Luminescent properties of amorphous Al_2O_3 prepared by sol–gel method

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Aluminum oxide has been well known as the host matrix for the luminescent materials doped by luminescent centers such as Eu or Ce because of the low cost and the ease of forming. In this work, a fluorescent characteristic of the solid alumina was examined using low cost sol–gel technique. As a result, amorphous alumina sintered at 500°C generated visible luminescence at~430 nm, and that sintered at 700°C showed visible luminescence at ~390 nm.

Key-words : Photoluminescence, Amorphous alumina, Sol–gel method, Visible emission, Sintering

[Received March 27, 2008; Accepted April 17, 2008]

Stable and low cost commercial supply is expected for aluminum because of the third highest Clarke number following oxygen and silicon. Further, alumina which is the oxide of aluminum is thermally and chemically stable with the melting point of 2020°C and the boiling point of about 3000°C, and high hardness. Thus it is used for the field of high mechanical strength polishing and cutting material, high temperature durable materials, the host matrix material for catalyst of the waste gas of the automobiles and so on.

As luminescent materials, alumina is frequently used as the temperature stable host matrix for Eu, Ce and Tb.[1)–8)] The luminescence of these materials results from the luminescent center formed by the doped rare earth elements. On the other hand, some papers reported luminescence from dopant-free alumina. In specific, alumina dispersed in silica glass,[9),10)] alumina prepared by cathode oxidation[11)–13)] and by ultrasonic spray pyrolysis technique[14)] and boehmite and γ-alumina prepared by hydrothermal method[15)] have been reported as luminescent materials. However, with respect to the cost and technique, the development of the new techniques is necessary to achieve self luminescent alumina ceramics. This paper reports the application of sol–gel technique to prepare self luminescent alumina.

Commercial available, analytical grade aluminum nitrate ($Al(NO)_3{\cdot}9H_2O$), acetylacetone ($CH_3COCH_2COCH_3$)(AcAcH), poly(vinylpyrrolidone) K–90 ($(C_6H_9NO)_n$) (PVP) and 2-methoxyethanol ($CH_3OC_2H_4OH$) were used as raw materials. AcAcH and aluminum nitrate were added to 2-methoxyethanol at room temperature and stirred until dissolving. Subsequently, PVP was added into the liquid at room temperature and stirred for 24 h. The reason why the addition of PVP and AcAcH is that the our final goal is the preparing the smooth luminescent alumina-based films. The liquid obtained was used as the precursor liquid. The composition of the liquid is $Al(NO)_3{\cdot}9H_2O$:AcAcH:PVP:$CH_3OC_2H_4OH$=1:2:1;30. The amount of PVP was estimated from the base of that of monomer vinylpyrrolidone (111.14). The liquid obtained was dried in air at 60°C for 3 d. Then, the liquid was fired at 500, 600, 650 and 700°C in air for 1 h. After the firing, liquid was turned to powder. X-ray diffraction(XRD) patterns were obtained by using Ni-filtered Cu Kα radiation and photoluminescence (PL) spectra excited by 275 nm light beam were measured. DTA-TG measurements were carried out by raising temperature of 10°C/min.

From the DTA-TG measurements, it can be found out that the oxidation peaks were seen below 300°C and remarkable weight loss could not be seen above 300°C. Hence, it can be said that the solvents evaporated by 300°C.

Figure 1 depicts XRD patterns of the powders obtained. No crystalline peak is detected from all powders obtained at each firing temperature, and thus the powders can be considered as amorphous.

PL spectra of the present powders are shown in **Fig. 2.** The strong peak at ~550 nm is the secondary excitation light beam. The peak at ~430 nm is observable for the powdered fired at 500, 600 and 650°C, and increased with increasing fiering temperature. The light was visible and it looks as violet. From the powder fired at 700°C, the peak generated at ~390 nm can be seen. Similarly, it looks violet, and the generated intensity is the strongest and clear. The further firing at higher temperature was not effective in the sense of luminescence. Peaks disappeared for

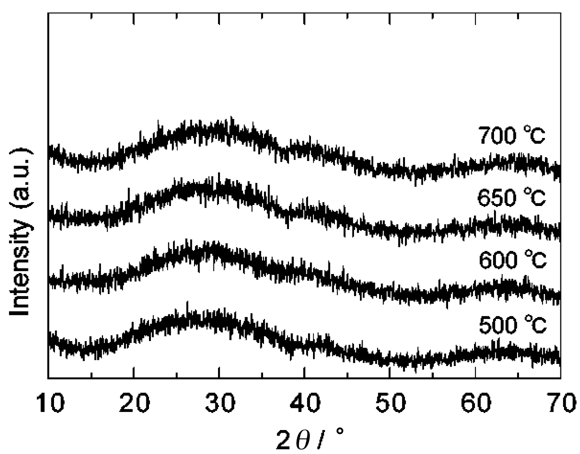

Fig. 1. XRD patterns of Al_2O_3 powder.

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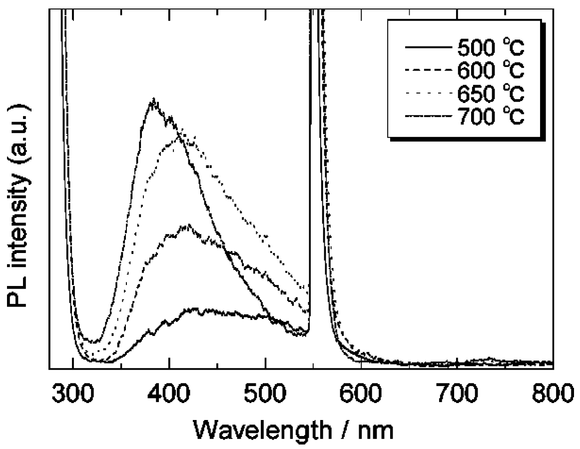

Fig. 2. PL spectra of Al_2O_3 powder.

those powdered, especially, crystalline powders. Therefore, it can be considered that the emission peaks results from oxygen deficiency, surface imperfection and/or OH bonding.

In conclusion, we successfully prepared amorphous alumina films by sol–gel method. From the XRD pattern, all powdered were confirmed to be amorphous. By PL measurements, peak observed at ~430 nm from the powered fired at 500, 600 and 650°C, and the intensity increased with increasing firing temperature. Further, strong peak at ~390 nm was obtained for the powder fired at 700°C.

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