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Application of Zeolites for Reducing Hydrocarbon Contamination in Electron Microscopes

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Contamination of objects under an electron beam is one of the principal factors limiting the possibility of achieving high resolution in electron microscopes. In modern electron microscopes the growth rate of the contamination layer is several Å/sec, reaching 10 Å/sec and more when narrow electron beams are used. Usually, 20–30 sec after the beam is switched on, a deterioration in image quality can already be observed visually.

In most high-resolution electron microscopes, measures are taken to protect objects from contamination. Among the many methods of combating contamination ([1]-[4]), the most widespread is the method consisting in surrounding the object with a chamber having narrow diaphragms and cooled with liquid nitrogen ([5]). However, this method too has a number of substantial disadvantages.

These include, above all, the design difficulty of introducing a cooled protective diaphragm into the narrow space bounded by the dimensions of the upper pole-piece tip of the objective lens. During operation the diaphragm rapidly becomes covered with films of oil and ice, which leads to an increase in astigmatism and to twitching of the image caused by the formation of charges on dielectric films. The effectiveness of the protective action of the diaphragm increases as its diameter is decreased; however, the disturbances caused by contamination of the diaphragm itself also increase.

In the present work, zeolites were used to reduce the partial pressure of hydrocarbons in an electron microscope. We selected zeolites from among other sorbents because of their high adsorption capacity at low pressures, relatively high mechanical strength, and ease of regeneration. Zeolite granules were introduced directly into the column of the instrument or into a glass side arm connected to the column near the object chamber. It turned out that this simple device, even without cooling, is capable of producing a large positive effect.

NaX zeolite with a pore size of about 10 Å was selected; this value is sufficient for the absorption not only of the simplest hydrocarbons, but also of large organic molecules, for example products of cracking of the oils used in pumps. Zeolites of this type, produced industrially, have a specific surface area on the order of 1000 m^2/g ([6]).

Fig. 1

Figure 1: Fig. 1

Fig. 2

Figure 2: Fig. 2

The rate of contamination of objects was observed at a current density on the object of 0.2-0.5 A/cm^2 and a beam diameter of 3-4 μ. The growth of the contamination layer was estimated from the rate at which holes in a carbon film became overgrown. The initial rate of contamination formation was 2 Å/sec. Several days after a side arm containing 50 g of zeolite was connected to the column, it fell to 0.25-0.2 Å/sec, i.e., decreased by a factor of 8-10.

The introduction of zeolite into the immediate vicinity of the object (in the form of a ring placed on the specimen holder) led to a decrease in the rate of contamination formation to 0.08-0.04 Å/sec, i.e., by a factor of 25-50 compared with the initial value. Contaminants forming at such a low rate play practically no role.

Figure 1 shows two photographs of a hole in a carbon film, taken on one plate at an interval of 30 min. Before the zeolite was introduced into the column, a hole of the same size, under the same irradiation conditions, became completely overgrown in 5 min. Figure 2 shows successive stages in the overgrowth of a hole as a result of the deposition of a hydrocarbon precipitate before the use of zeolite.

Fig. 1. Reduction in the size of a hole in a carbon film in the presence of zeolite. *a*—initial hole; *b*—the same hole after irradiation for 30 min; beam diameter on the object $d = 4\mu$, $j = 0.2$ a/cm

The positive role of zeolite was also clearly noticeable in reducing the frequency and amplitude of microbreakdowns in the electron gun, usually caused by the formation of oil films on the electrodes (7). Spark breakdowns leading to destruction of the electrode surfaces were completely eliminated.

In some cases, especially at high beam intensity, complete removal of contamination and even partial destruction of the carbon film serving as the object were observed. The maximum rate of film destruction was 0.1 Å/sec. Usually a slight decrease in the thickness of the substrate is useful, since the contrast and brightness of the image increase. As can be seen from Fig. 3, the sharpness of the image of alumina crystallites deposited on a carbon film increased greatly after 20 min of irradiation because of partial destruction of the substrate. Under ordinary conditions the layer of contamination formed under the beam in 20 min makes the object almost completely opaque.

Fig. 2. Reduction of a hole without zeolite. *a*—initial hole; *b*, *c*, *d*—the same hole after 1, 2, and 3 min, respectively; $j = 0.2$ a/cm, $d = 4\mu$

Fig. 3

Figure 3: Fig. 3

Destruction of organic films under the electron beam also occurs when objects are cooled to temperatures below −80°. This phenomenon is explained by the action of molecules of residual gases, primarily water vapor (5).

Thus, the use of zeolites makes it possible, when protecting specimens from contamination, to obtain practically the same results as with the use of a cooled chamber. At the same time, the difficulties listed above in working with a cooled chamber and the need to use liquid nitrogen are eliminated. It is also important that there is no worsening of the destruction caused by contamination of the protective diaphragm and by possible drift of the specimen due to a large temperature difference. The absorbed hydrocarbons prove to be firmly bound to the zeolite and can be removed from it during regeneration.

Fig. 3. Increase in the contrast of the image of aluminum crystals as a result of partial destruction of the carbon film in the presence of zeolite.
a –specimen before irradiation; b –after 20-minute irradiation; $j = 0.5$ A/cm, $d = 4\mu$

Hydrocarbon contaminants are a substantial interference not only in electron microscopes, but also in many other instruments operating with an electron or ion beam. These include, above all, instruments intended for studying the microstructure of matter. The effectiveness of zeolites was tested on an MAR-1 X-ray microanalyzer. Introduction of zeolite granules directly into the column of the instrument led to a significant reduction in contamination and made it possible to obtain reproducible results during repeated beam scanning.

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CITED LITERATURE

1. A. E. Ennos, Brit. J. Appl. Phys., **4**, 101 (1953).
2. S. Leisegang, Proc. Int. Conf. El. Micr., London, 1954.
3. R. Ruhl, Optik, **19**, 122 (1962).
4. H. G. Heide, Naturwiss., **47**, 313 (1960).
5. H. G. Heide, Zs. angew. Phys., **15**, 116 (1963).

6. Ya. V. Mirsky and M. G. Mitrofanov, A. Z. Dorogchinsky, *New Adsorbents—Molecular Sieves*, 1964, p. 21.

7. W. K. Mansfield, R. L. Fortescue, Brit. J. Appl. Phys., **8**, 73 (1957).

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