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Preface

Ten years ago the Electron Microscopy and Analysis Group held its twenty-fifth anniversary meeting in Cambridge. To mark that occasion the proceedings of the meeting were published and such was the popularity of this venture that all subsequent meetings in the biennial EMAG series have been accompanied by substantial proceedings. This volume bears evidence of the continuation of this tradition and it is fitting that after an absence of ten years the 1981 meeting should return to Cambridge.

Since the last meeting in Cambridge, the Cavendish Laboratory has moved from its original site in the centre of the city to a new site in West Cambridge. The new buildings provided not only excellent lecture theatres, but also a splendid exhibition site for the 33 firms whose products were displayed. The meeting itself followed a largely familiar format with sessions on instrumentation, electron scattering and diffraction, microanalysis, surface studies, image formation and analysis, high-resolution electron microscopy, beam-sensitive materials and studies of defects. However, in addition to these topics there were sessions on subjects not dealt with extensively in previous EMAG meetings. Foremost among these were data acquisition and processing, multi-signal analytical electron microscopy and semiconductor materials. Another venture introduced into the 1981 meeting was the incorporation of a half-day workshop on 'STEM -- the current and future state of the art' in place of one of the parallel sessions. No formal presentations were given, but a summary of the discussions which took place is included at the end of the volume.

Topics in the individual sessions were introduced by 14 invited speakers describing work carried out in Australia, USA, Canada, Germany and France, as well as in the United Kingdom. To open the conference Professor E Zeitler tackled one of the most difficult problems in electron microscopy, that of acquiring a quantitative description of specimens which damage under the electron beam. Despite the constraints this imposes on the experimentalist, the emphasis here and in later papers in the chapter is not on how little can be done but rather, with ingenuity and care, just how much information can be derived! Turning to specimens which are more stable under the electron beam, Dr J C H Spence and Dr P Goodman both show how detailed crystallographic information can be obtained using techniques far removed from the conventional imaging and selected area diffraction modes. These include convergent beam electron diffraction, beam rocking modes and the use of inelastically scattered electrons. Further papers describe theoretical beam–specimen interactions and the exploitation of carefully selected portions of the scattered radiation field to yield crystallographic data.

The instrumentation contributions fall into a number of categories. Dr K C A Smith describes how recent advances in computer technology, together with a decrease in cost of semiconductor memory, have led to a revolution in the collection and analysis of data in microscopy and related analytical techniques. Rather closer to the specimen itself are the many advances in the design of everything from individual microscope components to complete instruments. The state of the art here, together with a glimpse of what the future might hold, is provided by Dr E D Boyes.

Instrumental developments also play a large part in the contributions in the chapter on microanalysis where, despite competition from techniques such as cathodoluminescence, x-ray microanalysis and electron energy loss spectroscopy remain the most popular techniques. Dr G W Lorimer and Dr C Colliex provide excellent reviews of recent progress in the two techniques and many of the themes introduced here are developed in more detail later in the chapter. In many instances it is desirable to utilise both techniques simultaneously, along with the cathodoluminescent signal and different parts of the elastically scattered electron distribution, for complete characterisation of samples. This approach is introduced by Dr H Dexpert, who describes the need for it in a number of examples from solid state chemistry.

The advantages in using carefully selected portions of the scattered electron intensity distribution are described in the chapter on image formation and analysis. Two very different examples of what can be achieved are presented by Professor R E Burge and Dr R Sinclair, who show how innovative imaging methods can lead to information inaccessible using normal techniques. The large number of high-resolution investigations now under way highlighted in the paper by Dr D J Smith, and his subsequent papers in the chapter show the enormous advances that have been made in recent years in this field.

Finally, there are chapters on arguably the most important aspect of electron microscopy, namely its application to solving particular problems of widely varying natures. Dr A Howie introduces a chapter on surface studies in the electron microscope where he shows how information can be obtained which is not generally available using the conventional techniques of the surface physicist. The study of defects continues to be of great interest to electron microscopists and recent developments here are contained in the chapter headed by Professor G C Weatherly's paper on interfaces. A growth area within electron microscopy is the study of semiconductors, and Dr P M Petroff shows how a wide range of signals may be used to study these materials and in particular to provide information on electrically active defects.

In all, the topics contained in this volume cover a wide range of subjects of interest primarily, but not exclusively, to the physical scientist who wants to understand his specimen on a microscopic scale. It is hoped that those reading it will find it not only informative, but also a stimulus to the development and application of further advances in electron microscopy and analysis.

J N Chapman
Chairman
Programme Committee

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Paper presented at EMAG, Cambridge, 7–10 September 1981

Plenary Lecture: Radiation damage in beam-sensitive material

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1. Introduction

Williams and Fisher (1970) have clearly demonstrated that the production of a micrograph with high energy electrons degrades the structure of the (biologic) object to be recorded. As a remedy they propose applying the minimum dose required by the photographic recording medium. As a logical consequence, there exist two possibilities for improving this given situation--by (a) finding ways to make the specimen *less* sensitive to the interacting radiation and (b) making the photographic emulsion *more* sensitive to the interacting radiation. This dilemma is enhanced by the fact that the primary interaction of the radiation with matter--specimen, emulsion or any recording medium--is the same. The dilemma is also expressed in the nomenclature; i.e., the microscopist whose structures disappear speaks pessimistically of "radiation damage", while the chemist speaks optimistically of the "speed" of his emulsion (Mees, 1954).

In this context, we should not forget the many radiation-matter interactions which are considered beneficial and therefore investigated with vigor. For example, the shortage of energy has awakened new interest in photosynthesis with the aim of improving the conversion of light to energy which takes place in plants by means of man-made systems. The radiation-induced degradation of polymers (Tsuji, 1973) has become an inherent part of "lithographic" processes in the fabrication of micro-circuits. Here "radiation damage" is partly responsible for the advent of the microprocessor age. And there is radiation therapy. Of course we cannot overlook the other radiation damage problem more formidable than that of electron microscopy--namely that of mankind, where the awesome aspects are genetic and carcinogenic effects.

Radiation chemistry and radiobiology (Huttermann, 1978; Dertinger, 1969) have developed into vast and fast-moving fields with very serious problems to solve. The intention of this contribution is to vitalize the radiation damage studies in electron microscopy. (At present they are too descriptive; they are phenomenological and, especially, too isolated from the related above mentioned scientific endeavors.) Therefore I mention these active fields, which are sponsored by rich industries or responsible public health agencies, because there is a wealth of pertinent knowledge to be tapped and applied to our problems in electron microscopy.

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In turn, our findings will become knowledge in radiation chemistry. After all, the final purpose of *all* these studies must be to find a way to deduce the unadulterated structure of the microscopic specimen.

2. State of the art

In electron microscopy the term "radiation damage" has two meanings. For most of the users, it means destruction of the structure which the electron microscopist sets out to determine. To the materials scientist, it often means the displacement of atoms by electrons, simulating neutrons and other particles. Electrons offer the possibility for direct observation of the structure damage caused. We deal only with the first aspect, that of structure loss--which, except for newer low temperature efforts, is still covered adequately by Isaacson's review of four years ago! (Isaacson, 1977).

The loss of structure has not been quantified, although this is possible with modern image processing (Linfoot, 1956; Hawkes, 1980). Instead, standard procedures have been developed which record, as "analog signals", as it were, the appearance or disappearance of certain phenomena during the interaction of the electron beam with the specimen. The charge density, mostly measured in number of electrons per square Angstrom, is considered the cause of the fading signal and is therefore plotted on the abcissa. Over a wide range the law of reciprocity holds--that is, the effects depend only on the product of the current density multiplied by the exposure time. The customary phenomena are mass loss, fading of diffraction peaks, fading of energy loss peaks, and fading of optical absorption peaks. And, of course, a loss of visible structure. Most of the decay follows an exponential (single hit) law, so that the resistivity to radiation can be characterized by the charge density, which reduces the phenomenon under investigation to 37 percent (1/e) of its virgin value.

Probably for the sake of convenience, the fading of diffraction patterns is most often applied to the studies. It is found that the spots of higher-order reflexes fade more quickly than those of lower order. It is also found that at 4.2 K the diffraction spots of L-valine resist a charge density fifteen times larger than the critical charge density at room temperature (Müller, 1981). Although no relation between the various phenomena and the loss of structure information has been made, it is of advantage to have hard numbers signifying one aspect of radiation damage. For example, the success of attempts to reduce radiation damage can thus also be quantified. Many such studies can be found in the literature in which the effect of special specimen treatments --embedding and staining, freezing and cooling--lead to moderate success. As long as we do not know the chemical events which are triggered by the primary assault, however, all attempts to influence the radiation sensitivity will fall into the category of trial and error. In other words, enough measurements have been made within the microscope. We must now turn to radiation chemistry.

3. What goes on in the specimen?

In his classical contribution to radiation chemistry, Platzman (1967) distinguishes three phases in the response of molecules to irradiation.