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Origin of ultraviolet photoluminescence in zeolite-derived glass

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ABSTRACT

A series of silica glass compacts were prepared by rapid consolidation of zeolite powder using SPS under different conditions, and their photoluminescent properties were systematically studied in this work. XRD and high-energy synchrotron XRD results show that although the content of zeolite in glass decreases fast with increasing sintering temperature, the residual zeolite always can be found in obtained glass until 1300 °C. Moreover, despite the observed UV PL emission at ~370 nm for all prepared glass samples, the PL intensity decreases continuously with increasing consolidating temperature. Based on the FTIR results and HRTEM observation, it is deduced that the zeolite/glass interface which contains lots of defects plays a critical role for UV PL in the zeolite derived glass.

1. Introduction

Since the initial report of photoluminescence (PL) in mesoporous silicon [1],tremendous efforts have been made to the investigation of silicon-based light emitting materials, owing to the great possibility for the applications like optoelectronics, novel optical devices and light-weight panel display [2]. However, it has been realized quickly that the amorphous silica is more suitable for actual application in comparison to silicon, because of their excellent thermal and optical properties. For instance, the silica aerogels treated with microwave-energized reducing gas such as hydrogen and ammonia can induce permanent visible PL in the materials, probably due to the localized oxygen deficiency [3].Even without reduction treatment, silica aerogels prepared by hydrolysis also could display PL mainly in the UV region, which was deduced to be a result of excitation of surface defect states [4–6].In addition to silica aerogels, UV and visible PL could be detected as well in fused silica excited by a focused UV laser beam of ArF (λ_{exc} = 193 nm) or Nd:YAG (λ_{exc} = 266 nm)laser, which were attributed to the non-bridging oxygen hole centers, hydrogen related defects and self-trapped excitons [7,8]. Very recently, researchers found that the luminescent centers in single fused silica particle could be increased under irradiation of UV light, which is critical for solving the problem of photo bleaching of fluorophores [9].

Despite the large number of investigations in porous and particle silica, these materials usually suffer from instability and aging effect [5,10], which may result from their metastable surface structure (i.e. OH group) induced by the large surface area. In contrast, bulk silica glass with high relative density is more attractive due to the high stability and mechanical properties that will be favorable for application. Uchino et al. reported that a transparent bulk α-SiO_2 glass prepared from solid-phase sintering of nanometer-sized silica particles exhibits a unique white PL emission under ultraviolet excitation (λ_{ex} = 266 nm) [11]. Previously, we demonstrated a transparent silica glass derived from order-disorder transition (ODT) of zeolite using Spark Plasma Sintering (SPS) [12–15]. This novel silica glass exhibits PL emission under ultraviolet excitation though the reason behind the phenomenon is still not clear. In this paper, the zeolite derived silica glasses were prepared at different temperature using SPS. The relation between PL emission and the residual zeolite fragments in the densified samples was studied in detail.

2. Experimental procedure

The ZSM-5 powders (Si/Al ≈ 720) were loaded into a Φ12-mm graphite die and rapidly sintered using SPS at different temperatures with a holding time of 3 min. X-ray diffraction (XRD) patterns were collected on a Rigaku D/Max-2550 PC diffractometer (Tokyo, Japan) equipped with CuKα source. High-energy X-ray diffraction measurements were performed at the BL13W1 beam line of the Shanghai Synchrotron Radiation Facility (SSRF).·The details of experiment and standard data analysis are described elsewhere [16]. High resolution transmission electron microscopy (HRTEM) was performed on JEOL JEM-2010 microscope (Tokyo, Japan) operating at 200 kV. The transmittance was measured using a Perkin Elmer Lambda-950 UV-VIS-NIR

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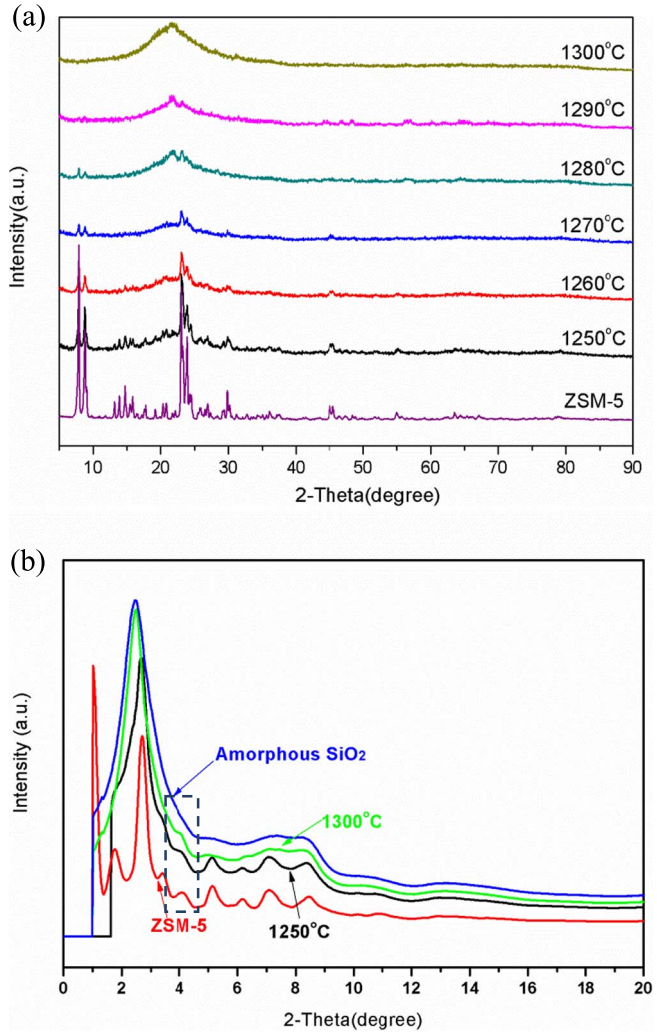

Fig. 1. (a)XRD patterns for zeolite (ZSM-5) and zeolite derived glass consolidated at different temperatures; (b) high-energy synchrotron XRD patterns for zeolite, amorphous SiO_2 and zeolite derived glass consolidated at 1250 and 1300 °C, respectively; the dash lined rectangle indicates the evolution of peak around 4°.

spectrometer. PL spectrum was obtained at room temperature using Steady-State and Time-Resolved Fluorescence Spectrometer (QM/TM/NIR, PTI, USA) with a Xenon arc lamp (75 W) at 300 nm as excitation source. FT-IR transmission spectra were performed using ThermoFisher Nicolet 6700 spectrum with 2 cm^{-1} resolution. The bulk transparent glass sample was crushed to powders and then pressed to a pellet with KBr for FT-IR measurement. The density of the compacts was determined by the Archimedes method. All emission spectra were corrected for the spectral response of the measuring system. All the measurements were performed at room temperature.

3. Results and discussion

In this work, the bulk glass was fabricated using zeolite powders as starting materials. Zeolites are crystalline aluminosilicates characterized by open structure of three dimensional frameworks composed of linked MO_4 tetrahedral units, where M = Si or Al atoms [17]. These structures contain microscopic channels and holes, which is easily collapsed and transform to an amorphous phase upon heating at high temperature [12]. This process was recorded using XRD as shown in Fig. 1. The peaks belonging to zeolite can be clearly seen for the sample consolidated at 1250 °C, though a hump ranged from 15 to 30° also can be observed, which represents the formation of amorphous glass phase. With increasing sintering temperature, the intensity of zeolite peaks decrease gradually, and completely disappear for the sample prepared at 1300 °C, indicating that most of the zeolite has transformed to glass phase. However, when the samples were examined by high energy XRD, which allowed us to distinguish the very fine structure reflected in the range of small angle, it is found that even for the sample sintered at 1300 °C, there are still very small amount of zeolite left in the glass matrix. As shown in Fig. 1b, the small peak around 4° which is a characteristic of zeolite becomes weaker with increasing sintering temperature but still can be identified in the sample prepared at 1300 °C. On the contrary, this feature cannot be observed in the ordinary amorphous SiO_2, since it is not prepared from zeolite.

The transmittance of the sample sintered at 1300 °C is shown in Fig. 2. The visible light transmittance is higher than 65% and near-infrared light transmittance is close to 90%. This high transparency in visible light range for the sample consolidated at 1300 °C also can be confirmed by naked eye directly, while for the other samples prepared at lower temperatures the transparency decreased gradually, finally

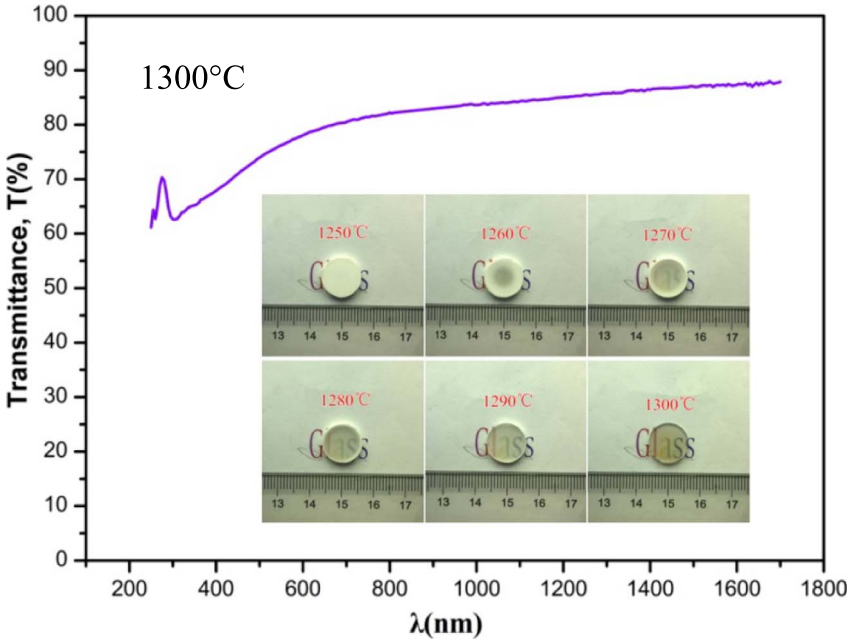

Fig. 2. The transmittance of the sample fabricated at 1300 °C for 3 min. The inset shows the photographs of as-sintered samples at different temperatures.