Preparation of Copper Nitride Nanoparticles in Long-chain Alcohol and Its Thermal Decomposition Property

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Abstract

We are attending copper nitride (Cu_3N) as a precursor of conducting material for printed electronics device. In this presentation, we demonstrate that Cu_3N particles were prepared in a long-chain alcohol with ammonia gas bubbling and characterized by XRD and TEM. Additionally, thermally decompose property of prepared and purchased Cu_3N were compared by TG-DTA measurement.

1. Introduction

Metal nitride has chemical and physical properties which metal oxide is unable to achieve. As the characteristic properties of metal nitride, semiconducting and magnetic properties are included.[1] Gallium nitride is well known as a material for blue LED. For a hard magnetic material, Ion nitride is a candidate of replacing material from rare earth metal such as neodymium and samarium.[2]

Copper nitride has not been used as oxidation-resistant of copper but also researched as a candidate of a recording material or a catalyst. As new application of copper nitride, we have focused on an electrical conducting material for printed electronics device because of its oxidation resistant property and thermal decomposition property which copper nitride decompose into copper and nitrogen at approximately 300 °C as bulk state.[3] Additionally, by combination with nano-sized effect such as decreasing melting point, it is able to decrease decomposition temperature of the copper nitride. Therefore, an electro conducting ink having oxidation-resistant and sintering around 200 °C will be developed.

Nano-sized copper nitride was prepared by solvothermal or high temperature condition over 250 °C, previously. Choi et al.[4] reported that copper nitride prepared from $CuCl_2$ and NaN_3 as reagents in toluene or THF as solvents in an autoclave. Wu et al.[5] and Wang et al.[6] reported that mono dispersed nano Cu_3N particles were prepared from $Cu(NO_3)_2$ in octadecylamine by heating at 280 °C.

In this presentation, we present new preparation method of nano-sized copper nitride without pressurized or high-temperature condition. Copper nitride was prepared in an alcohol solvent with ammonia bubbling at 190 °C heating. Obtained Cu_3N powder was characterized by XRD and TEM. Moreover, thermal decomposition property was compared between prepared and purchased copper nitride by TG-DTA measurement.

2. Experimental

Copper (II) acetate (400 mg) was dispersed in 1-nonanol (100 mL) in a three-neck flask. The solution was heated with ammonia bubbling at 190 °C by using an oil bath. At the time of heating, the color of solution changed from clear deep-blue to opacity red-brown. Red-brown powder was obtained by centrifugation of the heated solution.

The obtained powder was characterized by XRD and TEM. Thermal decomposition property of the powder was measured by TG-DTA.

3. Results

Crystal structure of the obtained powder was determined by XRD measurement (fig. 1). From observed peaks at 23.40, 33.34, 41.12, 47.84, 53.92, and 59.55° attributed (100), (110), (111), (200), (210), and (211), respectively, the crystal structure was determined Cu_3N having anti-ReO_3 structure (JCPDS No. 47-1088). No peaks deriving from Cu and CuO were observed.

Morphology and size of Cu_3N powder was observed by using a TEM (fig. 2). Obtained Cu_3N powder has shape of granular particle with less than 200 nm of diameter consisted by angular Cu_3N nano crystalline with less than 20 nm on a side. These small particles distributed in an observing view of TEM.

Thermal decomposition properties of Cu_3N nanoparticles and purchased Cu_3N (Santa Cruz Biotechnology, Inc.) under argon atmosphere were compared by TG-DTA (fig. 3). For the prepared Cu_3N, weight lost until at 221 °C. Over the temperature, the weight of sample increased after an exothermic peak with a peak top at 221 °C. For purchased Cu_3N, the weight lost until 439 °C. Over the temperature, the weight of the sample increased with an exothermic peak with a peak top at 439 °C. From these comparisons, decomposition temperature of prepared Cu_3N decreased over 200 °C compared with purchased one.

Crystal structure of powder heated by TG-DTA at 400 °C under argon atmosphere was characterized by XRD. Heated powder gave a diffraction pattern of CuO. Nevertheless powder was heated under argon atmosphere, no diffraction pattern Cu give from Cu_3N. This time, we cannot trace decomposition of Cu_3N to Cu and N_2 which was published literatures previously.[3]

4. Summary

Cu_3N nanoparticles having less than 200 nm of second particle diameter consisted first particle diameter having less than 20 nm on a side were prepared from copper acetate and ammonia as reagents in a long-chain alcohol as a solvent. Decomposition temperature of the prepared Cu_3N was decreased over 200 °C comparing with purchased Cu_3N.

References

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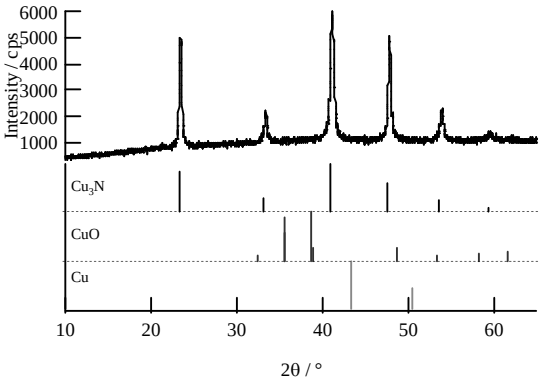

Fig. 1. XRD pattern of obtained sample.

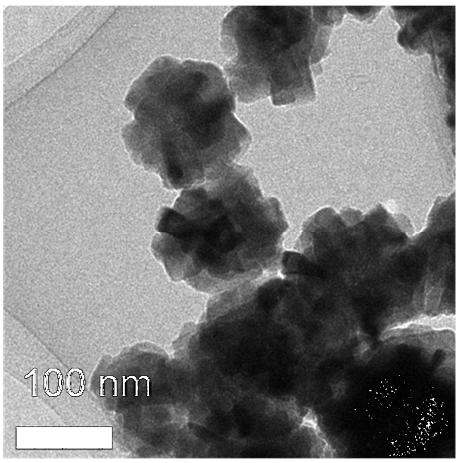

Fig. 2. TEM image of obtained sample.

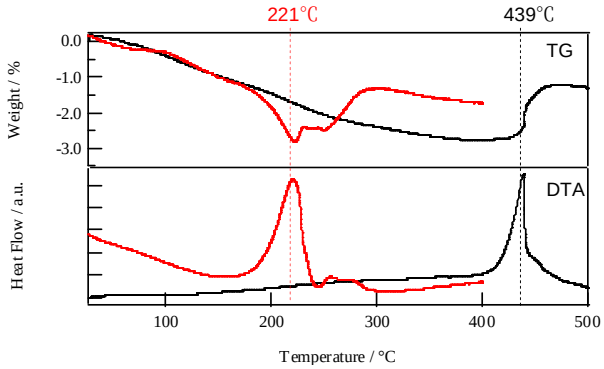

Fig. 3. TG-DTA curves of prepared (red) and purchased Cu_3N (black).