luzzi and M. Meshii, Northwestern University); diffuse x-ray scattering studies of neutron irradiated nickel, copper, and their dilute alloys and Si and Be (R. Averback, ANL and P. Ehrhart, KFA, Jülich, West Germany); and a study of the effects of solutes on neutron damage production and recovery in zirconium (S. MacEwen and R. Zee, Chalk River Nuclear Laboratory, with R. Birtcher, ANL).

Experiments Unique to a Spallation Neutron Source

Neutron Irradiation Effects in Organic Insulators

Two sets of experiments have been taking place at IPNS-REF in this field. Work at ANL in collaboration with the Japan Atomic Energy Research Institute (S. Egusa, visiting scientist at ANL) is in progress to investigate changes in mechanical properties of composite organic insulators following fast neutron and gamma irradiations at 5 K and 300 K. Mechanical and electrical property changes were measured in the composite G-10CR by a LANL group (G. Hurley, J. Fowler and D. Rohr) following 5 K irradiation at IPNS-REF. Both programs will be able to isolate those irradiation effects due only to neutrons; a result of the low gamma flux at IPNS. The interest in these materials for use as magnet materials in fusion energy devices is high.

The LANL results to date show small but significant changes in mechanical and electrical properties of G-10CR irradiated at 5 K with neutrons to 2.5 MGy.[18] Flexural strength was reduced and fracture surfaces show dependence on temperature and irradiation. Both DC and AC resistivities decreased slightly with this irradiation dose, but dielectric breakdown strength showed no significant change.

Four kinds of cloth-filled organic composites were irradiated in IPNS-REF at 300 K and 5 K and mechanical property changes were studied at ANL.[19] The same study was also made for composites irradiated with 2 MeV electrons at 300 K for comparison.[20] Gamma irradiations are currently taking place at ANL on these materials. The neutron results to date show the shear modulus and ultimate strength unchanged in all samples after 400 Mrad. However the fracture propagation energy in the glass fiber/polyimide matrix is seen to change with neutron irradiation at a much higher rate than with electron irradiation.

Fast Neutron Irradiation Effects in Semiconductors

Knowledge of the defect structure in semiconductors following fast neutron irradiation has been severely limited by the concurrent production of defects or charge carriers due to high gamma fluxes in fission reactors. The low gamma flux at IPNS-REF allows, for the first time, meaningful measurements of DC conductivity, annealing, and Deep Level Transient Spectroscopy (DLTS) during and following low-temperature fast-neutron irradiations. These measurements have yielded information on damage production, thermal recovery and defect identification.[21]

Resistivity measurements on several semiconductors during irradiation at 5 K are displayed in Fig. 2. The somewhat surprising behavior illustrated in this figure can be well described by the exponential relation:

$$\sigma(\phi) = \sigma_o e^{-K\phi}$$

where σ is conductivity, ϕ is fluence (in protons on spallation target in Fig. 2) and K is a damage constant dependent on material. This form of relation has been observed in metals and when analyzed in a similar fashion here yields information on defect cascades. In silicon the average cascade volume was determined to be 4.7 x 10^5 atom volumes with a defect density in this volume of 5.5 x 10^{-4}.

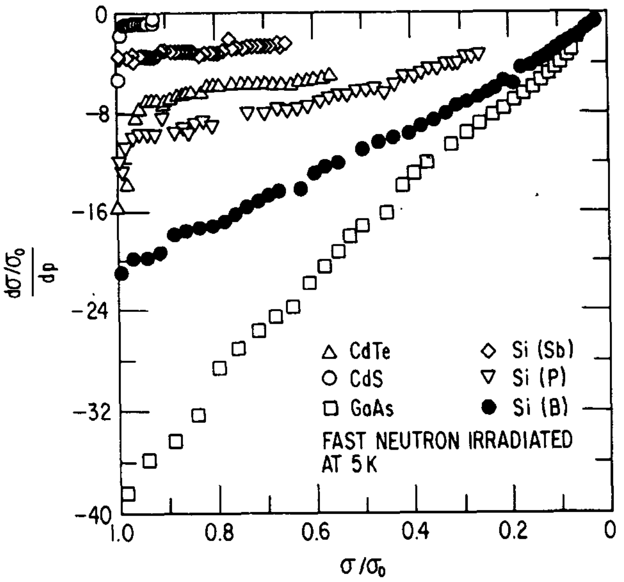


Figure 2. The normalized rate of conductivity change, $d(\sigma/\sigma_0)/dp$, vs. fractional normalized conductivity, σ/σ_0.

The annealing results in silicon yielded a very low temperature process at 22 K which has not been observed before. Results in CdTe indicated the vacancy may be mobile below room temperature which, if true, would be the only known vacancy in a II-VI semiconductor to possess this property.

DLTS has been used to determine the defect energy levels introduced into the band gap of n-type silicon following fast-neutron irradiation at 5 K.[22] The thermal production and annihilation of these defect energy levels during annealing is shown in Fig. 3. The increase of the defect level E_1 (known to be the vacancy-oxygen A center) occurs at the expense of a defect with a deeper electronic energy level whose concentration can only be inferred by the behavior of the low-temperature shoulder of its DLTS signal. This annealing is consistent with the single vacancy which is known to thermally migrate below 150 K. Decreases in the E_1 and E_3 (divacancy) levels between 250 and 300 K may be a result of the appearance of the E_0 level which suggests that this level may be associated with an interstitial type defect. Above 350 K the divacancy level E_3 anneals out while E_1 and E_2 grow. E_2 may be associated with larger vacancy clusters. These results suggest, in agreement with conductivity measurements, that the fast-neutron defect cascade in silicon is very diffuse and at 5 K contains primarily single vacancies and divacancies.

Fast Neutron Irradiations of Cu_3Au

Ordered alloys have been useful to study irradiations effects from the earliest days of nuclear reactors. The formation of displacements or defects usually causes a larger number of replacements during the process. These replacements result in the disorder of ordered alloys which can be measured in several different ways. Also, the migration of a defect, say a vacancy, can cause the ordering of many atoms and thus reveal itself to measurement more easily. We have been active for many years at ANL in the use of ordered alloys to reveal fundamental mechanisms of neutron irradiation effects.[9] Recent experiments on Cu_3Au reveal or take advantage of unique features of IPNS-REF.

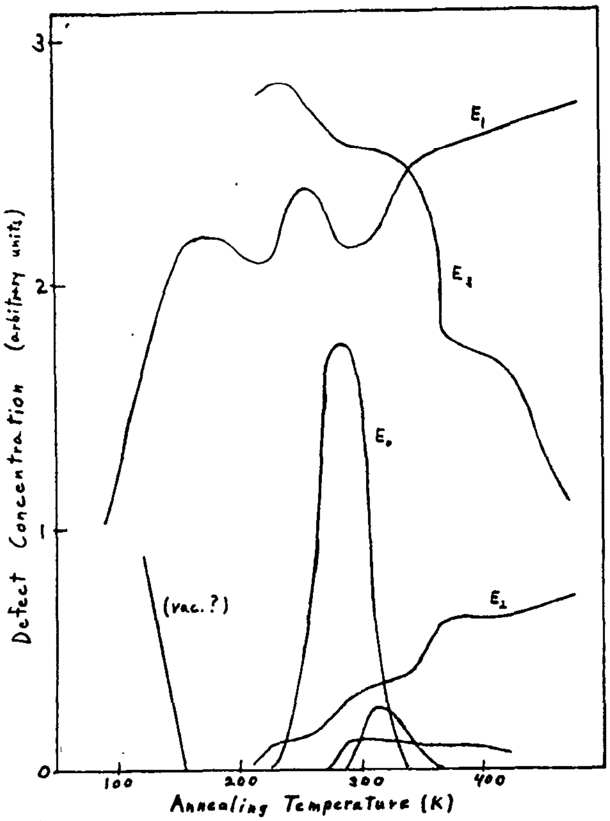
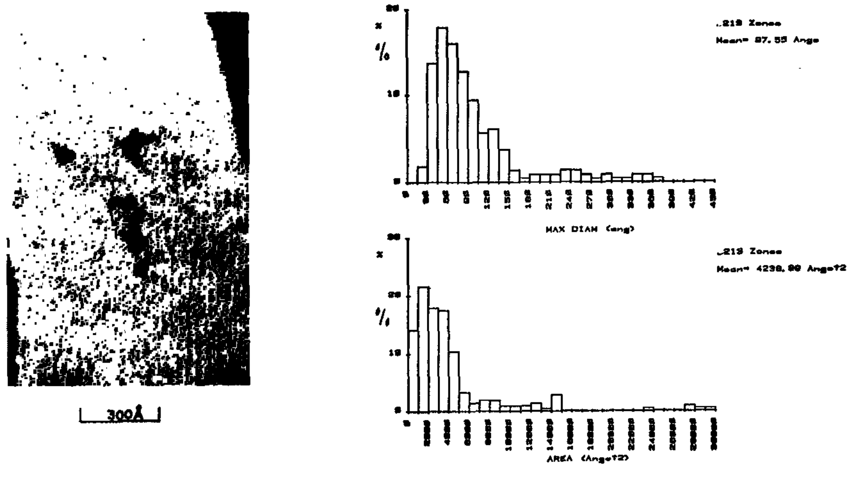


Figure 3. Annealing behavior of defect levels in fast-neutron irradiated n-type Si as measured by Deep Level Transient Spectroscopy (DLTS).

Individual defect cascades produced by fast-neutron irradiation can be revealed clearly as disordered zones in an ordered matrix by the appropriate transmission electron microscopy (TEM) technqiue.[23] One study was designed to compare images and sizes of disordered zones produced by fast neutrons from a fission source, CP5, and a spallation source, IPNS. We expected a strong similarity in size distributions based on the very similar distributions of neutron energies as seen in Fig. 1. This was indeed demonstrated in Fig. 4. In addition, some quite large disordered zones were observed and sized in the specimens irradiated with spallation neutrons. Although the numbers of disordered zones examined in this specimen was not sufficiently large to make a firm conclusion, it was probably true that we were observing a few disordered zones created by the very high energy neutrons in the spallation spectrum. Further study is currently in progress. This limited study, however, reveals the subcascade nature of these high energy events.

More extensive experiments on Cu_3Au are currently underway to study the irradiation enhancement of vacancy diffusion in this ordered alloy.[24] Several experimental runs over the past year have revealed

SPALLATION NEUTRON CASCADES (IPNS)



FISSION NEUTRON CASCADES (CPS)

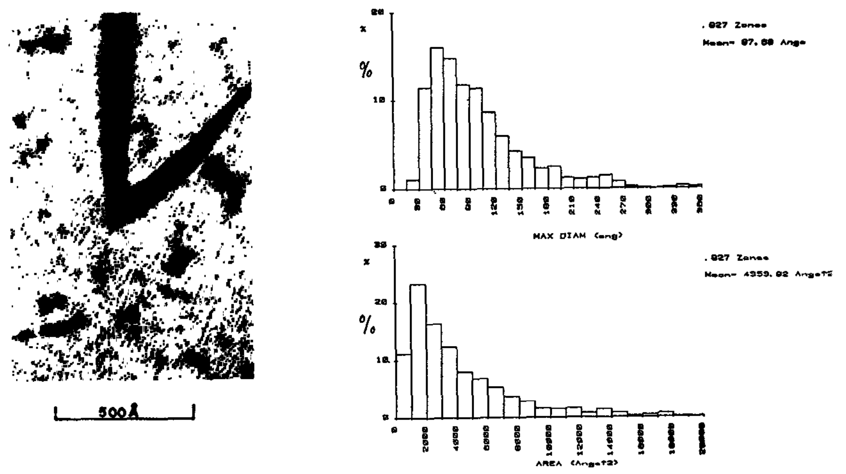


Figure 4. TEM high resolution images and size distributions of disordered zones (or cascades) in neutron-irradiated Cu_3Au. Images are recorded in dark-field superlattice reflection ($\bar{G} = 110$). Heavy dark lines are anti-phase domain boundaries.

very systematic behavior in the data. Changes in degree of long range order are measured by resistivity changes. Analysis with respect to theory is in progress. The data consists of the effect on ordering rate per incident neutron ($dS/d\phi$) of neutron dose (ϕ), dose rate ($\dot{\phi}$), irradiation temperature (T) and the sample order parameter (S). An example of the variation of ordering rate as a function of irradiation temperature and order parameter is shown in Fig. 5. This demonstrates that ordering is by vacancy diffusion only, and is not affected by interstitial diffusion; a result which has been demonstrated earlier by electron irradiation.[25] Note in the $S \approx 1$ sample that ordering by the vacancy diffusion is not possible and only disordering by the cascade disordered zones is observed. However some reordering of these disordered zones is apparently revealed and shows a temperature dependence.

Theoretical expressions combining the reaction rate theory of irradiation enhanced diffusion[26] with the reaction rate theory of ordering[27] have already been proposed by Zee and Wilkes.[28] We believe our data will be a quantitative test of both theories.

The effect of the pulsed irradiation structure has been investigated by us. No effect can be found in this particular experiment which demonstrates that time average values of flux, fluence and resultant damage parameters are suitable. This is in agreement

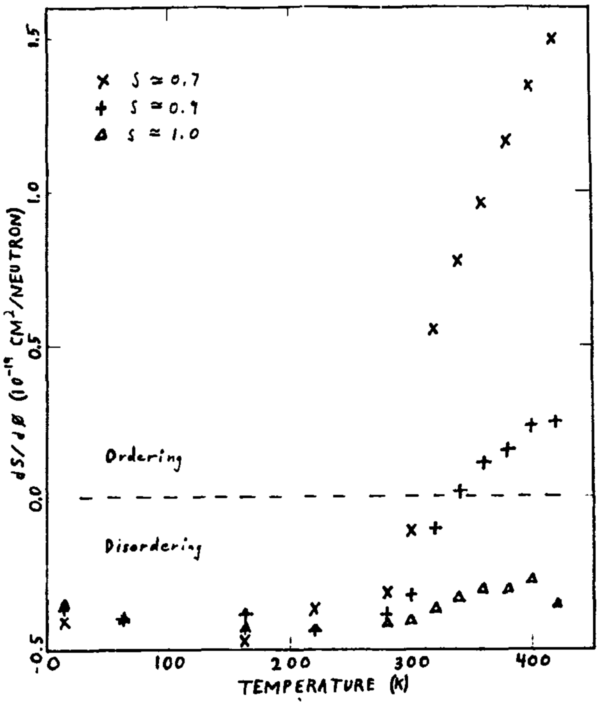


Figure 5. Ordering and disordering rates in fast-neutron irradiated Cu_3Au as a function of irradiation temperature in three samples with different degrees of long-range order (S).

with recent theoretical calculations appropriate to our experiment.[29] However, new experiments will begin soon in which an effect of the pulsed structure of the irradiation flux is anticipated.

A new and direct measure of the irradiation produced vacancy concentration and lifetime is proposed in Fig. 6. The change in sample order as a function of time is shown during irradiation and immediately following a precise shutdown of the flux. The decay of the irradiation produced vacancy concentration is observed over about 75 seconds. The difference in interception of the two straight lines at the precise time of shutdown is proportional to the vacancy concentration. The second linear slope was unexpected. Further investigation of this effect has suggested that we are observing a second mechanism for production of migrating vacancies, which may be the emission of vacancies by dislocation loops produced by the irradiation.

A central point to be made here is that precise control of neutron dose, dose rate and irradiation temperature is far easier with an accelerator based spallation neutron source (and dedicated target) than with a fission reactor. Precise and instantaneous full-flux start up and shutdown is possible only with a facility like IPNS-REF.

Summary and Observations

In this article we have described briefly the Radiation Effects Facility (REF) of the Intense Pulsed Neutron Source (IPNS) at Argonne National Laboratory and how it was designed, constructed and characterized by the group of Tom Blewitt. Coming from our experience with the CP5 research reactor we discussed the advantages and disadvantages of a pulsed spallation neutron source for irradiation effects research. The central advantages are the low gamma flux relative to a fission reactor source and a more precise control of irradiation parameters such as dose, dose rate, and instantaneous turning on and off. Two other differences of note are the high energy tail in the neutron energy spectrum and the pulsed structure of the irradiation flux (100 nsec pulse normally at a 30 Hz repetition rate). The high energy neutrons are few (3%) and of little or no consequence to irradiation studies in this, a degraded fission-like, neutron flux. The pulse structure, while little utilized at present, is expected to be useful to certain future experiments studying the kinetics of mobile defects.

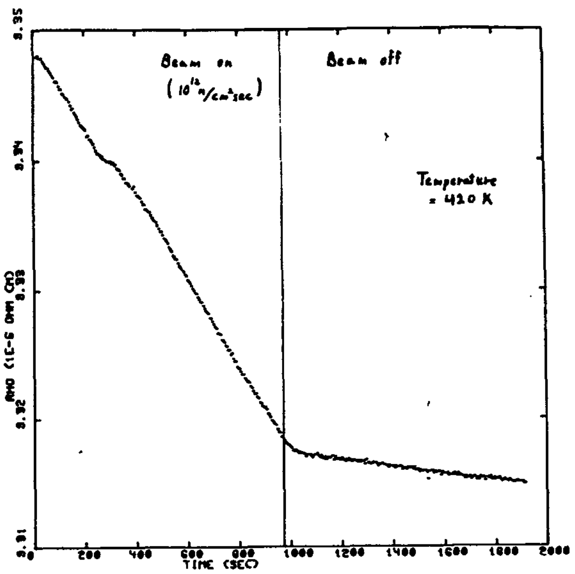


Figure 6. Ordering in Cu_3Au during neutron irradiation (beam on) at 420 K and following cessation of irradiation at 970 sec. Note decay of irradiation-produced vacancy concentration near 1000 sec and evidence for a continuing vacancy diffusion for time > 1000 sec.

The research program has been reviewed with a special emphasis on those experiments that employ the unique features of IPNS-REF. Approximately 50% of the research is performed by outside users of this national user facility; the balance is performed by the Neutron Irradiation Group in the Materials Science and Technology Division at ANL. Irradiation experiments on organic insulators and semiconductors are both strong components of this program as they require the relatively low-gamma flux. The experiments on irradiation enhanced diffusion in Cu_3Au require the precise control of irradiation parameters afforded by control of the accelerator. A neutron source such as IPNS-REF, essentially dedicated to this research, facilitates control of dose and dose rate without competition from other users.

If a next generation of spallation sources is constructed (with protons currents ~5 m·Amp.), it will be possible to include facilities for irradiations in neutron fluxes of 10^{14}-10^{15} neutrons/cm^2 sec and at controlled temperatures to as low as 4.2 K. This would exceed the highest practical neutron flux for this kind of research facility in a fission reactor by about an order of magnitude, and such a source would permit qualitatively new research in areas such as irradiation effects on mechanical properties. For this reason we view the IPNS-REF as prototypical and as an experiment itself.

It is possible, however, that the experiment will end within two years of the time of this writing. During this time a new facility in the Bulk Shielding Reactor at ORNL will be opened for use by the irradiation effects community. The new facility will have 5 times the flux of IPNS-REF and be available for experiments nearly year round. It will not have those features unique to a pulsed spallation source that we have been discussing. A foregone conclusion by many, including the funding agency, is that there is insufficient demand for two facilities. This author would seriously question such a conclusion in view of unique research opportunities at a pulsed spallation source and the possibility of a future facility. At the same time there is a need for more fundamental and applied research in the field of neutron irradiation effects. A recent study concluded as much if we are to have fission and fusion reactors as energy sources in the future.[30] The need is especially critical for students in this and related fields.

Any new facility in the future will need a strong in-house program in neutron irradiation effects. Good fundamental research is exceedingly difficult for outside users. The complexity and construction of many experiments make them impractical for the occasional visiting scientist. However, enlisting the aid of a local scientist at the facility can overcome much of this problem.

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