Contact electron micrography for characterization of paper in a transmission electron microscope: a new technique

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Modern electron microscopes are usually fitted with a plate camera system for recording high-resolution electron micrographs. In this paper a simple method of using this plate camera for recording a contact micrograph of thin paper on photographic cut sheet, using accelerated electrons at 80 to 100 kV in a Philips transmission electron microscope has been described and discussed. Results indicate that the scattered electrons through the sheet of paper do record the structural morphology and help in characterization of paper and its quality. In addition to paper technology, this technique may find potential applications in polymer film and solid state industries and several other areas which require characterization of thin specimens.

1. Introduction

X-ray microradiography is the simplest imaging technique in which the specimens of various kinds are exposed to X-rays and the image is subsequently viewed with a high-power optical or electron microscope [1–3]. The importance of monochromatic X-ray wavelength of suitable absorption coefficients was rightly recognized very early [2, 4], however, most of the developments in this field actually found wider use and applications only after new soft X-ray sources and improved photographic emulsions with grain sizes less than 1 μm were made available.

In addition to the use of conventional silver halide emulsions, several other effects, e.g. ionic crystals taking on colours as a result of irradiation [5], induced changes in solubility of some plastic materials as a result of cross-linking or changes in polymer chain length by irradiation [6], and the use of X-ray sensitive materials, have been used to produce images and their subsequent optical and electron optical enlargements [7]. The use of photoreactive dye derivatives [2, 8, 9] and the xerographic process have also been used [10, 11] with considerable success.

Essentially on similar lines, Feder *et al.* [12], in a recent communication, have described the use of X-ray sensitive material (X-ray resist) and a 100 nsec pulse of long wavelength X-rays to produce high-resolution stop-motion images of living human platelets. The major developments in soft X-ray microscopy have also been separately reviewed by Howells *et al.* [13]. However, we have found no reference to the use of electrons for recording a contact micrograph and subsequent analysis.

In this paper we describe the details of a simple technique abstracted earlier [14] for recording a contact electron micrograph of thin paper on photographic cut sheets using accelerated electrons.

2. Materials and methods

2.1. Samples

Standard filter paper sheets in various porosity grades nos 1, 40, 41, 42, 44, bacterial membrane filters and papers of several other kinds such as bamboo-made wrapping paper and draft pad paper sheets, were collected and accurately cut to the size of the photographic cut sheets (8.3 cm × 10.2 cm). Care was taken to see that the specimen paper sheets did not bend or fold during cutting.

2.2. Mounting of specimens

One piece of each specimen paper was held in position over the emulsion side of the photographic cut sheets under safe light in a photographic dark room. The specimen paper along with the photographic cut sheet was slid into the holding groove guide of the cut-sheet holder plate keeping the paper on top. A suitable packing of discarded blank photographic sheet or a sheet of cardboard (cut to a slightly smaller size than

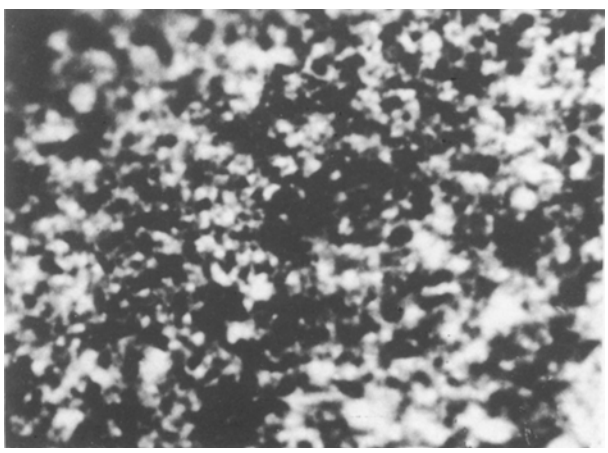

Figure 1 Contact electron micrograph of filter paper no. 1 (× 5)

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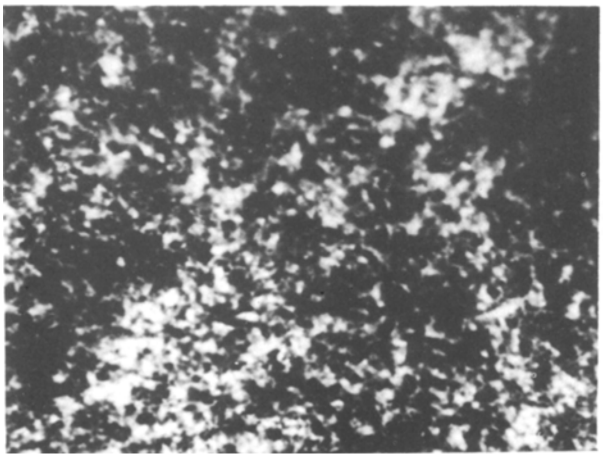

Figure 2 Contact electron micrograph of filter paper no. 40 (× 5)

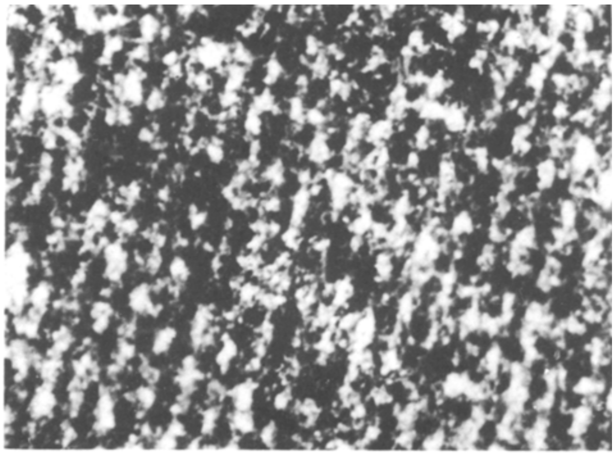

Figure 4 Contact electron micrograph of filter paper no. 42 (× 5)

the photographic cut sheets) was positioned under the recording film to hold its emulsion tightly in contact with the specimen paper. The full capacity of 16 such cut sheet holders of the plate camera were mounted with specimen filter paper sheets of various porosity grades and other papers. A Philips EM-300 transmission electron microscope was used, with acceleration potential set to 80 kV, and the objective lens excited to its minimum current value. The second condenser lens current was so adjusted to have the objective aperture (thin gold foil self-cleaning aperture) enlarged equal to the outer diameter of the circular flourescent screen of the plate camera system. In other words, the divergence of the electrons from the objective aperture was kept lowest for a near normal incidence of electrons on the specimen.

Exposure conditions for every kind of specimen paper were pre-calibrated. Two different exposures for different exposure times on each photographic cut sheet could be recorded for every specimen using the half-masking facility provided with the microscope. Exposure conditions and settings of lens current, once standardized, yielded almost reproducible results during subsequent exposures.

2.3. Scanning electron microscopy

Filter paper of grade nos 1, 40, 41, 42, and 44 were sputter coated with gold and their surface morphology observed in a Cambridge S4/10 scanning electron microscope at 25 kV acceleration potential.

3. Results and discussion

Figs 1 to 7 show the contact electron micrographs from the standard filter papers, bamboo-made wrapping paper and draft pad paper, respectively. Figs 8 and 9 show the scanning electron micrographs of filter papers of only two grades, nos 1 and 44, respectively, recorded without any tilt of the specimen. Table I summarizes the data in respect of these filter papers.

Observation of the filter papers under an optical light microscope with transmitted and reflected light could give no worthwhile details regarding pore size, mesh size used in their manufacture, etc., except visualizing the matrix of cotton fibres in them. However, the surface pore spaces of varying diameters in the filter papers of respective grades could be visualized in their scanning electron micrographs (Figs 8 and 9), the actual effective pore sizes of each grade of filter papers cannot be so easily assessed from them. Moreover, the scanning electron micrographs do not provide, to a paper technologist, any idea about the uniformity of distribution of the paper pulp and the extent of variation in paper thickness. On the other hand, the contact electron micrographs recorded from standard filter paper of various porosity grades (Figs 1 to 7) not only provide an idea about the

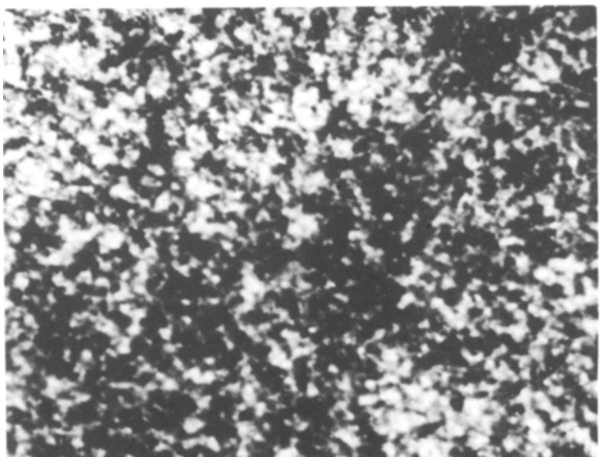

Figure 3 Contact electron micrograph of filter paper no. 41 (× 5)

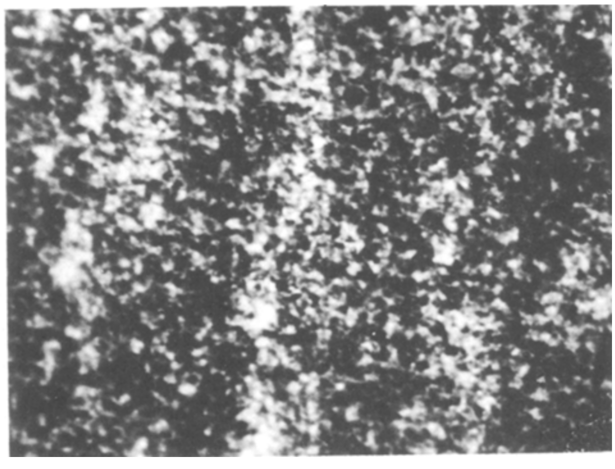

Figure 5 Contact electron micrograph of filter paper no. 44 (× 5)

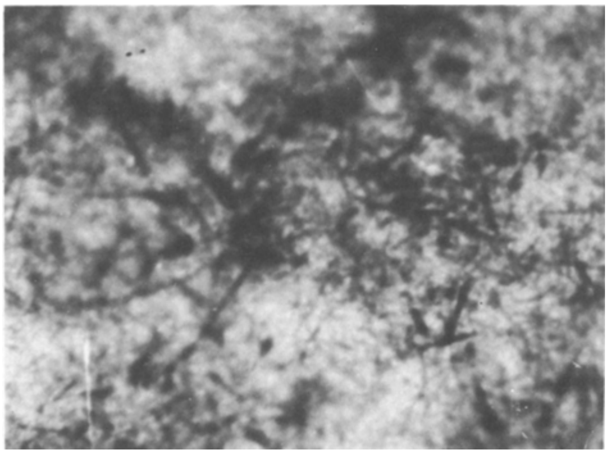

Figure 6 Contact electron micrograph of bamboo made wrapping paper (× 5)

effective uniform distribution/dispersion of the fibrous pulp with dimensions of wire/cloth mesh used during its manufacture (Figs 4, 5, 7) but also provide an estimate of the actual effective pore sizes and their distribution per unit surface area of the paper. The cotton fibres that make up the filter papers are clearly visible in the scanning electron micrographs and these are also distinctly seen from the negatives of contact micrographs with a low-power microscope. The black dots and dotted regions in contact micrographs (Figs 1 to 5) obviously represent the thick matrix of intermeshing fibres while the white dots and dotted regions, represent the actual clear pore spaces distributed within the matrix of the papers, because the electrons record their intensity on the photographic film only after transversing through these clear spaces. The dimensions of these actual clear pore spaces can be easily estimated from these contact electron micrographs and the data in respect of the filter papers is given in Table I, together with the particle size retention and filtration speed data mentioned by the filter paper manufacturers. It can be observed from the table that the slower filter papers, i.e. nos 42 and 44, have wider estimated pore sizes in them compared to the faster filter papers. Even the wire/cloth mesh used during their manufacture is wider for slow filter paper than for the relatively faster one. Obviously the thickness

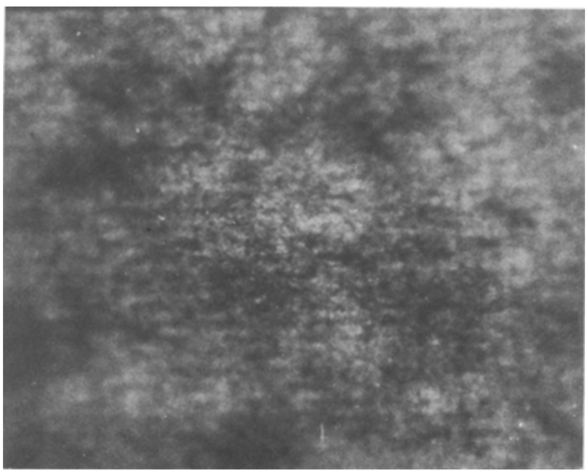

Figure 7 Contact electron micrograph of draft pad paper (× 5)

Figure 8 Scanning electron micrograph of filter paper no. 1 (× 215)

of the layered fibrous pulp and cross-meshing of fibres inside the pores appear to control the ultimate effective pore size and the particle size retention. Closer examination of Figs 1 to 5 would reveal that the surface density of pores per unit area is quite high in fast filter papers, e.g. no 41, compared to the slower filter paper, no. 44. In general it is difficult to resolve the cloth/wire mesh used in cases of paper where the surface density of pores is very high. The bacterial membrane filters did not show or resolve any structural details in their contact electron micrographs.

4. Conclusion

It is concluded that contact electron micrography can be a very useful tool in technical evaluation of paper and its quality in the hands of a paper technologist. It may also be useful in the characterization of thin film materials for their structure and defects. Extension of

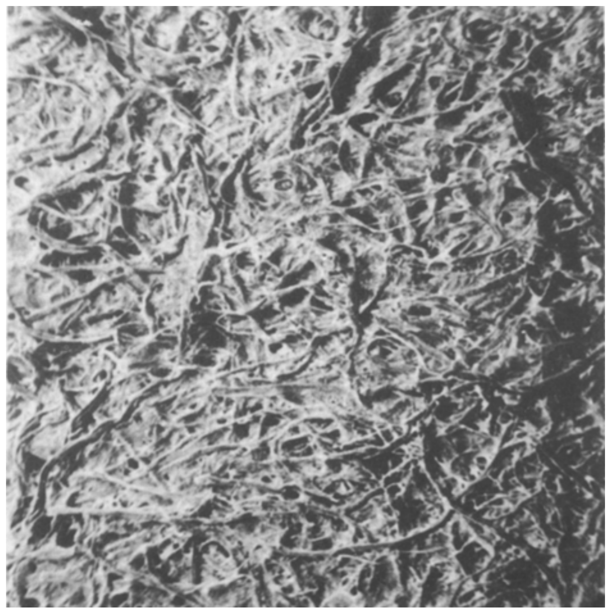

Figure 9 Scanning electron micrograph of filter paper no. 44 (× 215)

TABLE I Data on filter papers of various porosity grades

Sr. no.	Filter paper grade no.	Average pore size estimated (μm)	Filtration speed (*) (sec/100 ml)	Particle size retention (*) (μm)	Estimate of wire/cloth mesh used during manufacture (cm)
1	1	125.4	42.28	11.9	–
2	40	222.2	70.76	7.5	–
3	41	172.4	12.00	17.5	–
4	42	206.0	239.57	3.4	0.076
5	44	168.8	201.85	3.5	0.044

(*) From the data of the manufacturer.

this technique to a wide variety of such thin materials would further reveal the usefulness of this technique and may even pave the way for incorporation of a contact micrography camera system as a standard accessory to a transmission electron microscope.

Acknowledgements

The authors thank the Head, Division of Textile Technology, Indian Institute of Technology, New Delhi for permission to record scanning electron micrographs in their laboratory, the Project Director, Nuclear Research Laboratory, Indian Agricultural Research Institute, for necessary facilities, and Mr V. K. Sharma for his help with photography.

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Received 18 November 1987
and accepted 21 March 1988