ELECTRON AND COMPLEMENTARY MICROSCOPICAL STUDIES OF NUCLEAR TRACKS IN SOLIDS

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INTRODUCTION

In 1958 Young [1], working at Harwell, UK, studied the irradiation by fission fragments and etching of single LiF2 crystals and showed that shallow pits or tracks were visible by light microscopy (LM). Silk and Burnes [2], also at Harwell, observed that fission fragment (235U) irradiation in mica produced linear trails of radiation damage. These trails formed a crystalline defect and consequently an electron diffraction contrast or track that corresponded to the passage of an individual fission fragment. Tracks were cylindrical in shape with lengths of several mm and 4-8 μm in width.

Nuclear tracks in solids (NTS) have been visualized today by all microscopy methods, from LM to electron microscopy (EM) and by related complementary scanning probe microscopy (SPM). SPM, with characteristics such as high resolution and diversity of electron, photon and ultrasound interaction with solids, now has a dominant place in science and technology.

The solid state materials in which latent tracks of energetic charged particles can be formed are minerals, glasses and polymers. Charged particles passing through dielectrics leave a latent track and the radiation damage can be observed by the following methods without treatment of track detector or by chemical amplification of the latent tracks.

ELECTRONIC LM

Latent tracks in crystals can be “developed” by immersing in a reagent which dissolves the radiation image. Prolonged etching enlarges tracks that become channels visible in LM. When the size of tracks is comparable to the illumination wavelength, the tracks become scattering centres in bright field illumination (Fig 1a). Modern LM includes electronic attachments such as video and image analysis systems (IAS) and these systems can be called ELM.

The structure of tracks gives information on the characteristics of particles and solid-state detectors. Consequently, the application of NTS in modern technology (nuclear physics and chemistry, radiation protection and dosimetry, environmental science, geosciences, material science and biomedicine) is very impressive.

During the participation of the Institute of Physics in an international intercomparison image analysis project (14 countries) [3], seven series of detectors (allyldiglycol carbonate Cr-39) were exposed to alpha-tracks from Rn, Am-241, B (n, alpha) Li and protons from Pu-Be (n, p) source. The main goal of the project was the intercompar-

Figure 1:
(a) Alpha tracks after IAS processing.
(b) Distribution of the diameter of tracks.
(c) Distribution of roughness of track openings.

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ion of the results obtained with different IAS hardware and software in the measurement of track density, circular and elliptical diameters of track openings, and many other parameters.

Presented measurements (Institute of Physics, Belgrade) were provided with an IAS designed in Yugoslavia with the following procedures: image acquisition, different processing (enhancement, correction of defects, thresholding and segmentation), binary image processing, feature measurements, and so on. A polygonal region-of-interest tool was used for restriction of image operation to selected areas; this was very important because all parts of image do not have the same background level. The next step before binary image processing was the determination of the limit value on the grey scale. The number of tracks in the field of view was measured using the automatic object numbering option in Ozaria software. The alpha tracks were analysed on a Leitz Laborlux-3 microscope using a semiautomatic IAS. The IAS consists of a JVC KY-1900E tube type colour camera and a Snapcy external digitizer attached to a Pentium PC. As an illustration, the distribution of diameters and roundness of tracks is shown in Figs 1b and 1c.

TRANSMISSION AND SCANNING ELECTRON MICROSCOPY

If the visualization of NTS by TEM is performed without etching, diffraction contrast of latent tracks and consequently high resolution TEM detection of atoms in crystal lattice is used. Etching of polymers with alpha latent tracks makes thickness contrast. TEM and SEM micrographs of the CR-39 polymer irradiated by alpha particles and etched are shown in Fig 2.

It is clear from SEM micrographs that the most frequently application of NTS is track-etched membranes filters (TEMF). TEMF offer distinct advantages compared to conventional membrane filters due to determined structural elements: pore size (10-0.01 µm), shape (cylindrical channel, conical, funnel-like, etc) and density.

TEMF are widely used for different filtration processes: general filtration, the first filtration from etching liquids such as acids, photoreists, ester solution, in cytology, chemotaxis, and gravimetry, as well as for serum filtration, ultrafiltration, sterilization, immunology, virus and protein filtration.

Polycarbonate (CR-39) membrane is used for microscopical, chemical and cytological studies. Polyester membrane filters and crystal (mica) membrane are also in use. Two commonly used commercial TEMFs are Nucleopore membranes and a membrane filter from the Schubnikov Institute of Crystallography.

To date, the applications of TEMF are in the research of microhydrodynamics, conduction of bacteria and blood cells, developments of metal microstructure (Portoroz 2000 [4]) etc. In all these studies, for morphological discovery of track (pore) structures, the membranes are scanned by SEM or AFM.

In modern TEM, the main position belongs to STM and AFM. STM is based on the quantum mechanical effect of tunnelling in which a particle (probe) encounters a barrier. A very small electron current (lt) flows between a sharp conducting probe (cantilever/tip) and the conducting sample surface. Atomic surface topography is reflected in the value of lt. NTS are nonconductive but some recent results [4] indicate the presence of tracks in metals.

The principle of AFM is that the surface is sensed by the force exerted on the probe tip (cantilever). The force changes from attractive to repulsive as the tip approaches the surface and its magnitude is measured. An optical lever is applied to measure the displacement of the probe. AFM is suitable for non-conductive samples for all detectors.

SFM application in NTS started with the Marburg 1990 conference [4]. Price [5] has reported that atomic and subatomic resolution could be achieved by AFM and he used the "super-resolution" for alpha recoil etch pits in mica (individual steps 2 nm high).

Figure 2 shows an AFM image of CR-39 polycarbonate detector etched in aqueous NaOH solution irradiated by alpha particles. As it can be seen, the openings of the tracks (pores) are not flat which is important if NTS are to be used as artificial biomembranes [6].

CONFOCAL LASER SCANNING MICROSCOPY

CLSM has many applications and is particularly useful in the study of NTS. 3D imaging of fusion fragments in mica (from optical slices) has been reported [7]. The resolution in CLSM is better than 0.1 µm, edges are sharper and contrast is better than in conventional ELM after computer processing of the images that are produced with the laser light from limited zones in a focal plane. CLSM allows one to obtain independently images of different layers of the specimen ("optical sections"). The track image in the detector (as the sum of sections) can be processed by computer to provide 3D information.

In CLSM the objective lens is used twice, to illuminate the sample and to image it. The image eliminates scattered, reflected or fluorescent light from out of focus planes. The optical section is absolute, the resolution is better and the image intensity drops as the image is defocused. The contrasts are better than in conventional LM.

SCANNING ACOUSTIC MICROSCOPY

CLSM stimulated developments in SAM which generates images based on acoustic or sound waves. The mechanical properties of the sample (sound speed, specific weight, viscosity, etc) determine acoustic contrast for visualization of ultrasound – mostly in reflection. The SAM is useful for visualization of crystal anisotropy and surface cracks. Scanning on anisotropy of ultrasound waves enables detection of very fine cracks [8]. In NTS, etched tracks present the “cracks” that can be analysed by SAM.
SOFT X-RAY RADIOGRAPHY AND X-RAY MICROSCOPY

For XM, soft X-rays (SX: 1-30 nm) produced by synchrotron radiation, laser plasma or the plasma focus device (PFD) of pulsed SX [9] are in use. In PFD, electric discharges at Z-pinch produce very intense SX pulses of short duration (ns). There is an analogue of TEM in transmission XM (TXM) and scanning TXM (STXM) [10]. SX contact microscopy (SXCM) is analogous to microradiography or heavy ion microscopy (HIM) in NTS. Also used are X-ray tomography (XMT) and X-ray holography (XH). The detection procedure for final imaging in SXCM is relevant to NTS because of the use of recording resists (to X-rays, electrons and ions).

In the Institute of Physics, Belgrade, a deuterium PFD [11] with a plasma focus chamber (Mather type) has been developed and its schematic view presented in Fig 4. The device generates neutrons, positive particles and other electromagnetic radiation. The PFD consists of two brass coaxial rods. The chamber was designed for currents up to 1 MA and a yield of 10\(^{10}\) neutron/pulse. Low inductance capacitors (Vm = 40 kV, E=66 kJ) with triggered spark gap as switching device are used for production of electrical discharges (Z-pinch).

Studies of emitted SX in PFD are carried out using a discharge through pure deuterium gas with X-ray film as the detecting medium. When the working gas was pure D or H, only SX is detected (wavelength under 10 nm, or energy about 3 and 5 keV). The dependence of SX-ray energy emitted from plasma focus on the working gas (H, D, He, N and Ar) used is shown in Fig 5.

Appropriate windows for the PFD chamber were constructed for spectrographic measurements and also for contact uses of SXR, objects and possible SXM (Fig 6a). To demonstrate SXR, a fossilized garden snail was used as the object, positioned directly on X-ray film at a distance of 140 mm from the central electrode. The radiographic picture produced from a single SX pulse is shown in Fig 6b, which is the absorption distribution of the 3D shell of the object in a 2D projection.

It was extremely hard to focus X-rays, so this was done by using zone plates, for SX, which consisted of concentric rings whose thickness decreased with increasing radius via diffraction. SXCM is an imaging technique which does not require the X-ray flux to be focused. Contact with a silicon wafer coated with an X-ray sensitive photosensitive material such as polymethylmethacrylate acts as the recording medium. After etching (by isopropanol) the photosensitive will have a 3D surface structure (type of replication). For further analysis the surface has to be visualized by SEM or AFM.

CONCLUSION

Visualization of NTS as latent tracks or etched tracks by electrons or photons is of primary interest in fundamental research. All microscopy methods also lead to a different applied research. TEMF wide ranging industrial and medical applications, due to their precisely determined structure by EM and complementary microscopy (IAS, STM, AFM, CLSM).

Modern SXM is between LM and EM and can image the internal structure, unlike EM. Our PFD was developed to generate neutrons, positive particles and electromagnetic radiation, including SX (~3 nm). The system can image hard biological objects by SX. Concerning the study of NTS by SXR and SXM at the moment, it seems that from SXR to SXM there is only one step. But, mutual connection of NTS detectors (polymers) and registration media for SXR and SXM are similar in nature and processing. The small size of the PFD system for all emitting radiation might be of interest for future applications.

REFERENCES


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