Ultramicrotomy of Silicate Catalysts Powders

Introduction

Transmission electron microscopy (TEM) plays an important role in revealing the nano-structures of silicates used as catalysts. Ultramicrotomy is the most prominent technique used to produce sample sections thin enough to be imaged by TEM.

The silicates used in catalysts are generally hard materials. Therefore it is best that they be embedded in a hard epoxy resin prior to sectioning with the RMC Boeckeler PowerTome PCZ (PTPCZ) ultramicrotome

It is important that the catalyst bonds firmly to the resin so the particles do not move during sectioning, nor fall out of the sections once collected on water. To that end, a silanizing agent was incorporated in the embedding procedure to promote adhesion of the catalyst powders to the epoxy resin.

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About RMC Boeckeler

Boeckeler Instruments Inc. engineers sample preparation equipment for nanoscale research, under the RMC Boeckeler brand. Their solutions are employed in the advancement of a broad range of areas, with special emphasis in sample preparation for 3D electron microscopy solutions.

Instrumentation

The PowerTome PCZ is equipped with a touchscreen computer and a trinocular microscope with a camera. It can be controlled and programmed with the touchscreen. The camera is useful visualizing the sectioning of the sample, especially when training or collaborating, as well as capturing screenshots for reporting. Furthermore, the built-in measuring tool enables measurement of the sample block face using the camera image displayed on the screen.

Procedure

Two experimental catalyst powders were obtained from a catalyst research laboratory; a lamellar silicate (clay) and a mesoporous organo-silicate. The powders were pre-treated with the silanizing agent (3-glycidoxypropyl) trimethoxy silane in 50:50 H2O:Methanol for 1 hour at room temperature. They were then air-dried on filter paper at room temperature.

The particles were then embedded in 7mm wide flat moulds in a two-step procedure. First, the moulds were half-filled with epoxy resin, which was partially polymerized in an oven at 58°C for 12 hours forming a base.

The catalyst powders were then sparingly spread over this base and the moulds filled with additional resin. Final polymerization was completed at 75°C for 24 hours followed by 3 hours at 80°C producing hard blocks.

The resin used was a low-viscosity hard epoxy mixture (Spurr's) prepared with 1% silanizing agent at a ratio of 100:1. The blocks were carefully trimmed using the RMC Boeckeler PTPCZ ultramicrotome with a diamond trimming tool. The small block faces included only a limited amount of specimen below the surface (Figure 1).

The catalyst particles are seen below the surface at the interface between the first and second layers of resin. A 35° included angle diamond knife was then used for ultrathin sectioning.

To reduce the risk of damage to the diamond knife, sectioning was done slowly and under careful observation until the first particle appeared in a section. Sectioning occurred without damage to the knife and sectioning was continued to capture additional particles in subsequent sections.

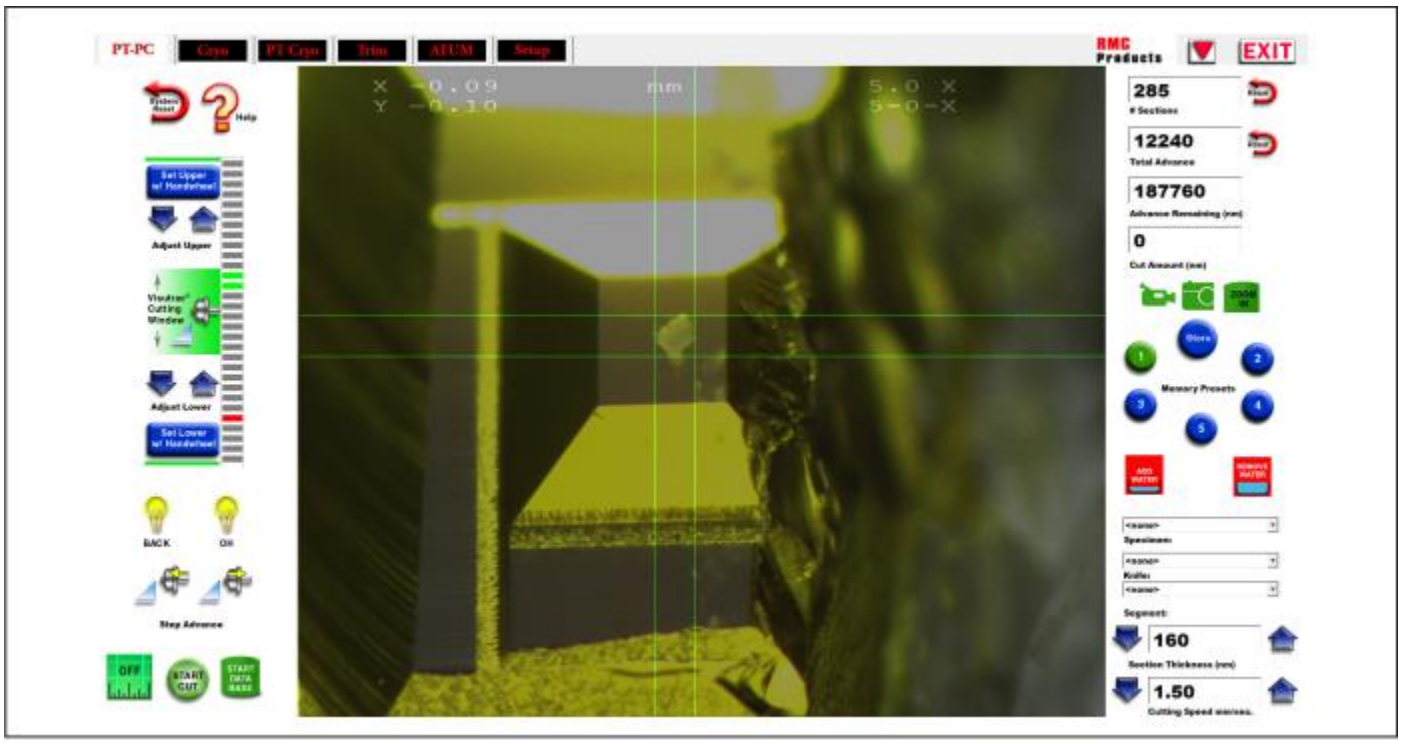

Figure 1: Using the PTPCZ Measuring Tool to measure the size of the clump of lamellar silicate clay particles. The clump of particles measured 0.09 x 0.10 mm. The figure shows the entire control screen of the PTPCZ.

Results

Figure 2 shows the block face of the lamellar silicate powder. The built-in measuring tool of the PTPCZ display was used to measure block size and the size of the particulates. Figures 2 and 3 indicate that both silicate specimens adhere to the resin for successful sectioning. A few holes were apparent in the lamellar silicate clay specimen, where particles had dislodged.

The two-step embedding process, where particles were distributed over the surface of partially polymerized epoxy resin and then covered with more resin, allowed thorough polymerization controlling the distribution of particles during embedding and easy localization at the interface between the two resin layers was successful. It was also possible to locate a single meso-porous organo-silicate particle during trimming, which was then successfully ultramicrotomed.

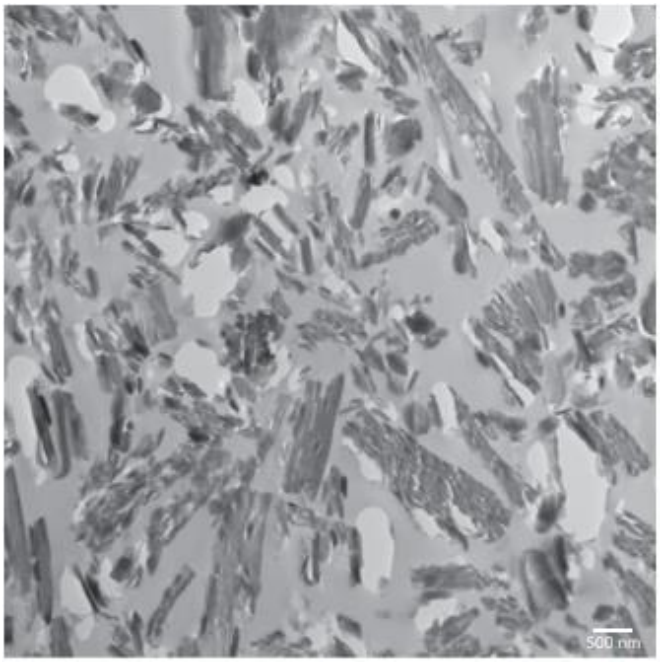

Figure 2 - Particles of the lamellar silicate are well-retained in the resin, although holes can be seen. Magnification = 15,000 x.

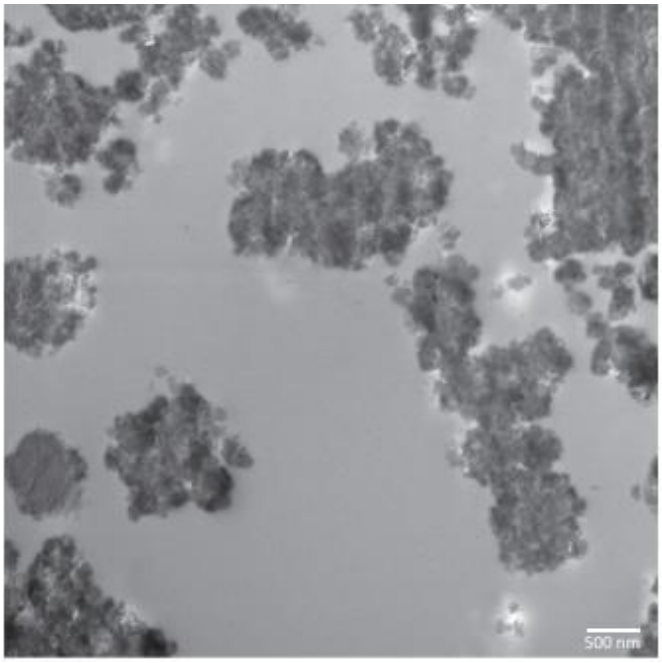

Figure 3 - The meso-porous organo-silicate powder particles adhered well to the resin. Magnification = 20,000 x.

Associated RMC Boeckeler Instruments

PowerTome PCZ

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