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GENERATION OF GOLD NANOPARTICLES BY LASER ABLATION IN SUPERCRITICAL CO₂

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1. Introduction

Gold nanoparticles and nanoclusters have been used as materials for surface-enhanced Raman scattering, the building blocks of nanostructures, catalysts for CO oxidation, cancer diagnostics and therapies. Recently, gold nanoparticles of well-defined size and shape have been synthesized by chemical reduction of gold ions in solution. This chemical method, however, can cause contamination from excess reagents such as residual reductants, surfactants, or ions and can lead to functionalization of gold nanoparticles with many kinds of molecules. As an alternative method, laser ablation has been used for the synthesis of gold nanoparticles over the last few decades. Laser ablation is usually conducted either in a vacuum or in ambient condition. Some studies, however, use a different ambient medium such as a gas, a surfactant solution, organic solvent, and liquid N₂. Supercritical CO₂ has been widely applied in a variety of processes including extraction and separation, polymerization and coating. Recently, it has been found that supercritical fluids such as supercritical CO₂ and supercritical water produce interesting reaction fields for nanotechnology and plasma technology.

In this work, laser ablation of gold substrate in SCCO₂ was conducted to generate synthesized gold nanoparticles. The effect of ablation environment, CO₂ density and irradiation time on the irradiated gold plate was investigated. The surface morphology of irradiated gold plate and generated gold nanoparticles were observed.

2. Experimental Method

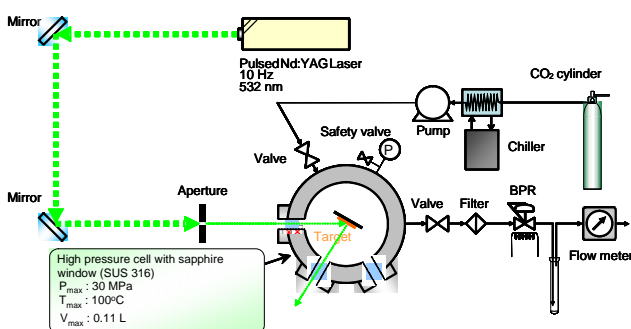


Figure 1 Experimental apparatus of laser ablation with a high pressure cell

Laser ablation was carried out in a high-pressure cell with three sapphire windows. **Figure 1** shows the schematic diagram of the experimental apparatus. The second harmonic of a Q-switched pulsed Nd:YAG laser (Spectra-Physics Quanta-Ray INDI-40-10, wavelength: 532 nm, pulse energy: maximum 200 mJ/(cm².pulse), pulse duration: 8 ns, repetition frequency: 10 Hz) was used. Target was fixed in a high-pressure cell (AKICO). Incident angle of the laser beam was 30° and the laser

was located 1 m from the target. A gold plate (Nilaco, purity: 99.96%, thickness: 0.03 and 1 mm) was used as the target. Liquid CO₂ was pressurized and pumped into the cell using a high-performance liquid chromatography (HPLC) pump (Jasco PU-1586). A glass slide was placed in parallel with the target to collect gold nanoparticles. Laser ablation was conducted at temperature of 40-60°C, pressure of 0.2 to 20 MPa for 1000 to 3000 s. The laser beam was collimated by a 1-mm-diameter of aperture without any focusing lens. Surface morphology of the target after irradiation and the crater depth after PLA were observed by laser scanning microscopy (Keyence, VK-9510). Absorption spectrum and visualization of gold nanoparticles collected in the glass slide were evaluated by UV-Vis spectrophotometer (Jasco, V-550) and digital microscope (VHX-1000).

4. Result and Discussion

Based on surface morphology analysis, a circle crater was clearly observed in the irradiated gold plate at all conditions. The crater was more clearly generated as increasing pressure. However, when 0.03 mm of gold thickness was used, the hole was generated at 8 and 10 MPa of laser ablation in SCCO₂. The increasing pressure of CO₂ caused increasing CO₂ density, on the other hand constant volume heat capacity of CO₂ decreases from 10 to 20 MPa.

Figure 2 shows the crater depth generated by laser ablation with constant volume heat capacity of CO₂ at various pressures. Gold with 1 mm of thickness was used at 8 and 10 MPa to understand the exact depth of crater. As expected, the increasing pressure causes increasing the crater depth due to CO₂ properties change in the system.

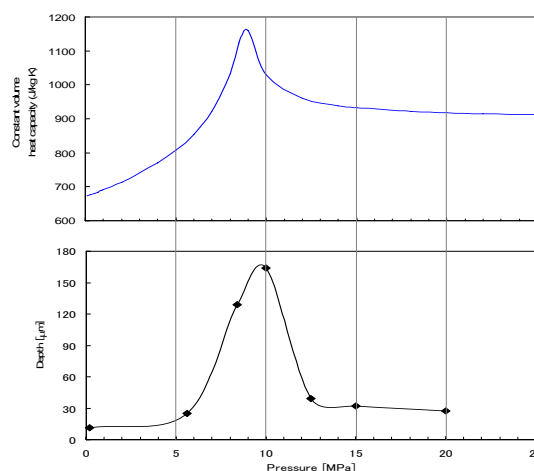


Figure 2 Depth of crater generated by laser ablation at 40°C and various CO₂ pressure

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