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Note

Preparation of support grains for transmission electron microscopic studies

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Diatomaceous supports are formed from diatomaceous earth or diatoms. Diatoms possess a very complicated crystalline structure composed mainly of aluminosilicates. Transmission electron microscopic (TEM) studies on the morphology and crystalline structure of supports are possible, but a sample of the particles of the support thinner than 800 Å is required.

Barna[1] proposed an ion milling unit for preparing samples for bulk materials and a layer system for TEM studies. The aim of this work was to employ an ion milling unit for thinning support grains and to obtain suitable samples for TEM studies. The support grains (each kind of support for chromatography can be used) were embedded in the resin and placed in a small holder of diameter 3 mm. After polymerization of the resin the sample was polished on both sides. When the thickness of the sample was 50–70 μm the sample holder was placed in the ion milling equipment. Experiments were carried out several times. The average time of the thinning process was 1–1.5 h. The operating voltage was 8 kV and the ion beam current was 200 μA. The advantage of the direct thinning method is that studies can be carried out on the morphology of the support inside the grain, while the replica method used previously allowed only the topography of the grain surface to be determined[2].

The small particles of diatoms shown in Fig. 1 are agglomerated into larger particles, so the support grain consists of a mass of diatomite fragments fused at the contact point, giving rise to macropores as shown in Fig. 2. Fig. 3 shows micropores originating from the natural undestroyed structure of diatoms, and Fig. 4 a "flat" surface of SiO_2 monocrystals, which often occur in the particles of the support[2]. Other types of monocrystals that were also found in thinned grains are shown in Fig. 5.

Figs. 6, 7 and 8 show the bright field (BF), the dark field (DF) and the diffraction pattern, respectively, at the same location on the support particles. The BF depicts a fine structure of the grain with black intrusions, which, as shown in the diffraction pattern (Fig. 8), possess a crystalline character. The DF shows the distri-

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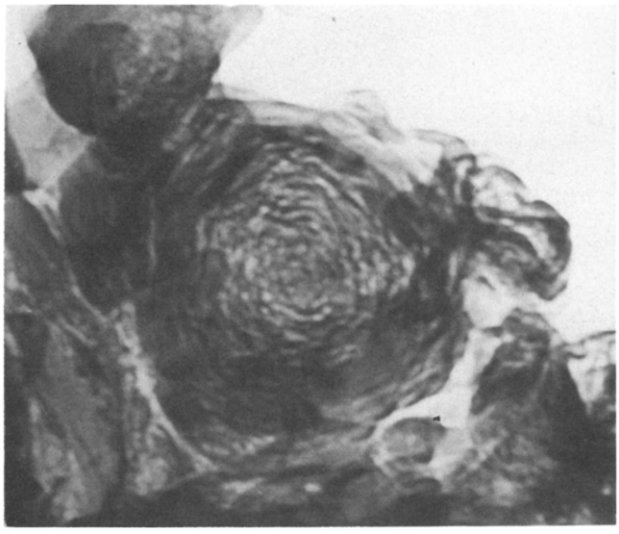

Fig. 1. Interior of support grain, showing that it consists of small particles of diatoms. (TEM, magnification 50 000 ×).

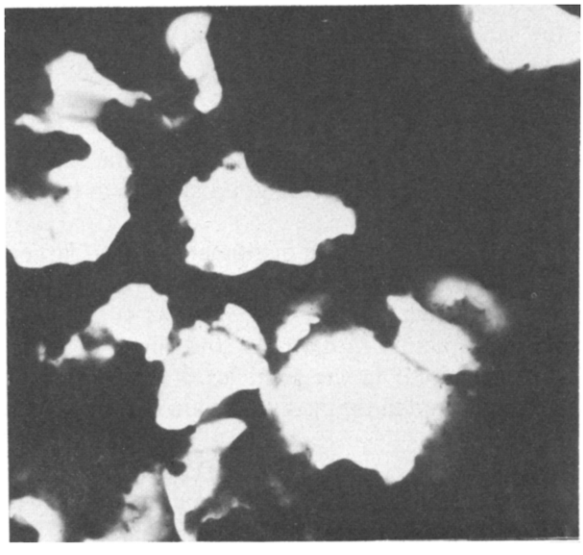

Fig. 2. Macropores in the support grain formed by the production process (TEM, magnification 500 ×).

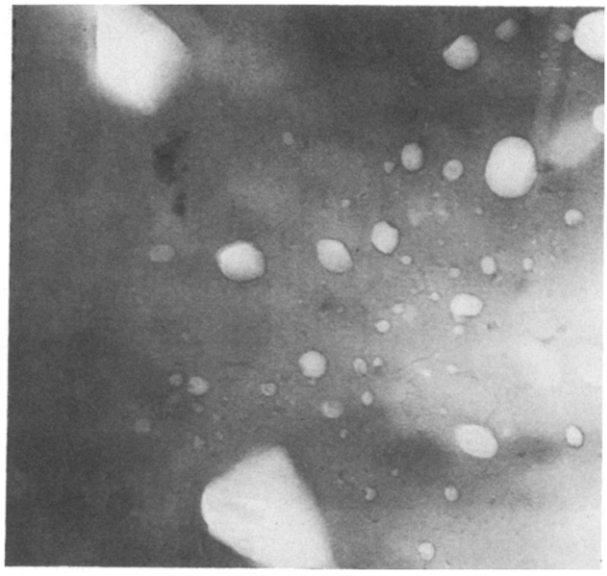

Fig. 3. Micropores originating from natural undestroyed structure of diatom (TEM, magnification 50 000 ×).

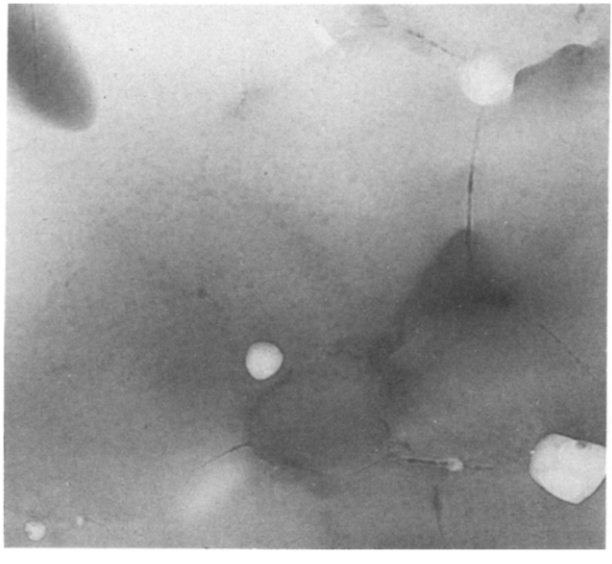

Fig. 4. "Flat" surface of monocrystal of SiO_2 as one of the main components of the support grain (TEM, magnification 50 000 ×).

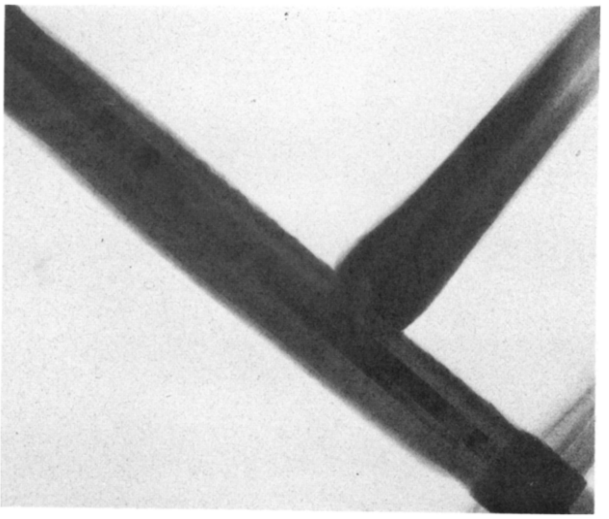

Fig. 5. Small crystallities that often occur in the grain.

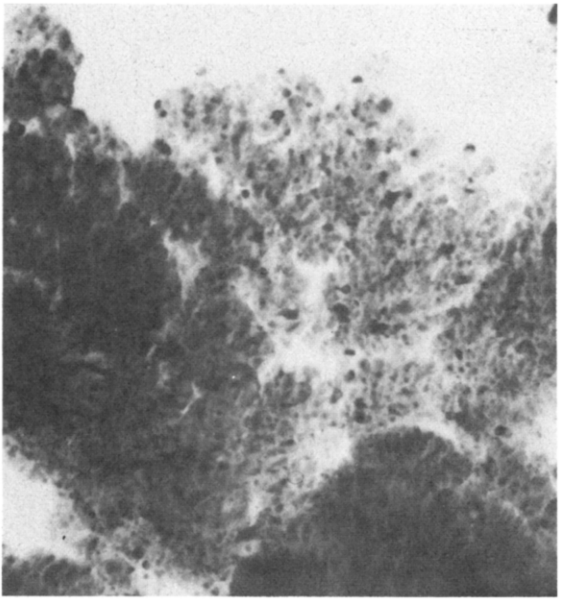

Fig. 6. BF image, depicting the microstructure of the grain with black intrusions (TEM, magnification 50 000 ×).

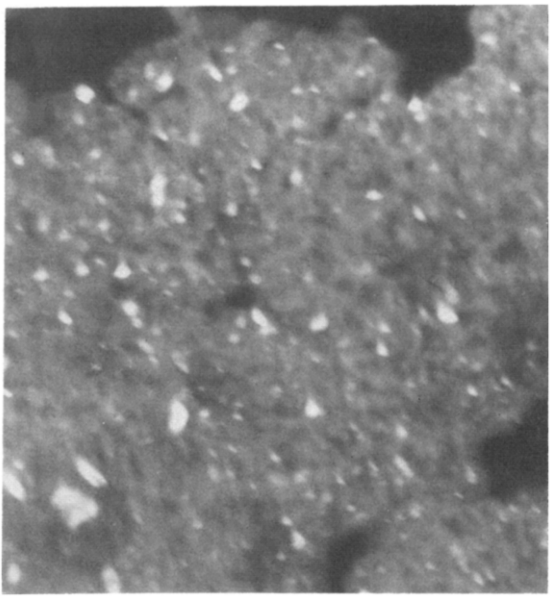

Fig. 7. DF image showing the distribution of crystals in the area represented by the BF.

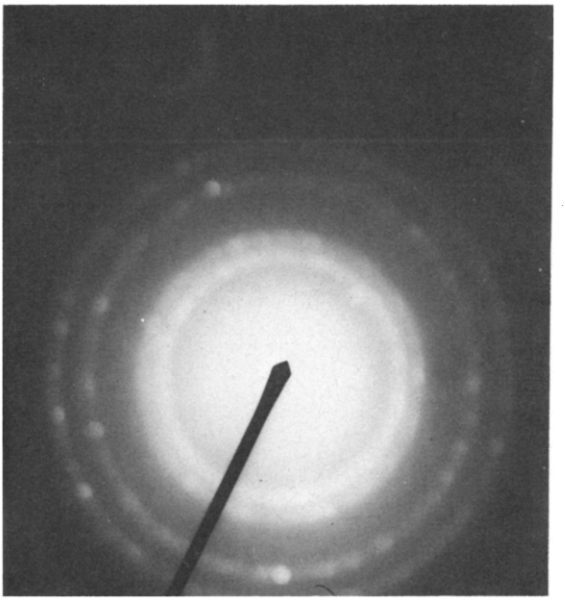

Fig. 8. Diffraction pattern illustrating the crystalline character of the intrusions.

bution of crystals in the area obtained from the reflex marked in the diffraction pattern.

CONCLUSIONS

Employing the ion-etching method for the preparation of support grain samples provides new possibilities for studying these materials by conventional TEM and energy loss spectroscopy, high-resolution electron microscopy and other techniques. By using the method described above, the crystal structure of the support, which undoubtedly influences its chromatographic performance, can be better distinguished.

REFERENCES

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