AML REPORT 3098

SCANNING ELECTRON MICROSCOPY AT THE

ANCIENT MONUMENTS LABORATORY

Carole A Keenax

The usefulness of the scanning electron microscope to archaeology is discussed with examples of work on wood, fibres, insects and eggshells carried out at the Ancient Monuments Laboratory.

ACKNOWL EDGEMENTS

I am grateful to Miss M Girling (AML) for her comments on insects and for supplying plate VII. Plate VIII was taken by Miss J Bond. The scanning electron microscope (SRF) is recognised as a valuable research tool and is widely used in many branches of science (eg. Bassi and Giacobini 1973, Boyde 1972, Chaloner and Collinson 1975, Chen and Schnitzer 1976, Echlin and Gregory 1975, Heywood 1971, Peat and Lloyd 1974, Todd, Cromack and Knutson 1973, Winter 1975). A Jeol JSM-S1 ((which operates at either 4 or 10 Kv) was purchased by The Ancient Monuments Laboratory (AML) in 1974 and has proved to be of value in the examination of archaeological materials.

The object is placed in a vacuum chamber and scanned by an electon beam. This causes the emission of secondary electrons from the atoms near the specimen surface. Some of these are collected in a scintillation detector. The resulting signal is amplified and used to alter the brightness of a spot on a cathode ray tube. The spot scans in synchronisation with the beam on the specimen. The resulting picture resembles the surface of the object magnified many times. Topographic information is obtained mainly because the parts of the specimen nearer to the detector appear brighter (for further details see Hearle, Sparrow and Cross 1972).

The main advantages of the SR⁴ are that better resolution and a very high depth of focus are obtained compared with optical microscopy. This enables a clear, three-dimensional image of the specimen surface to be obtained directly at magnifications of up to 10 000 times or sometimes more. Fairly large objects (up to about 1 cm³) may be examined. Combined with the ability to alter magnification easily, this means that a detailed study may be made of an area while its relation to the entire specimen is known. Sample preparation is generally fairly simple and involves drying and sticking on to a metal stub. Non-conducting materials are usually coated with a thin layer of a conductor such as carbon or metal evaporated under vacuum. Gold-palladium is used routinely in the AML. This helps to prevent the build up of an electrical charge which could cause image anomalies.

1

There are very few archaeological materials which might not be investigated rewardingly with the SEM. The mineral deposits themselves can be examined (eg.Keller 1976, Núñez and Alhonen 1974). Artefacts may be studied to obtain technological information (eg. Tite and Maniatis 1975), and material of biological origin can be examined to aid taxonomic and many other studies (eg.Conolly 1976). Examples of work carried out at the AML are discussed below. Most of this falls into one of two categories that of an investigative nature, and where the SEM is used merely as a convenient method of illustration. Whatever its function in the archaeological context, however, it is important that standards recommended for the larger scientific community (Clark and Glagor 1976) are still applied.

Much of the SEM work at the AML has been on wood. Routine identifications may be carried out by standard optical methods. The SEM has, however, proved to be particularly useful in the study of poorly preserved or altered wood remains.

Mineralised remains, which commonly occur when wood has been buried in contact with a metal object, had previously presented many problems in examination. The material superficially resembles wood but often consists entirely of metal corrosion products (eg. "rust"). It is usually fragile and opaque and cannot be sectioned easily for examination by transmitted light. Exposed surfaces or fractures tend to be uneven and difficult to observe at high magnifications using reflected light. In the SEM the apparent **cells** are often found to be separate tubes with walls composed of corrosion compounds. The structures observed, including bi-convex discs between adjacent "cells" (plate I), suggest that a layer of corrosion products is deposited on the cell wall which later decays away (Keepax 1975a). The discs represent the casts of

AP

2

pit-pairs. Further deposition may fill the remaining cavities.

Preservation of the superficial form of wood by mineral deposits occurs frequently in archaeological deposits when wood is next to iron objects (Keepax 1975a and plate I). Examples have also been observed of replacement by copper (eg. Keepax 1977b and plate II) and lead corrosion products. Copper and lead tend to slow down wood decay, therefore examples are sometimes found with copper or lead corrosion deposits in the cellspaces of preserved wood. This then appears as mineral casts in areas where the wood has partially or completely decayed away (plate III). Replacement by other substances present in the burial environment are also observed; for example, calcium carbonate complexes (Kcepax 1975 and plate IV) or natural iron pan deposits (Keepax 1977).

Merely identifying the type of replaced organic material involved is sometimes difficult. For example, a Roman brass sheet from Colchester (Essex, TL 965-236) had many remnants of organic material on the surface. These consisted mainly of longflat, parallel-sided fragments, cream in colour or stained green, with a maximum width of about 5 mm. This superficially resembled grass. A fragment was examined in the SFM and was found to display structures identical in appearance to corrosion - replaced wood. (Keepax 1977a and plate II). Wood shavings would resemble the remains in size and shape.

An alternative example is provided by some material from Mucking, (Essex, TQ 673 803) which was thought to resemble wood. Examination in the SEM, however, revealed structures camparable to impressions of grass epidermis (Keepax and Keeley 1974 and plate V).

Some archaeological textile samples have also been examined in the SEM: again, it is particularly useful for poorly preserved examples. Use of the SEM enables the entire sample to be quickly scanned and small areas displaying recognisable features studied in detail (plate VI).

3

The CEM has been found useful is the examination of (insect remains. The majority of these may be identified at low magnification but the SEM is useful for general illus trations because of the high depth of focus (Girling 1978 and plate VII). Certain species are difficult to separate, and the SEM is useful for high magnification study of small details such as cuticle ornamentation which aid identification.

ancient

4

The SEM has also been used to investigate the possibility of identifying birds' egg-shell from small fragments found on archaeological sites (Keepax 1977c). It is hoped to use fine details observed in the SEM together with features such as shell thickness to enable tentative identifications to be made. Preliminary examination of archaeological egg-shells has shown that recognisable structures are preserved (plate VIII).

In conclusion, the Ancient Monuments Laboratory has found the SEM to be of considerable use as a tool for the examination of archaeological materials. It should not be seen as an end in itself, but as a means of investigation complementary to other techniques. It is useful both for research and as a method of producing good photographs of small specimens. The SEM is used in these ways by other scientists working in archaeology (eg. Kenward 1978, Krysko and Lehrheuer 1975, Oddy 1975, Pilcher 1968, Werner, Bimson and Meeks 1975) and seems likely to be used even more in the future.

References

BASSI M, GIACOBINI C, 1973. Scanning electron microscopy: a new technique in the study of the microbiology of works of art. <u>International</u> <u>Biodeterioration Bulletin 9</u> No. 3: 57-68

BOYDE A, 1972. Scanning Electron Microscope Studies of Bone, in <u>The</u> <u>Biochemistry and Physiology of Bone</u> Vol I (Bourne G H, ed.) New York: Academic Press: 159-310

CHALONER W G, COLLINSON M E, 1975. Application of SEM to a Sigillarian Impression Fossil. <u>Review of Palaeobotany and Palynology</u> 20: 85-101

CHEN Y, SCHNITZER M, 1976. Scanning Electron Microscopy of a Humic Acid and of a Fulvic Acid and its Metal and Clav Complexes. <u>Soil Science</u> <u>Society of America Journal 40 No.5: 682-686</u>

CLARK J M, GLAGOR S, 1976. Evaluation and Publication of Scanning Electron Micrographs. Science 192 No. 4246:1360-1

CONOLLY A, 1976. Use of the scanning electron microscope for the identification of seeds, with special reference to <u>Saxifraga</u> and <u>Papaver</u>. Folia Quaternaria, Krakón 47: 29-32

ECHLIN P, GREGORY M, 1975. The application of scanning electron microscopy and X-ray microanalysis in the plant sciences: a bibliography. <u>Scanning</u> <u>Electron Microscopy</u> Part II IIT Research Institute, Chicago: 737-66

GIRLING M, 1978. Fossil Insect Assemblages from Difford's I Site, in Somerset Levels Papers 4 (Coles J M, ed) 107-113

HEARLE J W S, SPARROW J T, CROSS P M, 1972. The Use of the Scanning Electron Microscope. Oxford: Pergamon Press HEYMOOD V H, 1971. Sconning Electron Microscopy - Systematic and Evolutionary Applications. London: Academic Press

KEEPAX C A, 1975. Preservation and identification of wooden writing tablets from St Thomas Street, Southwark. Ancient Monuments Laboratory Report No. 1924

KEEPAX C, 1975a. Scanning electron microscopy of wood replaced by iron corrosion products. Journal of Archaeological Science 2: 145-50

KEEPAX C A, 1977. Identification of charcoal and iron-replaced wood; Brenig Valley. Interim report. AML Report No.2190

KEEPAX C A, 1977a. Replaced organic material; Colchester. AML Report No.2204

KEEPAX C A, 1977b. Copper-replaced wood; Brough. AML Report No. 2205

KEEPAX C A, 1977c. Identification of avian egg shell from archaeological sites and the potential use of the scenning electron microscope. AML Report No.2415

KEEPAX C A, KEELEY H C M, 1974. Botanical identifications; Mucking, Thurrock, Essex. AM, Report No.1609

KELLER W D, 1976. Scan Electron Micrographs of Kaolins Collected from Diverse Origins - III. Influence of Parent Material on Flint Clays and Flint-like Clays. Clays and Clay Minerals 24 No.5: 262-264

KENWARD H K, 1978. The Analysis of Archaeological Insect Assemblages: A New Approach. The Archaeology of York AY 19/1 London: CBA

KRYSKO WW, LEHRHEMER R, 1976. Metallurgical investigation of Roman lead pipes from Pompeii. Journal of the Historical Metallurgy Society 10 No 2: 53-63

AΡ

NÚNEZ M, ALHONEN P, 1974. Scanning electron microscopic examination of the depocits covering the southeastern slope of the Onnenvuori hill in Lammi, southern Finland. Bulletin of the Geology Society of Finland 46: 109-16

ODDY W A, 1975. Comparison of Different Methods of Treating Waterlogged Wood as Revealed by Stereoscan Examination and Thoughts on the future of the Conservation of Waterlogged Boats, in <u>Problems of the Conservation of</u> Waterlogged Wood. Maritime Monographs and Reports No.16: 45-49

PEAT C J, LLOYD B J, 1974. Direct Observation of Rock Macerates. <u>Nature</u> 251 No.5473: 295

PHICHER J R, 1968. Some applications of scanning electron microscopy to the study of modern and fossil pollen. Ulster Journal of Archaeology 31: 87-91

TITE M S, MANJATIS Y, 1975. Examination of ancient pottery using the scanning electron microscope. Nature 257 No 5522: 122-3

TODD R L, CROMACK Jr K, KNUTSON R C, 1973. Scanning Electron Microscopy in the Study of Terrestrial Microbial Ecology. <u>Bulletin Ecological Research</u> Commission NFR (Stockholm) 17: 109-118

WERNER A E, BIMSON M, MEEKS N D, 1975. The Use of Replica Techniques and the Scanning Electron Microscope in the Study of Ancient Glass. J. of Glass Studies Vol.XVII: 158-60

WINTER J, 1975. Note on the preparation and mounting of samples of chalk/ glue ground from paintings for scanning electron microscopy. <u>Studies in</u> <u>Conservation 20</u>: 169-173

PLATES

- I. Wood replaced by iron corrosion products (Mucking) showing bordered pit-pair casts. 0000 X
- II. Material from surface of brass sheet (Colchester) identified as replaced wood. 0000 X
- III. Wood near lead (Mucking) with casts of ray cells and cell wall partially surviving. 0000 X
- IV. Calcium carbonate-replaced softwood (Southwark). 0000 X
- V. Possible replaced grass from surface of copper alloy object (Mucking). 0000 X
- VI. Charred textile (Bolton, Lancs) showing interweaving bundles of fibres. 0000 X
- VII. Beetle elytron, Apion aeneum (F.), (Breiddin, Wales). 0000 X
- VIII. Egg-shell (Icklingham, Suffolk): inner surface with partially eroded mammillae. 0000 X



:

PLATE I



PLATE II



PLATE III







PLATE V



PLATE VI



PLATE VII



PLATE VIII