

# Crystal Illuminations

A Publication of Sawyer Research Products, Inc. May 2003



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## INTRODUCING NEW MATERIALS

Janet Radwanski

Dear Reader,

Sawyer Research Products has been a leader in the field of hydrothermal growth of cultured quartz crystal for over 45 years. Cultured quartz represents the most commercially successful application of hydrothermal growth technology.

Previous newsletters have included articles on improvements in quartz growth technology that benefit a variety of applications – bulk acoustic wave, surface acoustic wave and optical. This issue discusses our research work on other

hydrothermally grown materials, such as corundum and carbon nanotubes, which have many interesting commercial applications.

If you are attending the joint meeting of the Frequency Control Symposium and European Frequency and Time Forum in Tampa May 5th-8th, please visit us at booth 105. Our latest data sheets and technical briefs as well as a complete set of our engineering newsletters will be available.



## Development of Hydrothermal Production of New Materials

Wojciech L. Suchanek

Hydrothermal technology offers many advantages over conventional and non-conventional synthesis methods of inorganic materials. All forms of inorganic materials can be prepared with the hydrothermal synthesis, namely single crystals, powders, fibers, monolithic ceramic bodies and coatings on metals, polymers, and ceramics. The hydrothermal technology is more environmentally friendly than many other synthesis methods, which can be attributed in part to

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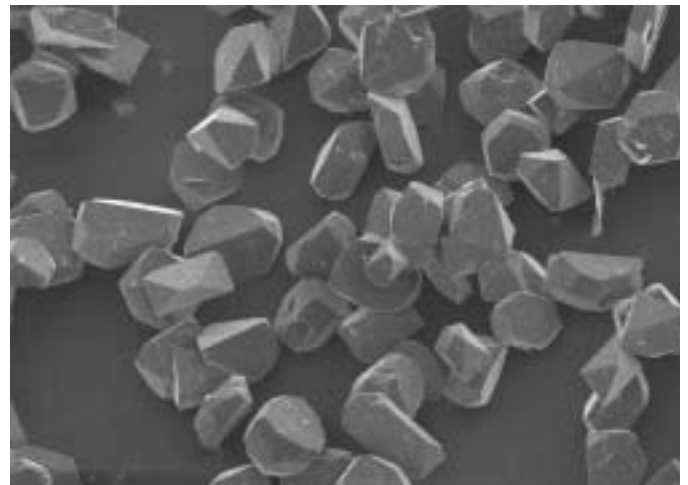
*(Development of Hydrothermal Production of New Materials Continued from p1.)*

energy-saving low processing temperatures, ability to recycle waste, and safe and convenient disposal of waste that cannot be recycled. In addition to lowering the overall cost of the synthesis process, the low reaction temperatures avoid common problems encountered with high-temperature technologies, such as poor stoichiometry control due to volatilization of components, presence of thermally induced defects, formation of undesired phases, and unwanted phase transformations. From the standpoint of ceramic powder production, additional advantages of the hydrothermal method are minimal aggregation levels and excellent control of crystal size and morphology.

So far, one of the best examples of well-established commercial applications of the hydrothermal synthesis is growth of quartz single crystals, with a production rate on the order of 3,000 tons/year. However, the largest potential growth area for commercialization of the hydrothermal technique is ceramic powders production. The widely used Bayer process utilizes hydrothermal methods to dissolve bauxite and subsequently precipitate aluminum hydroxide, which is later heat treated to crystallize corundum. Several companies have established commercial hydrothermal production processes for making dielectric powder compositions for capacitors. Unlike the above process for making corundum or most ceramic powder manufacturing methods, these hydrothermal powders are crystallized as anhydrous oxides directly from solution.

## Hydrothermal Production of Corundum Powders

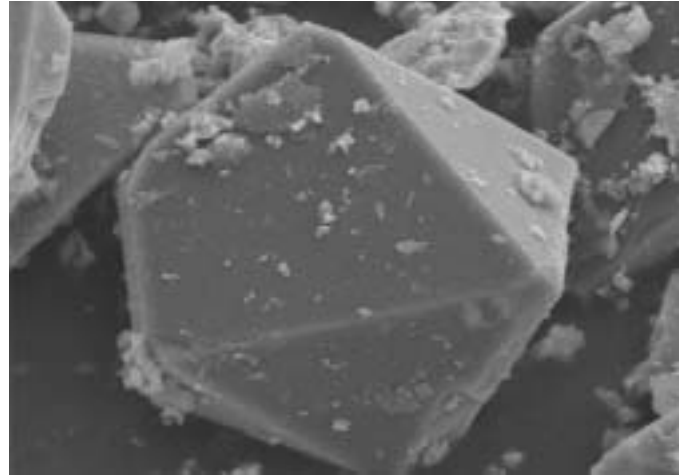
Sawyer Research Products, Inc. recently developed hydrothermal production of inexpensive high-purity corundum powders. This new product line utilizes advantages of the hydrothermal technology in synthesis of ceramic powders and the company's capacity for hydrothermal synthesis. Corundum powders are widely used as abrasive materials, in sintered ceramics (i.e. electric insulators, cutting tools, hip-replacement and dental implants, grinding media, abrasion-resistant tiles, etc.), refractories, and coatings. Corundum (i.e.  $\alpha$ - $\text{Al}_2\text{O}_3$  phase) combines excellent mechanical properties, such as high mechanical strength and hardness, with high chemical resistance, oxidation resistance and refractoriness.



*As-synthesized Corundum Powder (1000x)*

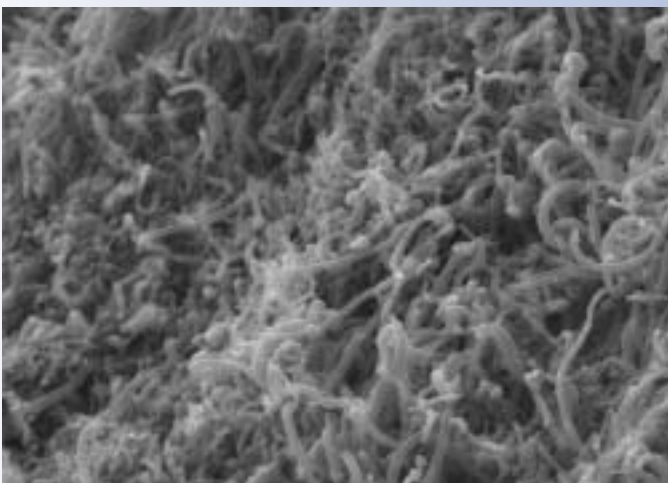
Sawyer's products line of the corundum powders called DiamoCor, comprises both undoped and Mn or Cr-doped compositions with the following grain sizes: 50 $\mu$ , 10 $\mu$ , 3 $\mu$ , 1 $\mu$ , and 300nm. Phase purity of the powders is 100% of the  $\alpha$ - $\text{Al}_2\text{O}_3$  phase. Chemical purity ranges between 99.9% and >99.97% with ultra-high purity materials (99.99-99.999%) being currently developed.

Photographs of corundum powders with 10 $\mu$  grain size are shown. The picture on page 2 (magnification 1,000x) reveals typical as-synthesized powder, with uniform particle size distribution and well-defined crystal faces in each corundum crystal. The morphology of the powders in addition to their phase and chemical composition make DiamoCor corundum very aggressive and long-lasting abrasive material. During industrial-scale lapping tests conducted in our Conroe, TX facility, several fold increase of the materials removal rate was observed (as compared to a typical commercial corundum abrasive with the same grain size). In addition, DiamoCor lapped easily materials which could merely be scratched by a typical commercial corundum abrasive with the same grain size. After prolonged lapping tests, Sawyer's corundum did not sustain any damage (right photograph, magnification 6,000x).



*Corundum Powder After Use In Industrial Lapping (6000x)*

## Hydrothermal Synthesis of Carbon Nanotubes



*50-100 nm Diameter Nanotubes (10,000x)*

Pursuant to Sawyer's involvement in a variety of new products, hydrothermal synthesis of multiwalled carbon nanotubes (MWNTs) was developed. The carbon nanotubes are considered first of all as reinforcing fibers because of high specific strength and high specific stiffness. Measured Young modulus and tensile strength of MWNTs are 1.3-1.8 TPa and 10-60 GPa, respectively, which makes them applicable as an alternative to widely used carbon fibers. Thus the reinforcing applications include asbestos replacements in concrete structures, fabrication of ultralight and strong composites for a variety of applications in automobiles, planes, and military equipment. Another attractive feature of the MWNTs is high level of reversible hydrogen uptake and Li<sup>+</sup> intercalation. Therefore, the MWNTs might find applications as H<sub>2</sub> storage media and in rechargeable lithium batteries. The MWNTs exhibit excellent field emission properties and could be used in flat panel displays. Moreover, the carbon nanotubes can serve as

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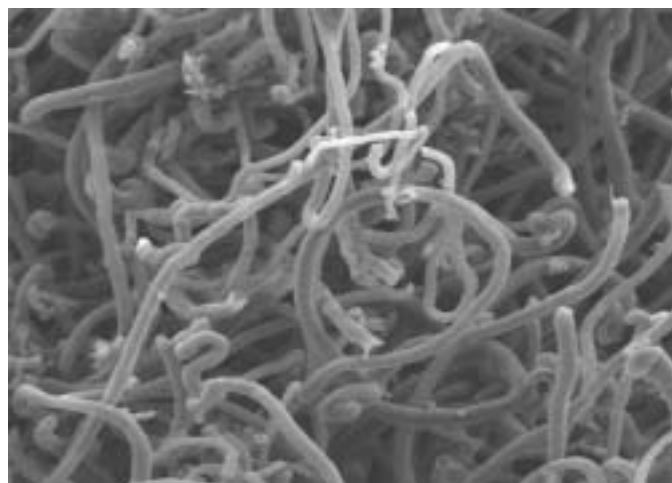
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*(Hydrothermal Synthesis of Carbon.... Continued from p3.)*

templates for other inorganic nanorods/nanowires, and be useful as constituent units for next-generation electronic nanodevices, gas sensors, and in electromechanical actuators.

A variety of sizes of the multiwalled carbon nanotubes with yields approaching 100% can be prepared by properly adjusting synthesis conditions of the hydrothermal treatment of very simple and cheap carbon sources. Nanotubes with diameters 50-100 nm are shown in the photograph on page 3 (bottom left)(magnification 10,000x) and fibers with 200-500 nm diameters are shown in the photograph on the right (magnification 6,000x). This hydrothermal technology gives promise for development of inexpensive mass-production of the carbon nanotubes in the near future.



200-500 nm Diameter Fibers (6000x)