Program for Advancing Strategic International Networks to Accelerate the Circulation of Talented Researchers
Japan-ASEAN Collaboration Research Program on Innovative Humanosphere in Southeast Asia:
In search of Wisdom toward Compatibility Growth and Community in the World

Effect of milling temperature to the state of milled $(CuO)_{0.5}$-$(CeO_2)_{0.5}$ Powder

Period of inviting: December, 10th to February, 8th
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Mechanical milling is a solid-state powder processing technique due to repeated welding, fracturing and re-welding of powders in a high-energy ball milling. This technique has now been shown to be capable of synthesizing equilibrium and non-equilibrium phases. Recently, this technique has been extended to a method by which metals and alloys can be synthesized directly by chemical reduction reaction. Some of the important parameters that have an effect on reduction reaction of the powder were milling container, milling speed, milling time, ball-to-powder weight ratio, milling atmosphere and milling temperature. In this report, we study Effect of milling temperature to the state of milled $(CuO)_{0.5}$-$(CeO_2)_{0.5}$ Powder.

Milling parameters such as the milling time and water cooled media were determined to be suitable for the reduction of CuO powders. Monoclinic CuO (Nilaco Corporation, <250 μm, 99.999% purity) and cubic CeO_2 (Kojundo Chemical, <180 μm, 99.99% purity) powders were used as staring materials. High energy vibratory ball milling (Super-Misuni, Nissin Giken Co. Ltd.) was employed with the rotational speed of 710 rpm, where the milling atmosphere was ambient. The powders and zirconia balls (ϕ10 mm) were charged in a stainless-steel vial (ϕ100 mm), where the ball-to-powder weight ratio and the milling time were 18:1 and 14h respectively. The temperature of milling was done by either dripping cooled water on the milling container to lower the temperature or electrically heating the milling vial to increase the temperature of milling (fig 1), the milling temperature were change from 10 to 45°C. The powders after milling were characterized by X-ray Diffraction.

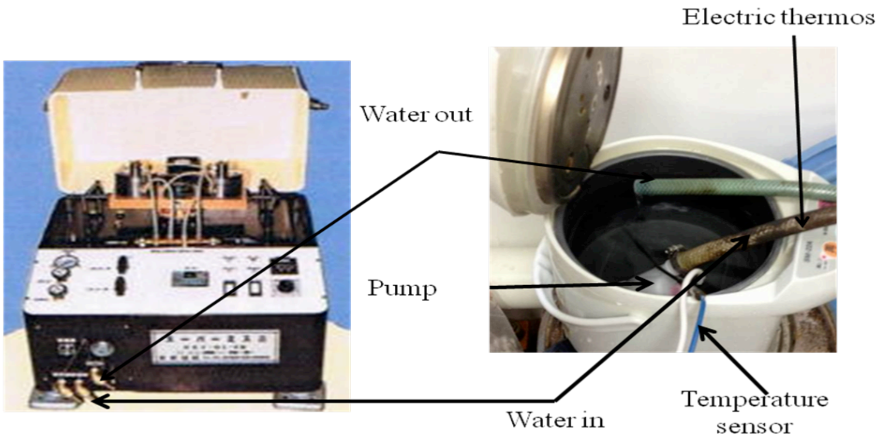

Fig 1.Experiment diagram

XRD patterns of $(CuO)_{0.5}$-$(CeO_2)_{0.5}$ 14h-milled powders under difference temperatures are shown in Fig 1. At low temperature of 10°C, only CuO peaks were observed. At temperature of 25°C, CuO was reduced to Cu_2O and Cu, where there were the peaks of Cu and Cu_2O (Fig 2). At higher milling temperature, only CuO peaks were observed. This showed that the collision energy could be different under different milling temperature. It has been reported that when the milling temperature increased, the greater efficiency of solid-solid collision and the reaction kinetics were enhanced. At low milling temperature, the collision energy was not high enough, the reactions cannot occur. When milling temperature increased, the collision energy enhanced, the reduction $CuO \rightarrow Cu_2O \rightarrow Cu$ was observed. However, when the milling temperature became higher, where the conversion of heating energy into chemical energy was utilized to bring about chemical reactions, Cu and Cu_2O could react easy oxygen in ambient air to compose CuO, it was reason that Cu and Cu_2O peaks disappeared completely when the milling temperature increased up to 35^{o}C. The state of milled $(CuO)_x$-$(CeO_2)_{1-x}$ under other milling temperatures will be study future research.

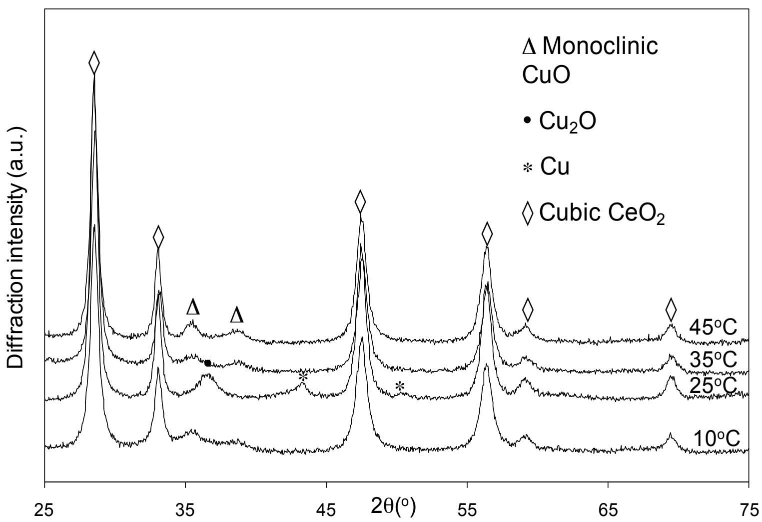

Fig.2. XRD patterns of $(CuO)_{0.5}$-$(CeO_2)_{0.5}$ 14h-milled powder with milling temperatures