

# Spray-coated Diamond Electrode for Sulfuric Acid Electrolysis

**Takeshi Kondo**

Department of Pure and Applied Chemistry, Faculty of Science and Technology, Tokyo University of Science, 2641 Yamazaki, Noda, Japan  
[t-kondo@rs.tus.ac.jp](mailto:t-kondo@rs.tus.ac.jp)

Boron-doped diamond (BDD) electrodes or diamond electrodes are known to be useful for a durable electrode for electrolysis with a high efficiency base on their wide potential window and physical and chemical stabilities. As a result of electrolysis of concentrated sulfuric acid at diamond electrode, reactive oxidizing species, such as peroxodisulfate, peroxomonosulfate, and hydrogen peroxide, can be generated at a high current efficiency. Such electrolyzed sulfuric acid exhibits strong oxidizing power, and is used for mineralization of organic compounds. In this study, we have developed a new method to fabricate a large-sized diamond electrode that can be used for electrolyzed sulfuric acid production by forming a BDD powder (BDDP)/silica composite film via spray coating.

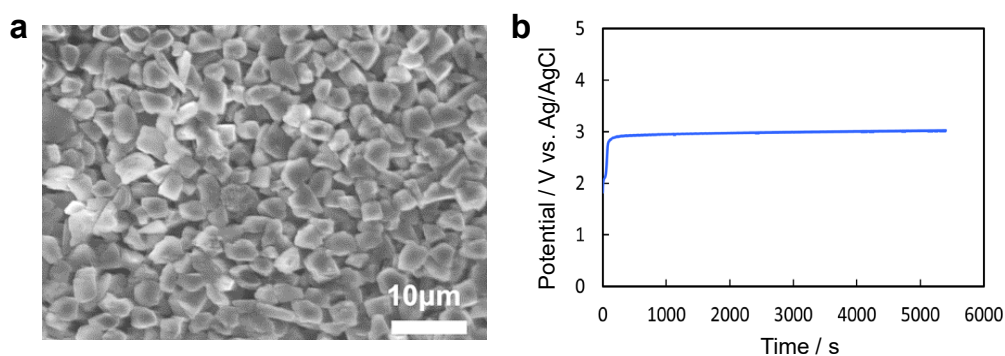
BDDP was prepared by deposition of a BDD layer on the surface of diamond powder with a particle size of 3–6  $\mu\text{m}$  via microwave plasma-assisted CVD [1]. The BDDP was added to tetraethyl orthosilicate/ethanol solution, followed by addition of ultrapure water and nitric acid and stirring to prepare a BDDP/silica sol solution. The BDDP/silica sol solution was spray-coated on a hydrophilic titanium substrate, and after baking at 150  $^{\circ}\text{C}$  for 1 h, a spray-coated diamond electrode consisted of a BDDP/silica layer formed on the substrate was obtained (Fig. 1a).

10 mL of 50% sulfuric acid was electrolyzed at a constant current density of 20  $\text{mA cm}^{-2}$  for 90 min at a spray-coated diamond electrode (electrode area: 0.5  $\text{cm}^2$ ). During the electrolysis, the electrode potential was stable around +3 V vs. Ag/AgCl, indicating that no deterioration of the electrode occurred even at highly positive potentials in concentrated sulfuric acid (Fig. 1b). Current efficiency of reactive oxidizing species formation was calculated to be 41%. These results confirm that the spray-coated diamond electrode can be used for electrolyzed sulfuric acid production. A large-sized spray-coated diamond electrode was also prepared in the same way using a 20 $\times$ 20  $\text{cm}^2$ -sized titanium substrate. Therefore, the spray coating method is expected to be useful for scaling up of a diamond electrode for sulfuric acid electrolysis.

## References

[1] T. Kondo, Chem. Lett., 50 (2021) 733.

## Figures



**Figure 1:** (a) SEM image of spray-coated diamond electrode surface. (b) Electrode potential during electrolysis (20  $\text{mA cm}^{-2}$ ) of 50%  $\text{H}_2\text{SO}_4$  at spray-coated diamond electrode.