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# THE DIRECT SENSING OF DAMAGE TO ION IMPLANTED MATERIALS

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## Abstract

Material damage caused by the implantation of a high concentration of hydrogenic ions requires regular remote monitoring in order to study the atomic and nuclear reaction processes taking place within each sample. Real time continuous measurements of acoustic emission, X-ray production and emitted particle flux enable processes such as bubble or crack formation, changes in crystalline order, and nuclear fusion reactions can be studied in detail through examination of secondary or associated emission products. Fracturing of a material may generate a unique signature which, when taken in conjunction with time-averaged quantities such as changes in resistivity, surface strain, and induced radioactivity, enable an overall picture of the onset and nature of crack formation to be acquired. The overall usefulness of the remote sensing of damage processes and nuclear reactions is discussed. Surface studies involving inelastic Raman scattering and atomic force spectroscopy can contribute substantially to the overall picture, and identify clustering and cluster processes.

KEY WORDS: materials, implantation, fracture, remote and direct sensing, nuclear processes

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## Introduction

In discussing the possible ways in which damage to materials can occur, it is important to realize that some effects are long term, some immediate, and others largely indeterminate. The environment in which a material exists can eventually determine significant changes in its properties and behaviour. When a radioactive species is deposited within a material, damage is created internally which can be observed and diagnosed externally. Implantation of a material with significant doses of ions, hydrogenic ions in the present case, can alter surface and material properties in a dramatic way. The change in surface properties and the observation of induced damage to implanted materials, are the salient features of the review paper which follows.

## Clustering on Surfaces

Recent work at the University of Manitoba on the topic of surface characterization following hydrogenic implementation of a material surface has shown that low energy - keV hydrogenic ions - implanted in graphite, form complexes of the form $(CH_4)_n$ or $(CD_4)_n$ on the surface (Mathur et al., 1984). Later work with titanium carbide (TiC) has shown that the energetic implanted ion can separate the Ti and C elements and form $(CH_4)_n$ complexes on the surface despite the absence of clustering in the incident beam (McKee and Mathur, 1993). Why and how do ions conspire to form molecular species otherwise nonexistent in either beam or surface?

A recent paper by Khanna and Jena (1992) has addressed this question. According to their thinking, atomic clusters have become one of the most exciting areas of research in the last decade not only because their study can bridge our understanding between molecular and condensed matter physics but because the mechanism by which individual H atoms conspire to form a $CH_4$ molecule when meeting with a carbon atom in the surface is somewhat of a mystery. On the other hand it is clear that minimising the potential energy available is a high priority, and fracture of the TiC molecule rather simple. Clearly in many situations the cluster-cluster interaction is weak and the individual properties of individual clusters are maintained. Knight et al. (1984) have shown that alkali-metal clusters containing 2, 8, 20, 40,... valence electrons can be very stable since electron shells are closed. In general it seems that close atomic packing and electron shell closures can together give cluster-enhanced stability.

Recently, Guo et al. (1992) have published work relating to a new class of molecular clusters. In their work they discuss metallo-carbohedrenes and identify an exceptionally stable and abundant cluster which contains 8 titanium and 12 carbon atoms $Ti_8C_{12}^+$. In their studies of dehydrogenation of hydrocarbons by metal ions, atoms and clusters, a particularly abundant and stable species with molecular weight 528 AMU has been observed. M. S. Mathur, at our laboratory, has indicated that Raman inelastic data available to him may include spectroscopic data for such complex units. The $Ti_8C_{12}$ structure is of a pentagonal duodecadahedron kind which accounts for the observed stability if properly understood. The pentagonal rings each contain 2 titaniums and 3 carbons, each Ti being bonded to three carbon atoms, and each carbon bonded to its adjacent carbon neighbour. Details of surmise and calculation can be found in Knight et al. (1984) and Guo et al. (1992). Figure 1 shows an idealised cluster structure for $Ti_8C_{12}$.

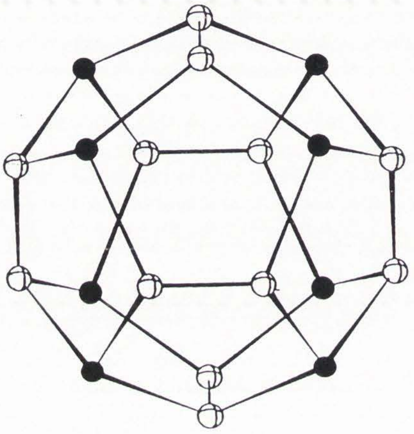

Fig. 1 Geometrical structure of $Ti_8C_{12}$. The filled circles are Ti sites and the shaded circles represent C sites.

Clearly, from experiments carried out at the University of Manitoba (e.g. Mathur, 1990), clustering of implanted ions at the surface of implanted materials is an important ingredient in the production of unlikely complexes in the surface region. Inelastic Raman scattering has identified such units.

## Damage to Materials

The nature of the damage induced in many materials by continuous ion implantation is not only difficult to assess but challenging to explain. Clearly this is a non-equilibrium thermodynamic system in some respects not unlike electrolysis. However in electrolysis surface modification stops when all available interstitial sites have been filled (Wang et al., 1990), by which time an observable expansion of the basic electrode material can be readily observed by means of a travelling microscope. In the case of continuous implantation on the other hand, implantation can continue despite the fact that all normal sites and interstitial locations have been occupied, and the question arises as to what happens in detail to the material or crystal concerned, and what becomes of the ions that continually arrive at the surface, lose energy through Coulombic and nuclear scattering processes and then attempt to find a proper site.

If the temperature of the implanted solid is controlled and the solubility of the implanted species has reached its normal maximum value, then we should expect the Fick diffusion coefficient to be small and that diffusion of ions out of the medium would be very small compared with the flux of incoming particles. It is, of course, no mystery that such an insult to a material whether metal or dielectric is likely to result in bubble formation, blistering and even fracturing of the material. It is also possible that a normal metal may locally undergo a metal-dielectric transition with a possible change in the conductivity or resistivity of the bulk, probably of a transient nature (Chechin and Trarev, 1990). The reality of such transitions will be discussed in a later section.

In addition, the routine monitoring of incident flux, and temperature is essential to the detailed study of implanted substances. Of great diagnostic value are strain gauge measurements and the analysis of acoustic emissions from ion implanted solids. These will be discussed in section 5 of this paper. Finally, the possibility of enhanced warm fusion cross sections either due to fractofusion effects in solid materials or enhanced tunnelling effects due to pyconuclear reactions cannot be totally disregarded. Pyconuclear reactions are nuclear processes at high density, where reactions are insensitive to temperature but very sensitive to density. Neutron detection should therefore be carried out on a routine basis, neutrons being the fusion products most likely to escape from a damaged material.

Means of monitoring these phenomena remotely, if not always in real time, will be described in detail in the paragraphs to follow, with some theoretical justification attached where appropriate.

In experiments carried out at the University of Manitoba, a linear ion accelerator employing a duo-plasmatron ion source is used as a 20-120 keV ion implanter. In order to achieve good vacuum a clean environment is maintained and a liquid nitrogen cooled jacket surrounds the target. A computer controlled beam scanning mechanism is used to deflect the ion beam across the target surface. In this way uniform implantation is achieved.

In the work described in the following sections, implantation of 30 keV $d^+$ ions is involved. Recent experiments on the implantation of heavy metals (Pd, Ti and In) with low energy deuterons have led to the observation of neutrons from the $D(d,n)^3He$ interaction. As the concentration of deuterons in the heavy metal matrix increases, deuterons form the target nuclei for bombardment by subsequent deuterons. Since some deuterons have energy exceeding the threshold energy of the reaction, neutron emission is expected. The flux of generated neutrons has been examined for anomalies in the production rate, or in the absolute number of neutrons produced during the experiment. Two strategies have been adopted for the observation of such neutrons: Firstly, by placing a piece of In foil close to the target heavy metal, a meta-stable state, $^{115m}In$, is formed by inelastic neutron scattering in the foil which acts as a target. After the implantation has finished, the decay of the meta-stable state is observed in a low background environment. The total number of neutrons generated during the experiment is then estimated. Secondly, a cylinder of NE-102 plastic scintillator is placed in the vicinity of the implantation chamber, and direct monitoring of neutron production observed. This device is called the On-line Neutron Monitor (ONM) and an investigation of the character of the neutron generation rate as a function of time in the experiment is made. This device is described in detail in Mathur et al., 1990).

These methods are checked against one another, since the record of the second experiment can be summed to yield an estimate of the total number of neutrons detected during the experimental run. Details of this method have been published elsewhere (Durocher et al., 1989; McKee et al., 1992).

## Surface Damage, Blistering and Cavity Formation

The advantage of implantation with particles of energy in the 20→100 keV range is that surface penetration is assured for light ions and although surface sputtering may occur, most energy is deposited within the implanted material. A characteristic hydrogenic ion range would typically be several microns for a medium Z material (in this energy regime, from our experience). The formation of surface cavities by energetic ion bombardment is a recently studied phenomenon and most of our understanding is due to Wilson et al. (1988) who made pioneering measurements on an implanted $SiO_2/Si$ interface, and examined the nature of hole formation from ions of various species and energies. In this work the oxide surface was etched away after bombardment and measurements of both the depth and diameter of resulting cavities made. The data were then interpreted in terms of nuclear and Coulombic energy deposition. The production of electron cascades by heavy ions results in the pseudo-cylindrical holes that are punched in the surface as they have demonstrated.

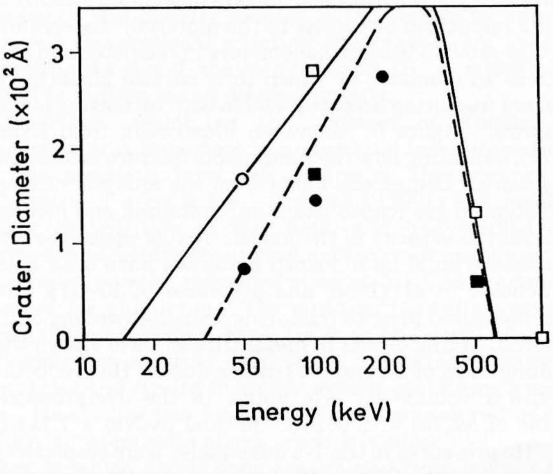

Fig. 2 Plot of the mean crater diameter vs ion energy and the theoretical prediction of the lateral extent of the cascade based on an energy-deposition cutoff at 90 eV/Å at the $SiO_2/Si$ interface for oxide thicknesses of 40 and 200 Å (4 nm and 20 nm).

An analytical implantation code incorporating predicted energy deposition profiles called SUSPRE has been used to predict lateral extension and crater depth (Webb and Wilson, 1986). A plot of mean crater diameter vs. ion energy is shown in Figure 2 as is the dimension of the cascade shower. The agreement between theory and measurement in the study of $SiO_2/Si$ interfaces is good, and scanning tunnelling microscope measurements (STM) show a projecting rim to the craters.

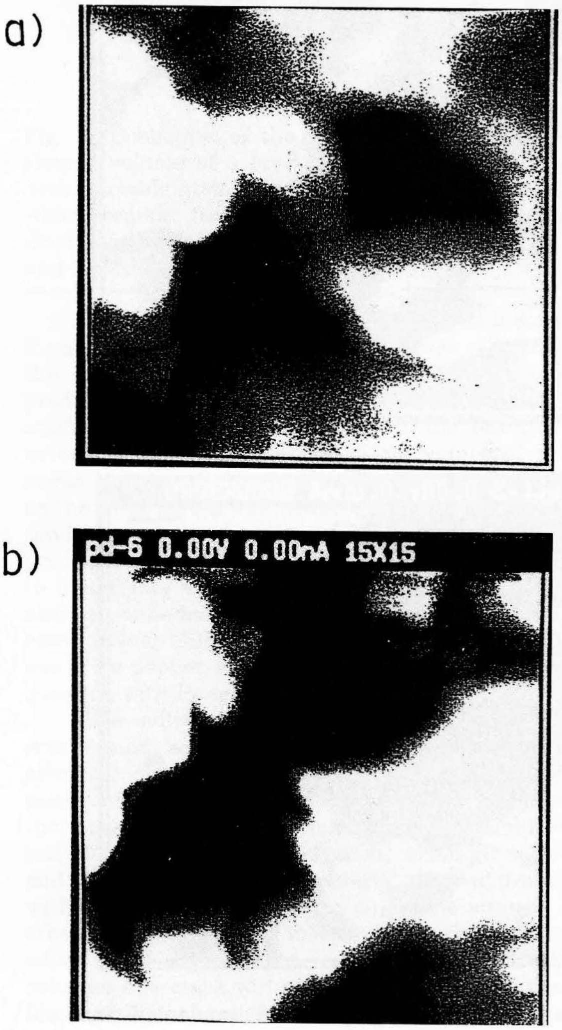

Fig. 3a,b AFM micrographs of $d^+$ implanted Pd, scale 500 nm x 500 nm. Regions a) and b) of same sample.

The new microscopies, STM and AFM (atomic force microscopy), allow direct observation of solid surfaces on the atomic scale and are well suited to the investigation of surface defects. We have used a commercially available AFM (Park Scientific) to study both implanted and unimplanted Ti and Pd. The surface damage in the case of the implanted material was clearly visible in the case of 30 keV $d^+$ implanted Ti (random orientation) at a dose of $10^{18}$ particles, as can be seen from Figure 3(a,b). The Pd samples were exposed to a much higher total fluence and the damage shown in Figure 4(a,b) is extensive but not possible to interpret. Crater diameters of 50 nm seem to be the norm for $d^+$ implanted Ti samples which is somewhat larger than might be expected for 30 keV ion implantation. Profile depth is unknown but probably several nanometers in magnitude. The force used in this AFM experiment was around 3 N. It is hoped to carry out detailed studies of crater formation and energy profile distributions for this system in the near future.

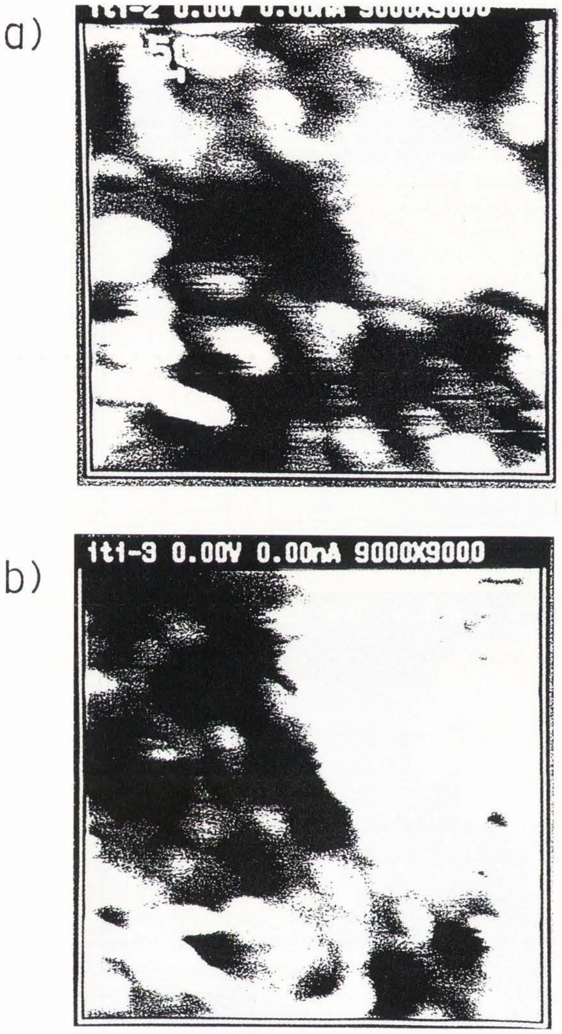

Fig. 4a,b AFM micrographs of $d^+$ implanted Ti, scale 400 nm × 400 nm. Note 50 nm craters. Regions a) and b) of same sample.

Despite the significant fluxes of incident ions used in the implantation of Ti and Pd samples, little blistering of the surfaces was observed in the initial stages. Such blisters as were observed were of several microns in dimension as studied by SEM (scanning electron microscopy), which we believe reasonable for a rough surface implanted with gaseous ions of the energy quoted. Evans (1976a,b) has indicated that the dimension and existence of blisters is dependent upon the uniform smoothness/roughness of the surface and that an irregular rough surface may inhibit blister formation to a large extent. Our observations would confirm that premise. Of course, the situation with >10 keV ions is quite different (Armour and Al-Bayat, 1992), and unlike the situation in which low energy ions (<100 eV) are implanted in a surface causing minimal damage and not more than one atomic displacement per ion. Here collision cascades occur involving many atomic displacements over considerable depths, and the changes in surface structure reflect the residual damage remaining around the track of each ion after the collision cascade has decayed. A schematic picture of surface damage due to an energetic ion is shown in Figure 5, showing the creation of vacancies and interstitials and the collapse of affected material (Wilson et al., 1988).

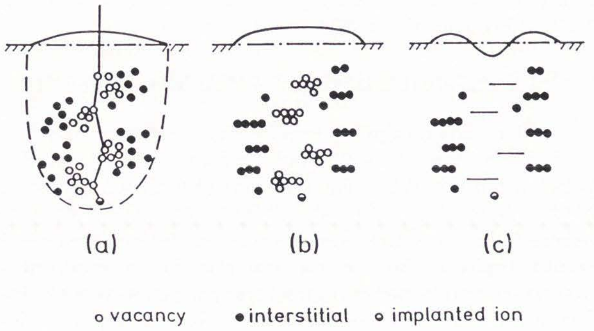

Fig. 5 Schematic illustration showing the formation of an ion impact crater.

In conclusion to this section it can be seen that STM, AFM and SEM facilities can be invaluable in assessing the overall effect of ion implantation of material surfaces, after the event.

## Fracturing of Materials - Theory and Sensing

When gaseous ions are implanted in a metal, a succession of events will occur; firstly there is bubble formation - the nucleation of bubbles depending sensitively on the impurities or defects in the material. Later, small bubbles grow to become nanometers in diameter and then microns in diameter at which time surface blistering or internal fracturing become possible with increasing bubble pressure. Figure 6 shows an illustration from Evans (1977), indicating how the interbubble fracture mechanism may work. Deductions made from the analysis of crack formation in gas loaded titanium, tantalum, and niobium indicate the veracity of the model. Actual measurements of pressure build-up in loaded materials have been made by Schober et al. (1986) and pressures of 10 GPa have been measured prior to fracturing, causing swelling of the material. Aging effects in metal tritides have shown that swelling rates of Ta and Nb tritides due to $^3$He production require a volume for $^3$He atoms in the overpressured bubble of $\Delta v(\mathrm{Ta}) = 9.98\times10^{-30}$ m$^3$ and $\Delta v(\mathrm{Nb}) = 7.7\times10^{-30}$ m$^3$. $^3$He pressures in the 1-2 nm bubbles were respectively ≥5.3 and ≥10.6 GPa. The dilatometric strain gauge technique required two 3 mm length gauges to be bonded to the two faces of the plate-like TaT and NbT samples. Resistivity changes were measured each second day using a 4-point probe technique and Wheatstone bridge. Resistivity changes, $\Delta R/R$ were measured, and the volume change found from the relation: $\varepsilon = \frac{\Delta L}{L} = \frac{\Delta V}{3V} = (1/k) \cdot \frac{\Delta R}{R}$,

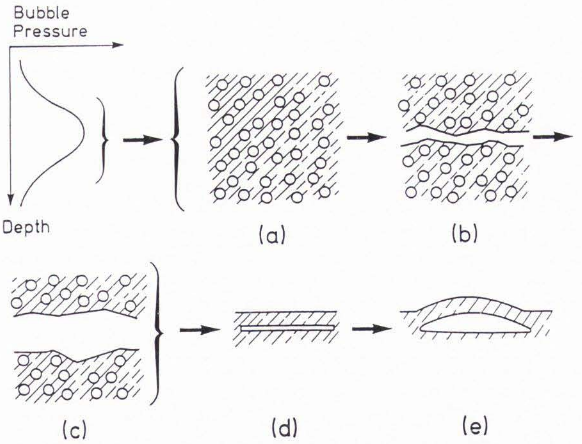

Fig. 6 Interbubble fracture mechanism: (a) high density of overpressurized bubbles; (b) crack formation; (c) bubbles adjacent to original crack become involved to widen crack and increase pressure; (d) penny-shaped crack which either extends to cause flaking or (e) forms blister by gas-driven surface deformation.

where k is the strain sensitivity, nominally 2.0 and known to ±1%. In these experiments the gradual change in gas pressure with tritium decay can be monitored on a continuing basis. In addition to monitoring pressure build-up however, fracturing can be observed indirectly through the study of acoustic emissions. More particularly, when small nm sized bubbles grow to become interstitial agglomerates, the formation of such a microstructure is accompanied by acoustic emission (AE) (Wilson et al., 1988).

Bubble nucleation is complete before AE sets in. Indeed it is believed that a single dislocation loop will give no observable signal, it being typically 1 nm in dimension (Schober et al., 1986). However a single loop punching process can trigger a whole avalanche of loop punching processes and a significant pressure wave.

As a monitor, a piezo transducer of appropriate frequency was directly coupled to the sample by silicone grease. Conventional counting electronics were used in detecting the signal, the gain being 90 dB. Samples were found to radiate sound discontinuously.

When examining the problem of fracture in materials, it is important to understand how interactions at the atomistic level can determine the advance or otherwise of a crack tip. Hoagland (1991) has made a major contribution to this field, and has studied the stresses and displacement gradients in crack models which lean heavily on the embedded atom model (EAM) potential devised for aluminum. The question basically concerns what happens when a crack tip is on the verge of either advancing or emitting dislocations. Hoagland computes the stress and strains in atomic models containing a crack tip. He studies two models of the same material one in which an atomically sharp crack is maintained and the other in which dislocations are emitted from a crack tip. In the computational procedure atoms are displaced from their crystalline configurations by means of a displacement field denoted by a stress intensity factor K.

If the force on the crack tip within the closed contour that surrounds the defect (tip) is J, then $J = AK^2$ where A is a stiffness dependent constant for the material, and indeed the component of force available to extend a crack tip is given by $F = J\text{-}2\gamma$, $\gamma$ being the surface energy. A schematic of the model is shown in Figure 7.

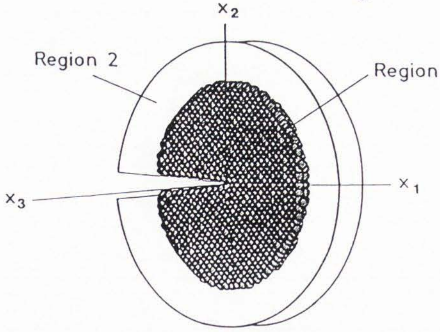

Fig. 7 Schematic of the model. The model is a disk-shaped volume of a cracked linear elastic infinite solid with movable atoms in region 1 and atoms in region 2 which remain fixed in the positions assigned by the displacement field for the crack with its tip at the origin and loaded to some K-level.

In summary, Hoagland's calculations indicate that for an anatomically sharp crack, stresses calculated from the model are in good agreement with linear elastic predictions except within one lattice parameter of the crack tip. It also appears that at the onset of crack extension and dislocation emission, values of principal stress and shear stress can be calculated, while the force on the crack agrees well with elastic predictions. Once the conditions for extension of the crack tip and the production of dislocations are identified, it is appropriate to investigate whether in fact a crack tip advance may also halt or be halted and self healing of the fracture then occur. Evans (1980) has suggested such a phenomenon in one of his papers, and the Manitoba group has raised this question with Hoagland.

He indicates that there are only two ways that a crack could accomplish that which is described, i.e., advance, arrest and then retreat (Hoagland, private communication). These two ways derive from the fact that there are two forces on a crack: one due to the loads and body forces applied to the system, which gives rise to J, and the other due to the material itself and associated with the surface free energy, $\gamma$. In the absence of any other energy dissipating mechanism, such as plastic flow, which is of course quite likely in ductile metals, the behavior of a crack will generally be subject to these two forces acting in opposition, J acting to advance the crack and $\gamma$, to remove it. Thus, in current experiments it appears that, if a crack advances, it does so in response to applied or internal stresses, and therefore a J, but with time the stresses change causing J to decrease, or perhaps even go negative, and the crack closes, healing in the process. The alternative, and more unusual explanation is that the surface energy increases with time thus causing the reversal.

There are no other "basic" conditions than these that will influence the behavior, but according to Hoagland there are many effects which may play a role and a few come to mind. Of course, one factor is the internal stress created by the dopants. Another derives from the fact that, as the material is doped it is very likely that dislocation emission from the crack tip will become more difficult due to increased hardening associated with dislocation/defect interactions and solid solution strengthening. In this regard the material becomes more brittle, the crack tip opening displacement decreases, and in so doing makes it easier for both the crack to extend (at a lower J) and to retreat (because the crack faces will be closer together). Another effect would seem to derive from surface contamination. If the experiments are all done in high vacuum, then any freshly formed crack faces would immediately coat making it difficult for the crack to heal.

Clearly in order to investigate this matter several criteria must be met. Firstly, remote sensing of crack formation must be possible, and AE seems the most appropriate means to routinely monitor such an occurrence (in our experimental situation). A Japanese group has reported its ability to differentiate between bubble emission and fracture on the basis of AE (Hiraga et al., 1990). In particular, acoustic emission following the β-decay of tritium in a titanium foil has been observed using PZT detectors. Signals due to acoustic emission following bubble emission and cracking have been detected and resolved from noise by means of high and low pass filters. These signals have been classified into three types of frequencies, and it seems that gas emission and fracture sound are clearly distinguishable. We have yet to confirm this fact.

Finally, in a previous paper which discusses the possibility of fractofusion in solids, McKee et al. (1992), the possibility of continuous X-ray emission from electrons accelerated across fracture gaps is examined and some data presented in relation to the phenomenon. For a high electric field to exist across a crack, the material must either locally or as a whole behave as a dielectric. A semiconductor is a possibility, or a metal with a high local density of implanted $d^+$ ions – thus reducing the conduction band and sharing valence electrons. The detection of X-rays from accelerated electrons could enable an estimate of the voltage gradient across the crack, considered as a shorted capacitor, to be made. For this reason, an intrinsic germanium detector should be continually monitoring the characteristic X-rays emitted by the target material, and also monitoring in real time the end point of the X-ray spectrum for evidence of fracturing of the material.

In the case of a conducting material, regular resistivity measurements can be useful, but an implanted insulator could generate evidence for X-ray emission following ion induced fracturing of the material, if such exists.

## Detection of Nuclear Particles

Where evidence of nuclear reactions between implanted ions is available, such as deuterons in a metallic lattice, neutrons can be expected to emerge at full energy from the implanted material. In this case, neutron flux from the sample can be measured immediately following implantation, from examination of residual γ activity in a foil of indium placed directly in touch with the bombarded sample. Neutrons of energy in excess of 2 MeV liberated in the nuclear reaction process, activate the indium, and a 0.336 MeV gamma ray with half-life $\tau$ = 4.50 h, can be readily detected. From analysis of the decay spectrum recorded by means of a NaI crystal, the incident neutron flux at completion of the irradiation can be calculated. A second monitor which can continuously register data in real time is the plastic detector viewed by two end mounted photomultipliers (ONM) referred to in the Damage to Materials section of this paper.

## Conclusion

Various techniques exist with which continual monitoring of damage to materials can be assessed. Damage to surfaces can be detailed and understood from studies of implanted material using AFM, STM and SEM, but this analysis is usually performed after bombardment. Resistivity measurements are also made irregularly, as is the neutron monitoring with indium foil.

Nuclear detector measurements and the study of acoustic emissions can however be used to observe events as they happen, and real time measurements can be made of nuclear processes in implanted solids. Measurements of temperature and beam current are two essential quantities which must be available throughout any implantation experiment. With such a variety of tools available it is to be hoped that much of the mystery surrounding damage to implanted materials can be solved.

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**Editor's Note**: All of the reviewer's concerns were appropriately addressed by text changes, hence there is no **Discussion with Reviewers**.