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An Improved Method for Thin Sectioning of Particulate Catalysts

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Zeolites are microcrystalline alumino-silicates that have commercial application as heterogeneous catalysts or as supports for metal particle catalysts. In both instances their catalytic activity is linked to their porous internal structure: chain-like alumino-silicate supercages separated by open channels with diameters ranging from 0.3 to 0.8nm. At such dimensions the microstructure of zeolites is best analyzed by transmission electron microscopy, requiring thin sections (< 100 nm) of pulverized material.

Although some reports of ultramicrotomed sections have appeared in the literature (Hall, 1984; Hall and Hruskoci, 1983), zeolites are extremely difficult to embed. An embedded block must present a homogenous surface to the knife; the embedding resin must penetrate any open space in the particles, bind firmly to their surfaces, and polymerize to an equivalent hardness. The epoxy resins most commonly used for embedding biological samples (e.g., Araldite, Epon) are quite viscous. They neither infiltrate nor bind zeolites well; most particles "tear" out of the block face when sectioning is attempted.

LR White (London Resin Co., from Pella, Polaron, Fullam, etc.) is a new acrylic embedding resin with several unusual qualities (Murphy and Price, 1983). Its 8 centipoise viscosity is lower than that of any epoxy now in use; if heat-cured, it is a one-component resin requiring no mixing; and unlike the epoxy monomers which are all somewhat mutagenic, it has a very low degree of toxicity. It has two major disadvantages; it won't polymerize well when exposed to oxygen, and it penetrates most polyethelene embedding molds. Most biological tissue cannot be polymerized at elevated temperatures, so the usual embedding protocol used to ensure fully cured blocks is 24 hours polymerization at 60°C, in capped 00 gelatin capsules using a vacuum oven (Murphy and Price, 1983).

The zeolite structure is not harmed by elevated temperatures; oven drying at 400°C is part of its fabrication process. Although LR White tends to become brittle with long exposure to temperature, it can be speed-set at 90°C in one hour with no loss of sectioning quality (Leaffer). Such quick polymerization negates the adverse effect of oxygen, eliminating the need for vacuum embedding. It is also possible to use standard silicone flat embedding molds (Pelco #105 or equivalent), which permit even distribution of the powdered zeolite in the tip of the block.

The zeolite powder was ground in an agate mortar. Small particles were spread in an 1-2 mm area at the tip of flat embedding molds. The molds were filled with LR White "Hard" grade, placed in a 90 °C oven immediately without a room temperature infiltration period, and polymerized for 1-2 hrs. Although the resin sections well with glass knives, the catalyst particles do not; therefore, 100 nm sections were cut with a 55° diamond knife (Sawruk, et al, 1975) (LAB Instruments, Carson City, Nevada) on a Dupont-Sorvall MT-6000 microtome. The blocks consistently produced good, uniform sections (Fig. 1) which were thin enough to permit high resolution imaging (Fig. 2).

This embedding method should prove to be useful for a variety of hard, dry, particulate materials.[1]

ACKNOWLEDGEMENTS

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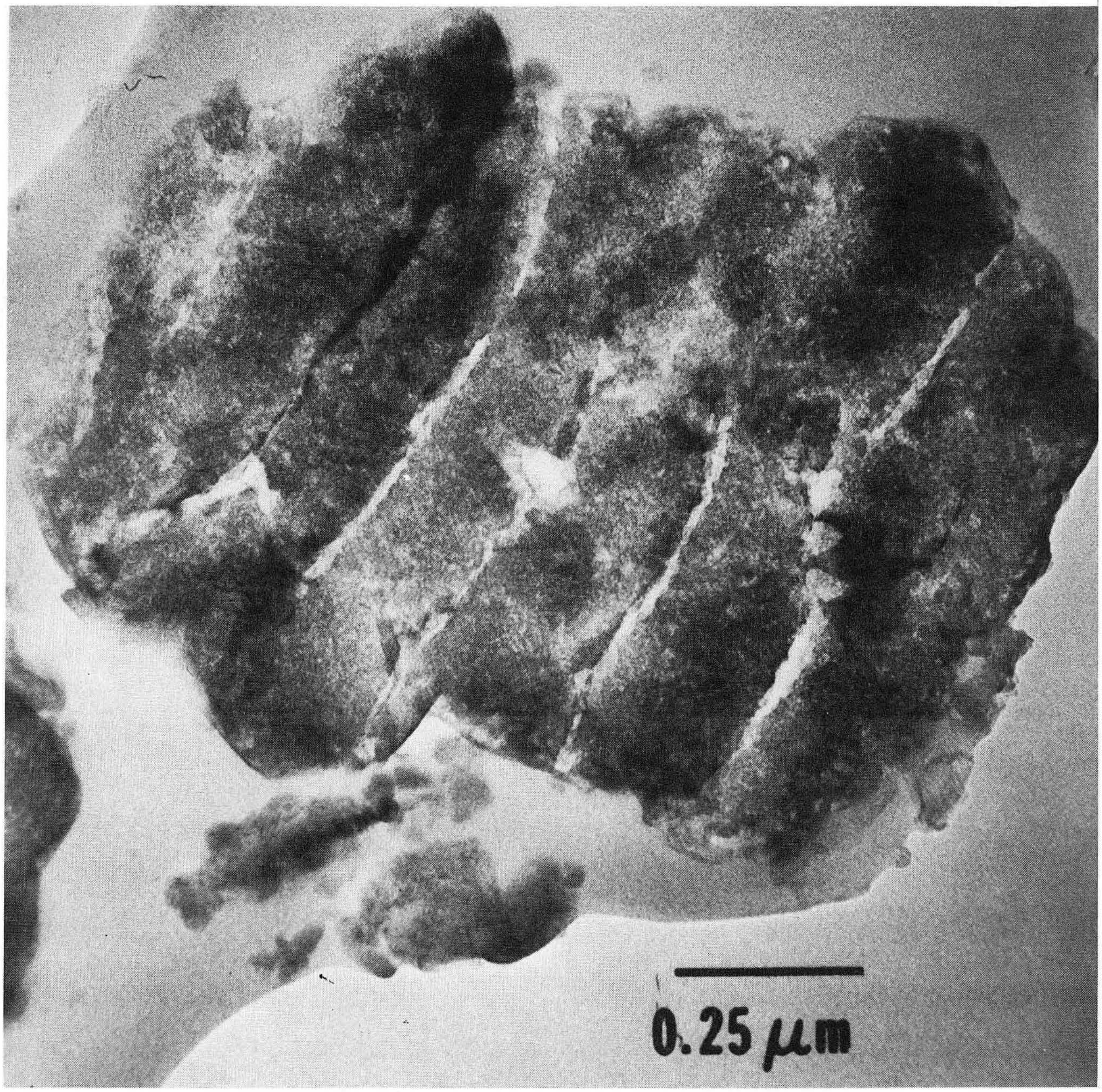

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Fig. 1

FIGURE CAPTIONS

Fig. 1. Low mag-showing several sectioned particles.

Fig. 2. High mag-showing lattice fringes.

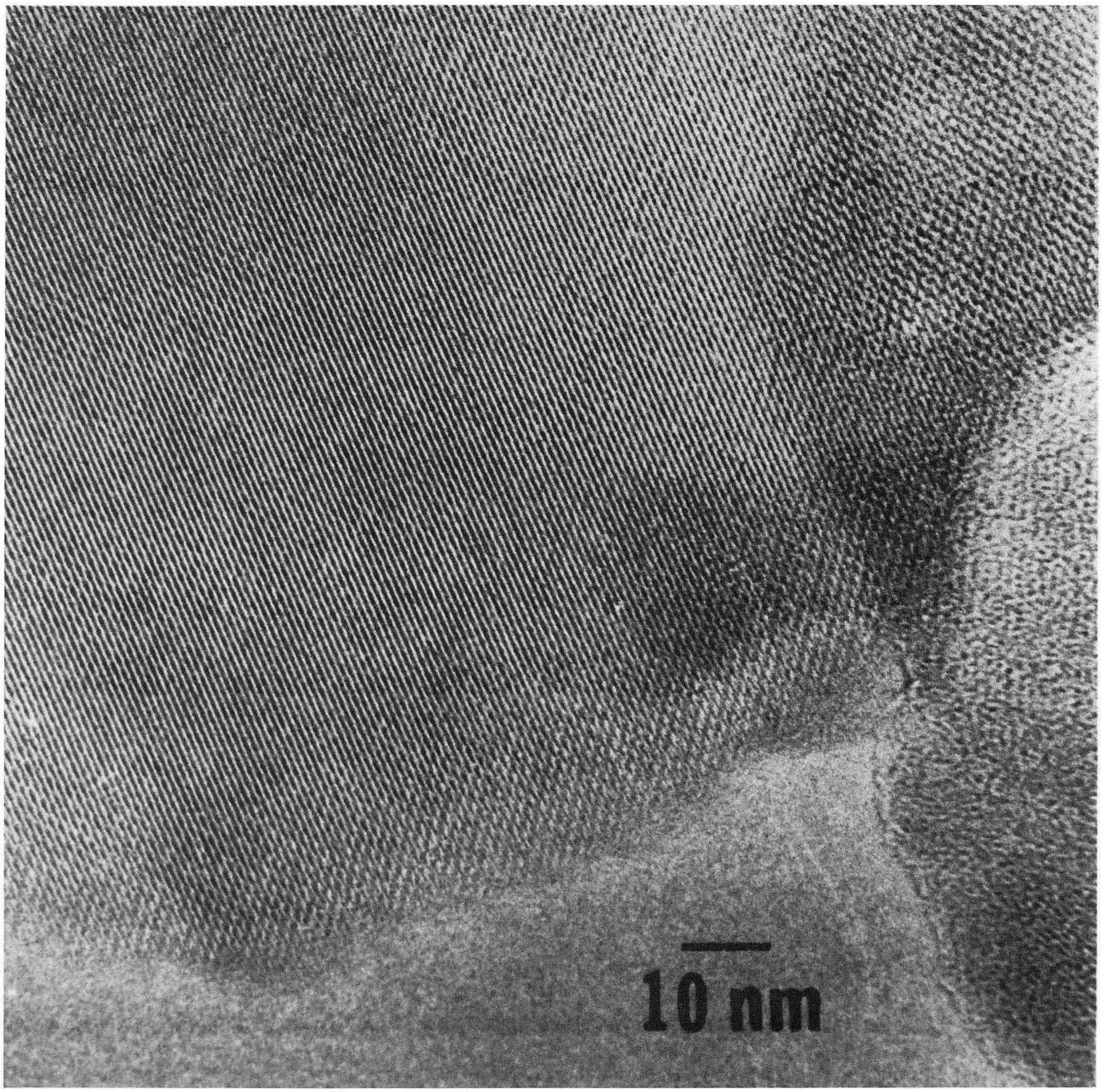

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Fig. 2

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