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Note

An improved technique for observing the inner surface of open-tubular columns for gas chromatography using scanning electron microscopy

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Attempts to improve the performance of open-tubular columns in gas chromatography by treating the inner surface of the column are considerably aided by observing the surface structure under magnification sufficiently high to resolve structural detail. Scanning electron microscopy (SEM) is most useful in this regard, as has been demonstrated by Alexander and Rutten[1,2] and others[3]. These workers prepared samples for viewing by first cutting or grinding away a portion of the column wall to expose the inner surface. Since the surface is non-conducting, it is coated with layers of carbon and gold to eliminate charging effects[4].

In our studies of whisker-walled open-tubular columns[5], this method of exposing the inner surface of the column tends to damage the surface and to introduce foreign matter, each of which is undesirable. We have attempted to avoid this difficulty by viewing the inner surface directly through the open end of the column. To accomplish this, a short length (approximately 5 mm) of the column is mounted vertically in a hole in a SEM stub and cemented into position. This is then observed through the open end under the SEM at an angle of approximately 30° from the vertical. In the first studies, specimens were vacuum coated in the usual way with carbon and gold layers, but considerable difficulty was experienced in obtaining the required image quality. This is illustrated in Fig. 1, which shows an extreme case of charging of the column surface leading to very poor image quality. It became clear that, since the carbon and gold particles emitted during the vacuum coating travelled in essentially straight lines, they were unable to penetrate the heavily textured inner surface of the column. This problem was overcome by applying a technique described by Pfefferkorn[6], but not commonly used. The column sample is exposed to the vapour of a 0.1 % solution of osmium tetroxide in water for 16–30 h at room temperature. The vapour easily penetrates and coats all parts of the inner surface. This provides a layer that, when further lightly coated with a carbon–gold film, prevents charging and enhances image quality. This latter coating is carried out with the specimen rotating on

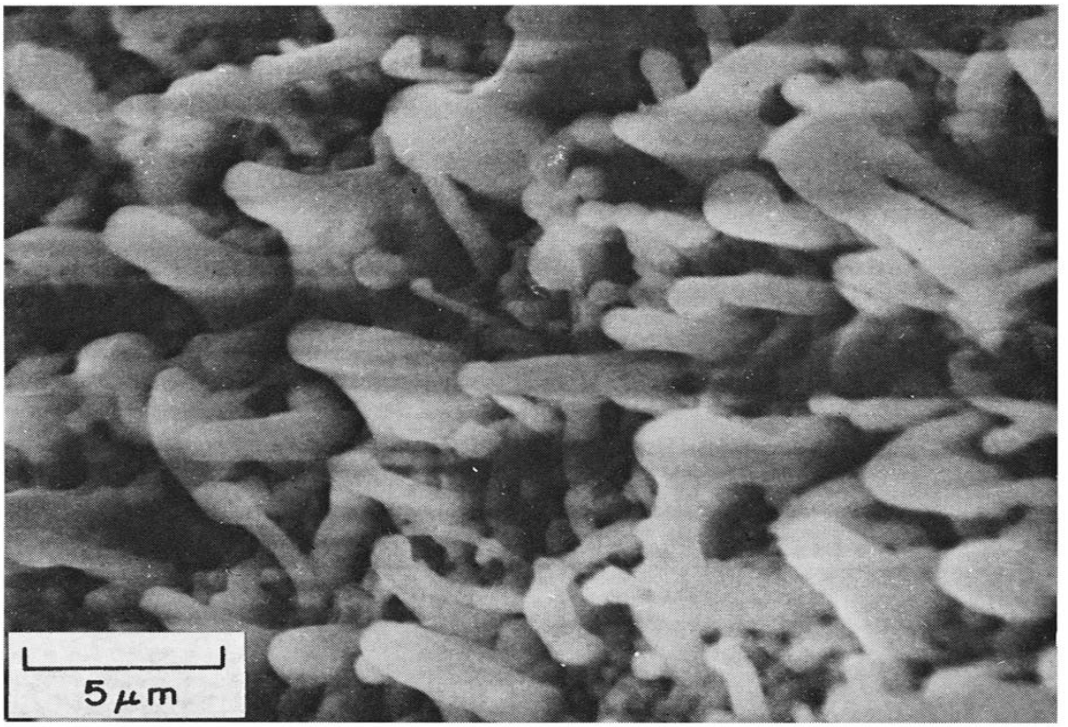

Fig. 1. Whisker-walled glass open-tubular column coated with a layer of carbon and gold showing poor quality imaging due to extreme charging effects.

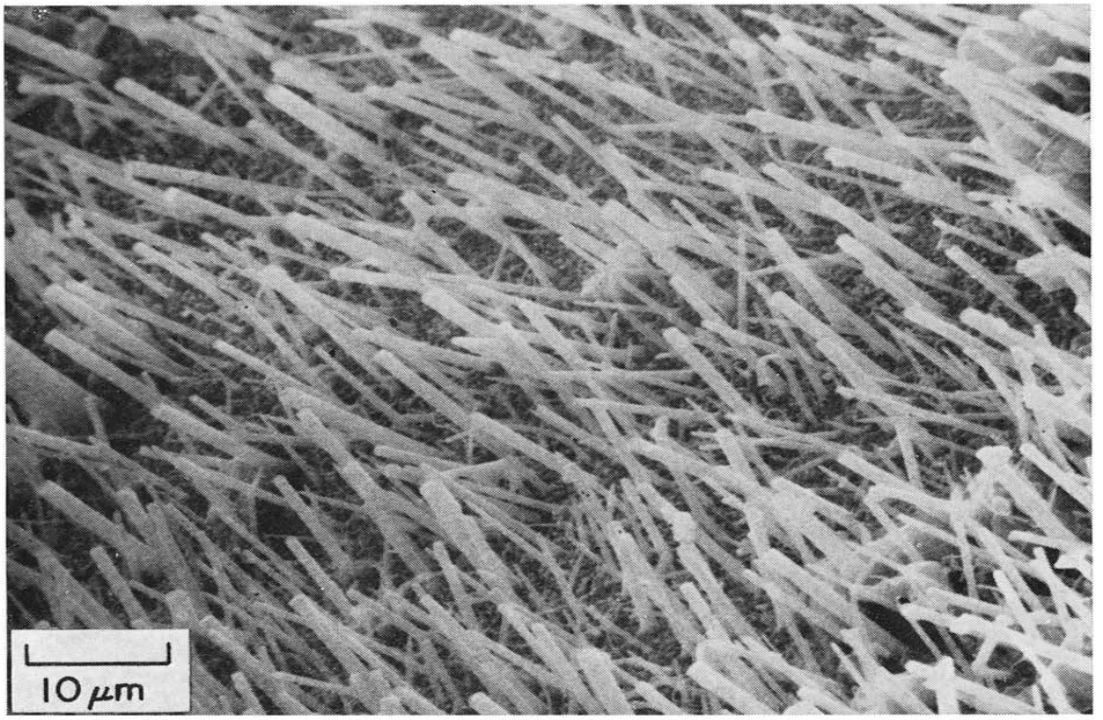

Fig. 2. Whisker-walled glass open-tubular column exposed to osmium tetroxide and coated with layers of carbon and gold.

a planetary rotation device. A micrograph of a typical whisker-walled column is shown in Fig. 2 and demonstrates the quality of image that can easily be obtained by using the osmium tetroxide coating method.

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