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Electron Microscopy and Analysis, 1985

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Preface

The new medical and dental school of the University of Newcastle upon Tyne provided the venue for EMAG '85. There were over 220 participants, 148 contributed papers and a large and well equipped trade exhibition.

For the majority of the electron optical techniques discussed, a quantitative understanding of the optics and specimen scattering relationships has been gained over the last two decades. This was reflected in the contributed papers, as the topics mainly concerned the interpretation related to a particular specimen rather than broad generalisations. It was also noticeable that the techniques were being used to their fullest extent and on specimens that a few years ago would have been considered beyond the capability of electron microscopy or analysis.

There were twelve invited speakers—four from France, two from the United States and six from Britain. After a welcoming address from the Vice-Chancellor of Newcastle University, Professor Humphreys commenced the technical proceedings with a provocative paper in which he demonstrated the electron beam drilling of 1 nm holes in alumina and other ceramics. The mechanism of this electron beam interaction was the subject of much discussion. In many ways this paper was typical of the rest of the meeting in that the papers were seeking an understanding of the specimen that was hitherto unknown because the electron optical techniques were not previously applied with such rigour or understanding. Other notable examples later in the meeting were the imaging and image simulation of pentagonal quasicrystalline structures in contributions by Portier and Knowles, and the movements of atomic columns in small gold crystals shown by Smith and Bovin.

A paper by Bourret opened the session on boundaries and together with the other papers in this session showed that high resolution electron microscopy has become a standard tool for the study of interfaces and non-periodic regions in crystals. This use of HREM was also seen in the work of Audier who discussed graphite formation on small metal particles and also in the session devoted to ceramics and surface layers. It was of interest in this latter session that the plenary paper by Lewis and many subsequent ones were using HREM as well as microanalysis to characterise technologically important materials.

Another method of structural determination is convergent beam electron diffraction. An enthusiastic session dealt with many aspects of this technique and showed that it is sensitive to strain and minor symmetry changes even in thick specimens. This type of sample is unsuitable for HREM and thus the two techniques are complementary.

Structural determination is also complementary to elemental analysis and sessions on EELS—with a plenary paper by Sevely—microanalysis of metallic systems opened by Titchmarsh, and the techniques and developments in microanalysis introduced by Goodhew, gave a well balanced assessment of the techniques and applications of analytical microscopy. The use of analysis and structural determination was highlighted in the session on surface reactions introduced by Flower. It is symptomatic of the importance of electron microscopy that complex chemical reactions at interfaces can now be examined.

The sessions described previously were concerned with the application of well developed techniques to stable specimens. One aspect of EMAG is that it is also concerned with new techniques and phenomena. The session on STEM showed that information can be obtained in reflection and microdiffraction modes that is unobtainable by TEM and that specimen excitation by the narrow, intense beam can provide spectroscopic information. Scanning Auger microscopy was the subject of several papers, notably by Prutton and his co-workers; and a workshop on high voltage microscopy chaired by Boyes examined the results and capabilities of the latest commercial instruments.

One session dealt with organic crystals and polymers. Apart from certain compounds, e.g. phthalocyanines, these materials have been considered only suitable for low resolution imaging—often by replication. Dorset and other authors in this session showed that high resolution information can be obtained by electron diffraction and lattice imaging. In a cooled specimen of paraffin, lattice resolution of 0.25 nm was obtained. The symmetry of these crystalline materials is usually low but Wittman showed how, with controlled epitaxial growth, the orientation and growth habits of the crystals can be tailored to an optimum geometry. High resolution imaging, convergent beam and microdiffraction methods were all described in successful applications to organic crystals, thus opening up an important new field for electron microscopy and analysis.

In the meeting nearly half of the papers were presented as posters which in many cases comprised the total contributions on certain subjects. My only retrospective regret of this meeting is that I wish it had been extended by a day devoted entirely to the posters.

On the social side Newcastle proved popular. The City of Newcastle and the Lord Mayor gave a lavish welcome to the delegates with a meal in the City chambers on the opening evening. The dinner was followed by a display of Morris dancing and folk-singing but despite these ancient festivities it continued to rain for much of the next four days. The following evening there was a buffet provided by the exhibitors and the conference dinner was on the final evening.

The biennial EMAG meetings have grown steadily over the years and are regarded as representing the state of the art in electron microscopy. I extend my thanks to the invited speakers, contributors and all participants for maintaining this standard.

John R Fryer
Programme Organiser

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Paper presented at EMAG '85, Newcastle upon Tyne, 2–5 September 1985

Nanometre-scale electron beam lithography

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Abstract. Holes and lines only 1nm across may be directly cut in a variety of inorganic materials using an intense focussed beam of electrons. The mechanism involves electron stimulated desorption, and EELS spectra recorded during the cutting process reveal structural changes occurring. Material may be removed 'at a distance' without being in the path of the electron beam. The aspect ratio of the holes is so great that the Uncertainty Principle is apparently violated.

1. Introduction

If an intense electron beam, current density typically 10^7 A m^{-2}, is focussed onto certain inorganic materials it drills holes which can be less than 1nm in diameter (see Fig.1). If the beam is moved it cuts lines, which can be less than 1nm wide.(2) We have called this process SCRIBE (Sub-nanometre Cutting and Ruling by an Intense Beam of Electrons). This writing is much narrower than other forms of electron beam lithography, which use organic resists, and it is also a one-step process, requiring no chemical development. Materials which have been drilled by the SCRIBE process include β and β"alumina (Mochel et al. 1983a), NaCl (Isaacson and Muray 1981), amorphous alumina (Mochel et al., 1984, Salisbury et al., 1984), CaF_2 and MgO (Salisbury et al. 1984) and AlF_3 (Muray et al. 1984). Patterns cut in most of these materials (not NaCl) are stable to the atmosphere. Not only may holes be drilled and lines cut, it is also possible to machine the external shapes of the above materials on a nanometre scale.

Applications of the SCRIBE technique may be far reaching, since we can use this to fabricate structures at dimensions previously inaccessible. For example, nanometre-scale 3-D electronic devices may in principle be fabricated which are extremely fast because of their small size. By drilling an array of holes of chosen size molecular sieves may be made, tailored to specific molecular dimensions. Memories may be fabricated with a density one million times greater than at present. Custom-built

biotechnological materials could be synthesized by patterning a substrate in a desired form on a molecular scale, etc.

2. Basic features of the SCRIBE process

The resolution of conventional electron beam lithography is limited by the spreading of the primary electron beam in the resist, by backscattering from the substrate, and by the distance over which low energy secondary electrons are created and travel (organic resists are exposed mainly by secondaries). Resolution in the SCRIBE process appears to suffer from none of these factors. Fig.3 shows that a hole drilled through 1000Å of Na β-alumina maintains its straight sides (Mochel et al. 1983b) with no evidence of broadening. The hole diameter is 20Å ± 2Å: the ±2Å corresponds to surface roughness on an atomic scale. In the SCRIBE process the electron beam undoubtedly broadens and creates secondaries, however, because there is a minimum threshold beam current density for drilling we believe that only the central position of the primary beam drills - hence the very straight holes.

Do the holes drill from the top or bottom of the specimen? To answer this question we have partially drilled a hole then tilted the specimen (Mochel et al. 1983b). Fig.4 shows that for a thin specimen, the hole starts at both surfaces, the craters move inwards and join up to form the hole. For thick specimens the drilling is mainly from the electron entrance surface.

Is the drilling at a uniform rate? The transmitted electron intensity, I, was measured as a function of time, t, during drilling. For some materials, e.g. Na β-alumina, it is found that ln I is proportional to t, i.e. the material drills at a uniform rate (Mochel et al. 1983a) of typically 250Å s^{-1}. For other materials, e.g. amorphous Al_2O_3, the material drills rapidly at first, followed by a plateau region which may last for many seconds before perforation finally occurs (Fig.5). Some other materials drill extremely rapidly, in milliseconds. The reasons for these different behaviours are not yet clear, but it appears that somewhat different hole drilling mechanisms may be operating in different materials.

3. Mechanisms of the SCRIBE process

Specimens in good thermal contact with a conductor may be drilled, hence the specimen is not melting or vaporising. The threshold beam current density for drilling increases as the incident electron energy increases (Salisbury et al. 1984), hence direct electron displacement damage and sputtering are not responsible for the drilling. Since the cross section for ionisation damage increases with increasing electron energy, this suggests that ionisation damage is the primary mechanism responsible for electron beam cutting. The fact that thin specimens drill from both surfaces indicates that surface desorption is important.

We suggest that the material drills atom plane by atom plane from both surfaces, with the anion (e.g. oxygen in Al_2O_3) being desorbed following ionisation by the incident beam and the cation migrating to the sides of the hole. A number of different mechanisms exist for electron stimulated desorption (ESD). We will apply one of these, the Knotek-Feibelman (1979) theory, to the particular case of Al_2O_3. An energy level diagram of Al_2O_3 is given in Fig.6 : the highest occupied level of the Al^{3+} ion is the