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EDITED BY

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PREFACE

The five chapters in this volume range over the traditional subjects of these Advances, with an aspect of scanning electron microscopy in first place. Very low temperature SEM is a relatively new development: it merits separate treatment, for not only does it enable us to study superconductors by the techniques well developed in the scanning field, but it also proves to generate information of a different kind, due essentially to the localized heating effect caused by the energy deposited by the scanning beam. Not much of the work described here has yet found its way into the textbooks of SEM and we are delighted to publish so complete and authoritative an account by H. P. Huebner in these pages.

The second chapter, by R. L. Kashyap and K. B. Eom, reflects my efforts to increase the coverage of digital image processing in these pages. Image models are needed in various types of image processing, image restoration by statistical methods and image segmentation in particular. K. L. Kashyap has made important contributions to our understanding of image models, especially those that are robust in the sense that they remain reliable even if the assumptions on which they are based are not exactly satisfied. He and K. B. Eom cover the theoretical background of AR and ARMA models, robust estimation in the causal case, and restoration and edge detection based on these statistical foundations.

The third chapter examines questions raised by the extraordinary rate at which miniaturization is progressing. There must be a limit to this progress, but what is it, what governs it, and is it likely to be reached? R. W. Keyes considers the numerous factors involved, which are of very different kinds. At one extreme, we have physical laws governing speeds of propagation, mathematical laws associated with topology, the slippery rules of uncertainty, and the constraints of thermodynamics. At the other, there are economic pressures, which tend to mean that large scale progress, as opposed to isolated achievement, can only be expected if it pays off. R. W. Keyes succeeds in keeping all these pressures in mind in his discussion of the various devices used to process information.

In the fourth chapter we return to imagery, and, in particular, to synthetic aperture ultrasonic images. Ultrasound images are found in medicine, wherever non-destructive testing is vital and in the study of natural resources. Unfortunately, the wavelengths employed are frequently comparable with those of the structures of interest, and synthetic aperture techniques have hence been developed to provide good images despite this. K. Nagai examines the whole field, from wave propagation to the properties of transducer arrays and the performance of modern systems. This authoritative account will surely be of use to the experienced and invaluable to newcomers to the subject.

The final chapter is concerned with a problem that is encountered in all branches of electronics and electron physics: the calculation of electromagnetic fields. The dual complementary variational techniques presented here are not as well known as they deserve to be, despite the ubiquity of the finite-element method. J. Penman first sets out the mathematical tools needed and then examines first the two static cases, with examples from electrostatics and magnetostatics. He subsequently turns to the electromagnetic field, devoting a section to eddy current problems. This clear account of the ideas will surely help many of us confronted with the problem of field calculation to appreciate these methods.

As usual, we conclude with a list of forthcoming chapters.

Peter W. Hawkes

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CONTENTS

Scanning Electron Microscopy at Very Low Temperatures

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I. Introduction

Today scanning electron microscopy (SEM) is a widely used analytical tool providing structural information in many different fields such as materials science, solid state physics, microelectronics, biology, and the medical sciences (Reimer, 1985). The principle of SEM simply consists of scanning the surface of the specimen with a well focused electron beam and recording simultaneously a proper response signal generated by the interaction of the beam with the specimen. If this signal is displayed following the same geometric pattern as in the scanning process, a two-dimensional image of some specimen property can be generated. Usually, the response signal is utilized for modulating the brightness on the screen of a cathode-ray tube, which is operated synchronously with the electron-beam scanning process. In many applications of SEM, the response signal consists of the emitted secondary electrons or the back-scattered electrons. In addition, the emission of Auger electrons and x-ray photons is often utilized for structural imaging. Of course, the interaction processes generating these signals take place only within the penetration depths of the primary beam electrons in the sample material. Hence, the information obtained in this way is restricted to a region close to the specimen surface. The spatial resolution of SEM is determined by the diameter of the region perturbed by the electron beam and acting as the signal source. Hence, the beam diameter represents the ultimate spatial resolution limit. However, the beam-induced perturbation of the specimen often extends appreciably beyond the beam diameter, resulting in a corresponding deterioration of the resolution limit, perhaps by several orders of magnitude.

If the primary electron beam irradiating the sample is temporally structured, time dependent phenomena can also be investigated by SEM. Using the stroboscopic principle, strongly time-dependent structures can be observed with high temporal and spatial resolution.

Of course, the principle of scanning microscopy can be extended to any other probe that is movable in a two-dimensional pattern (Ash, 1980). A moving laser beam, acoustic beam, or mechanical micro-contact represent some examples that have been used for two-dimensional imaging. However, due to the well developed technology for generating and manipulating a sharply focused electron beam, so far electron-beam scanning has found the widest application in scanning microscopy. Here the small value of the beam diameter and the long working distance between specimen and lower pole-piece of the final lens, which can be achieved, represent distinct advantages of SEM.

Although SEM is now widely used as an analytical tool, its extension to the regime of very low temperatures is still relatively rare. Here we have in mind the temperature range provided by liquid helium, i.e., temperatures around 4 K and below down to about 1.5 K. For experiments in this temperature range one needs a scanning electron microscope equipped with a well-functioning liquid-He stage. With such an apparatus, two types of studies can be performed. First, typical low temperature phenomena such as superconductivity and low-temperature devices used in cryoelectronics can be investigated. Second, experiments can be performed where the temperature range of liquid He is required by the measuring principle. The ballistic phonon signal represents an example for the second case. This signal requires a long phonon mean free path and a highly sensitive phonon detector, both being realized only in the temperature range of liquid He. In the following review, we will deal with both types of applications of low temperature scanning electron microscopy (LTSEM).

Of course, in addition to electron beam scanning other scanning techniques can also be extended to the liquid-He temperature range (see Huebener, 1984; Bosch *et al.*, 1986). In the following we only discuss these other scanning experiments if they bear directly upon the results obtained by LTSEM. We do not present a critical evaluation and comparison of the different scanning techniques which can be performed at low temperatures.

The signals mainly to be utilized in SEM performed in the temperature range of liquid helium are expected to be different from those usually used during room temperature operation and mentioned above. Generally, the latter signals do not provide any new information if the sample is cooled to low temperatures. We will see that it is the localized heating effect caused by the electron beam during the scanning process that generates the important response signal providing the structural information about the specimen. This prominent role of the electron beam as a localized heat source and the importance of the beam-induced thermal perturbation of the sample results from the fact that at low temperatures many material properties can depend sensitively upon temperature. Here superconductors represent a particularly striking example since their energy gap often corresponds to thermal energies of only a few K. Of course, in an ancillary and helpful way the "usual signals" discussed above are always used in LTSEM in addition to the new signals only obtained at low temperatures.

In this review we summarize the results obtained recently by LTSEM. Following a brief discussion of the main features of the low-temperature stage in §II, we treat the important underlying principles of LTSEM in §III. In §IV we discuss the interaction between the electron beam and the specimen, concentrating only on the signal generating processes important for the low temperature experiments treated in the remainder of this article. In Sections V–VII we deal with spatial structures observed by LTSEM in superconducting tunnel junctions. In §VIII we discuss experiments relating to spatial temperature structures in current-carrying superconducting microbridges. Spatial structures generated in semiconductors during avalanche breakdown at low temperatures and observed by LTSEM are treated in §IX. The signals discussed in Sections VI–IX have some similarity to the concepts of the electron-beam induced current (EBIC) or electron-beam induced voltage (EBIV) utilized often in studies of semiconductors by means of SEM (see Reimer, 1985; Ehrenberg and Gibbons, 1981). In §X–XII we deal with a distinctly different signal for spatial imaging, namely the ballistic phonon signal. Here the region locally heated by the electron beam acts as a source of ballistic phonons (quanta of sound energy) in a similar way as the heated filament in a light bulb acts as a source of photons (quanta of electromagnetic energy). The ballistic phonons can serve for imaging the phonon focusing effect based on the elastic anisotropy in a single crystal. They can also be utilized for imaging structural defects in a crystal. These two applications of the ballistic phonon signal are treated in §XI and §XII, respectively.

II. Low-Temperature Stage

The best experimental setup for performing scanning electron microscopy at liquid-He temperatures appears to be an arrangement where one side of the specimen is in direct contact with the liquid-He bath, whereas the opposite side of the sample can directly be scanned with the electron beam. Such an arrangement is shown schematically in Fig. 1. Further, it is highly advantageous if the temperature of the liquid-He bath can be reduced from 4.2 K down to about 1.5 K by pumping. The operation in the temperature range below 2.17 K is of particular interest, since here the cooling efficiency of liquid He is strongly increased due to its superfluid state. Because of these considerations a bath cryostat extending into the sample chamber of the scanning electron microscope appears to be the best possible choice.

The typical features of a low-temperature stage based on the principles indicated above is shown schematically in Fig. 2 (Seifert, 1982). On the left side we see the lower part of a conventional ^{4}He cryostat consisting of a cylindrical liquid-He tank surrounded by a liquid-nitrogen tank for precooling and thermal shielding. The cryostat extends horizontally into the sample chamber of the microscope. On the right side in Fig. 2 we see the lower part of the electron-beam column and the sample chamber of the microscope. For thermal shielding it is important that the part cooled to liquid-nitrogen temperatures extends into the sample chamber in addition to the liquid-He tank. Horizontal adjustment of the sample position is possible by mechanically shifting the base plate carrying the low-temperature stage. Flexible connections between the low-temperature stage in the sample chamber and the cryostat on the left serve for gaining the necessary mechanical freedom. Helium gas bubbles forming perhaps near the sample and impeding the cooling process for the specimen can be removed by a circulation pump within the ^{4}He cryostat.

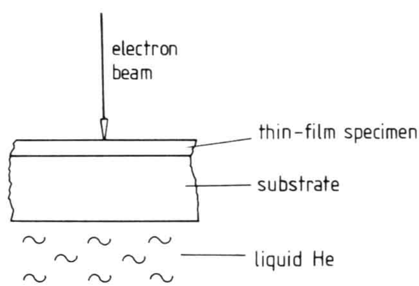

Fig. 1. Sample configuration for LTSEM.

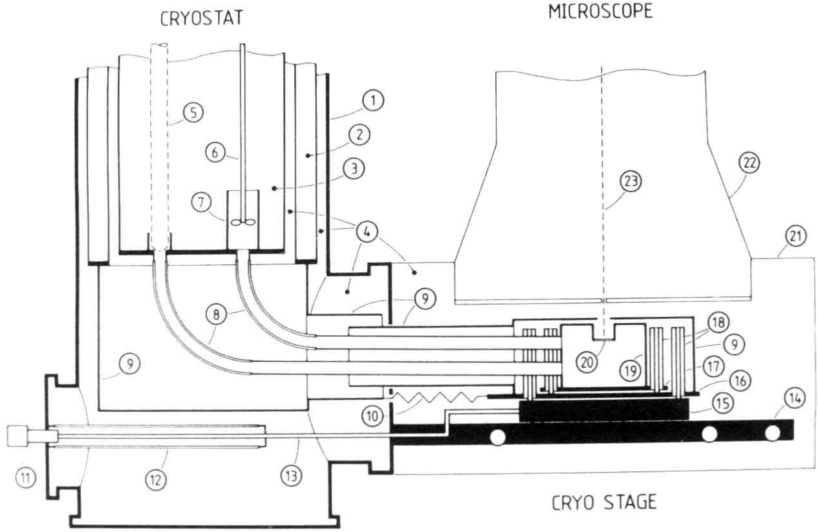

FIG. 2. Schematics of the low-temperature stage. 1, outer wall; 2, LN_2 reservoir; 3, LHe reservoir; 4, vacuum space; 5, position of LHe transfer line; 6, driving shaft; 7, circulation pump; 8, LHe transfer tubes with bellows; 9, LN_2 shield; 10, copper ribbon for thermal coupling; 11, micrometer screw for shifting micropositioning stage; 12, mechanical vacuum feedthrough; 13, push rod; 14, mounting plate; 15, x-y micropositioning stage; 16, LN_2 base plate; 17, LHe base plate; 18, spacers for thermal isolation; 19, LHe tank; 20, sample; 21, microscope chamber; 22, microscope column; 23, electron beam (reproduced from Seifert, H. *CRYOGENICS*, 1982, *22*, 657–660, by permission of the publishers, Butterworth & Co (Publishers) Ltd. ©).

The top plate of the He tank in the sample chamber can be used directly as sample holder. Such a sample mounting configuration is shown schematically in Fig. 3. Here the sample material separates the liquid He from the vacuum of the electron-beam column. The sample, shaped preferably as a round disk (of, say, 20 mm diameter and 2–3 mm thickness), is fixed mechanically by a clamping screw which also compresses the indium seal between sample and top plate. It is important to keep the sample position sufficiently low such that the liquid-He level is always higher. The top plate is fixed by a clamping ring and sealed also with indium. A shield above the sample with a hole for the electron beam provides protection against thermal radiation. Typical dimensions of the He tank are about 4 cm height and 5 cm diameter, corresponding to a volume of about 80 cm^3.

Of course, this sample mounting configuration is also very useful for investigating thin-film structures deposited on the top of a proper substrate, the substrate being again preferably shaped as a round disk. Due to its high heat conductivity, single-crystalline sapphire is well suited as subtrate

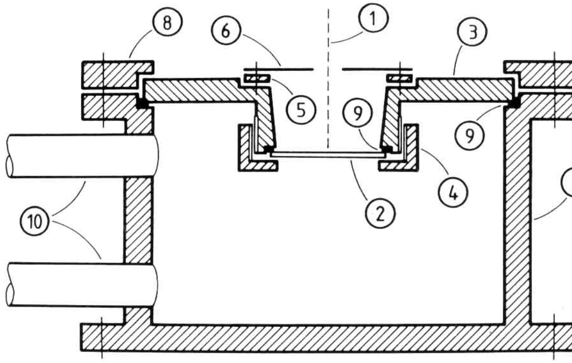

FIG. 3. Sample mounting configuration. 1, electron beam; 2, sample; 3, sample holder; 4, clamping screw; 5, copper ring for wire heat sinking; 6, thermal shield; 7, LHe tank; 8, clamping ring; 9, indium seal; 10, LHe tubes (from Seifert, 1982).

material for such thin-film structures. In the same way, other specimens which are unsuitable to act directly as the separating wall between the liquid He and the vacuum because of their small size or their mechanical weakness, can be mounted on such a substrate material with high heat conductivity. Sufficient thermal contact to the substrate can be attained by a proper medium such as stycast cement, vacuum grease, etc.

A photograph of a low-temperature stage which has been used for several years in the laboratory of the author is shown in Fig. 4. The circular flanche (diameter = 6.8 cm) represents the top plate of the He tank, the sample being located below the opening in the middle. The whole stage is to be inserted into the sample chamber of the microscope. In the back, the end plate of the horizontal extension of the cryostat with the rubber O-ring seal can be seen.

If electrical current and voltage leads are to be attached to the top side of the sample or of the substrate (electrical connections to a thin-film structure, etc.), it is important that these lead wires are thermally anchored to the liquid helium bath after a short distance, in order to minimize sample heating effects. In some applications of LTSEM, it is necessary to apply an external magnetic field to the sample. Such a field can be generated by a small superconducting coil placed in the liquid He surrounding the sample location. On the other hand, it can become important to carefully shield the sample against any ambient external magnetic field such as the earth's magnetic field. An effective magnetic shield of the sample can be fabricated from a magnetically soft material such as cryoperm (obtained from Vakuumschmelze GmbH, Hanau, FRG). Figure 5 shows a cross-section of the complete sample mounting configuration with the magnetic shield in place. Such a magnetic shielding can

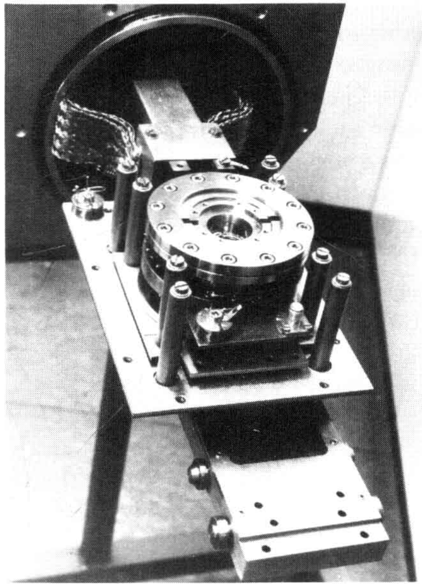

FIG. 4. Low-temperature stage.

has turned out to be highly effective in LTSEM studies of superconducting tunnel junctions, as will be discussed in Sections V–VII.

The ease with which the low-temperature stage can be attached to and removed from the scanning electron microscope represents an important consideration. A quick turnaround time for changing the sample is always attractive. Further, intermittent operation of the microscope at room temperature for conventional applications is often required. A low-temperature stage of the type shown in Figs. 2 and 3 can be self-supporting and

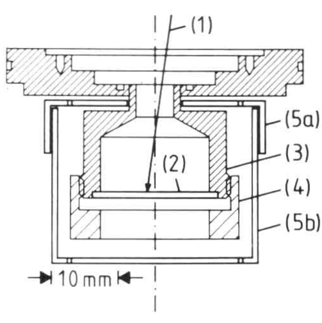

FIG. 5. Cross-Section of the sample mounting stage including the magnetic shield. 1, electron beam; 2, sample; 3, sample holder; 4, clamping screw; 5, magnetic shield.

provides this flexibility. On the other hand, scanning electron microscopes are commercially available today, where the low-temperature stage can directly be attached to the hinged door of the sample chamber without any further mechanical provisions. The fact, that the weight of some of the more elaborate sample tables available today is similar to the weight of the complete liquid-He stage including the cryostat, provides such a possibility. (As an example, in the laboratory of the author, a liquid-He stage is mounted in this way to the hinged door of a Camscan Model S4DV scanning electron microscope).

It is important to note that we have concentrated so far on a form of the low-temperature stage which is most universal in terms of its applicability and most effective in terms of its cooling power. For special applications simpler and less expensive cold stages are often adequate and commercially available. Here the specimen is mounted on some cold finger extending into the sample chamber of the microscope, and no direct contact between the liquid He and the specimen is provided. At present the lowest temperature which can be reached with such simple cooling stages is often limited to 10–15 K. In this review we exclude such applications from our discussion.

III. Principles and Electron Beam Parameters

The principle of SEM is illustrated in Fig. 6. The primary electron beam is scanned over the specimen surface and produces a localized perturbation of the sample. As a consequence the sample generates a response signal which generally depends upon the coordinates of the electron beam focus. The electron beam of a separate cathode ray tube (CRT) is operated synchronously with the primary electron beam. If the beam intensity of the CRT is modulated by means of the sample response signal, a two-dimensional image of the specimen property corresponding to this response signal is obtained. In addition to this two-dimensional display by means of the brightness on the CRT, a linear scan with the signal amplitude plotted against the scanned coordinate is often useful for a quantitative analysis of the results. This latter operational mode is referred to as "*y*-modulation".

For SEM studies at low temperatures, the localized heating effect of the electron beam already results directly in a highly useful response signal (see Clem and Huebener, 1980). At liquid-He temperatures the electronic properties of superconductors as well as semiconductors respond sensitively to small changes in temperature. Therefore, in many applications of LTSEM the electron-bombardment induced conductivity plays a central role for imaging. (A similar effect is often used at room temperature in SEM studies of semiconductors or electrical insulators; see, e.g., Reimer, 1985; Ehrenberg and Gibbons, 1981.)