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マイクロ波を利用したプラズマ合成シリカ微粒子の生成制御
Production and control of silica nano-powder by microwave inductive coupled plasma (MW-ICP)

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Silica nano-powder have attracted both academic and industrial interests for years because of their advantages of availability in various application fields, such as cosmetics, catalyst supports, biomaterials, and medicinal additives. The characteristics of the nano-power depends on their particle size and shape, which have to be controlled for future various applications. Kogoma et al. has proposed and developed a new silica nano-powder production method using a microwave inductive coupled plasma (MW-ICP) in an atmospheric pressure Ar-O_2 mixture gas [1]. They produced powder particles with the size of 50- 200nm by using chlorotrimethylsilane(CTMS) vapor. We have changed it to less harmful material and synthesis rate of the silica nano-powder is investigated with changing the operation conditions.

Figure 1 shows the schematic of the MW-ICP plasma reactor. A quartz tube (inner diameter : 18mm) is inserted in the microwave cylindrical TE_{011} cavity and cooled by fluorinert oil. Argon (Ar) gas is injected as whirl flow to stabilize the plasma and to cool the tube. Ar-O_2 mixture carrier gas is introduced into the tube via a 9-mm-diam quartz tube, where the argon flow rate is maintained at 1.7 L/min and the oxygen flow rate is controlled in the range of 0.1-0.3 L/min. 2.45 GHz microwave is injected with the power up to 1.0 kW from the waveguide connected to a microwave cavity. It is designed so that the wave field has a maximum at the discharge tube center. Then the plasma is produced only inside the discharge tube due to the presence of the strong wave field there.

Tetramethylorthosilicate (TMOS) is vaporized by using a bubbling system and mixed with the Ar-O_2 mixture carrier gas. TMOS is dissociated and oxidized immediately by the high temperature plasma region according to the following reaction,

$$Si(OCH_3)_4 + 6O_2 \rightarrow SiO_2 + 4CO_2 + 6H_2O. \quad (1)$$

The silica nano-powder is synthesized downstream of the plasma production region.

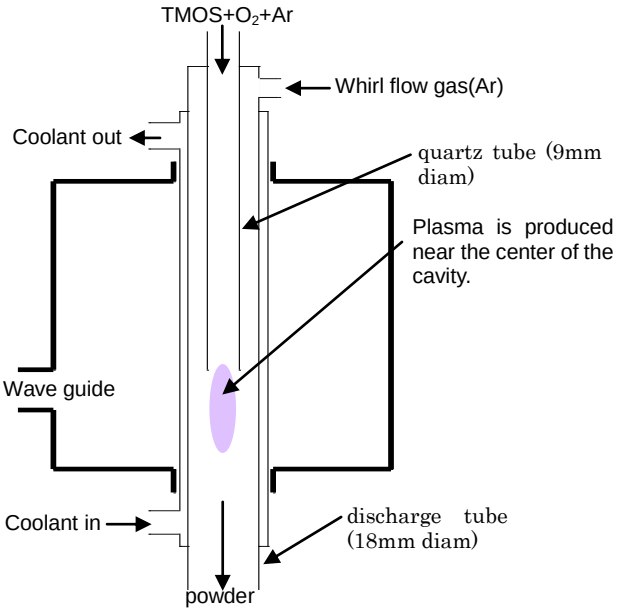

Fig. 1. Schematic diagram of the microwave cavity plasma reactor.

Synthesis rate is measured as a function of the oxygen flow rate, where the rate is defined as the mass of the nano-powder normalized by the TMOS mass used for the synthesis. The maximum synthesis rate of ~50 percent is obtained at the flow rate of ~0.25 L/min. The particle diameter and structure are also investigated by using both a scanning electron microscope and a tunneling electron microscope. These images suggest that the diameter distribution of the nano particles are presently in the range of 20-50 nm. The detailed results will be presented.

Reference

[1] M. Kogoma, *et al.* :" Silica nano-powder production by MW plasma using TE011 mode cavity" proceedings of ISPC 20 (2012).