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Devitrification of ultrafast laser plasma produced metastable glass layer

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ABSTRACT

Erbium-doped tellurite modified silica (EDTS) is a newly formulated silicate glass that has the potential to realize on-chip optical amplifiers. The devitrification process of EDTS layer on host silica glass has been studied in the temperature range 300 °C–1000 °C. In-situ high-temperature X-ray diffraction, selective area electron diffraction and high-resolution transmission electron microscopy revealed the amorphous phase of EDTS as-fabricated up to 600 °C and rapid irreversible crystalline phase developments above 600 °C, contrasting the host silica. Distinct structural evolutions of EDTS with the variation in temperature were observed, including a complete evaporation of TeO_2 at 800 °C from the matrix.
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Erbium doped silicates are the sole material platforms for manufacturing erbium-doped fiber amplifiers (EDFA) that are efficient optical signal boosters used in a fiber optic communication systems [1–5]. Generally, the amount of erbium and its homogeneous distribution in silica determine the magnitude of light amplification, known as the optical gain [6,7]. The optical gain is high with higher erbium concentration in silica. However, due to the chemical compositional limitations of silica, even a moderate concentration of erbium (>0.1 at.%) in silica leads to clustering that degrades the signal strength [6,8,9]. Therefore, minimally doped long lengths (>5m) of EDFAs are maintained in current optical communication networks, which cannot be incorporated in the new generation compact integrated circuits that combine both optical and electrical components on a single substrate such as silicon photonics [10–15].

To realize compact optical amplifiers, we have developed a prospective material platform - erbium-doped tellurite modified silicate (EDTS) - by overcoming the fundamental limitations of erbium doping in silica utilizing a femtosecond (fs) laser plasma assisted novel process [16–18]. In this process, a tellurite glass target of composition (80-x)TeO_2-10ZnO-10Na_2O-xEr_2O_3 (TZNE) was prepared using standard glass melt-quenching process and then ablated using a fs-laser, inside a vacuum chamber. The highly energetic fs-laser generated plasma (ions and nano-particulates) [19], in 70 mTorr of oxygen within the vacuum chamber reacts with the host silica glass kept at a temperature of 700 °C and 70 mm above the target glass. These complex interfacial reactions between the TZNE plasma and the silica substrate cause the gradual dissolution of silica into the tellurite network to form the EDTS [16,18]. Such a homogeneous mixing of immiscible systems (tellurite and silica) and glass formation cannot be achieved through any existing glass fabrication process. The optically transparent EDTS could contain record high levels of Er^{3+} ions [18] and have potential applications in the field of optical communications such as on-chip optical amplifiers and loss compensated devices for *Si*-photonics. However, the microstructural differences of metastable EDTS from the base silica are not completely understood, except its higher material density than silica and the superior spectroscopic characteristics [16]. Therefore, it is essential to evaluate the structural characteristics and stability of EDTS at high temperatures, which in turn could validate its compatibility with a standard CMOS manufacturing process for the imminent product developments.

In this study, the high-temperature (300 °C to 1000 °C) investigations were carried out on a representative EDTS sample ×1 of thickness 1 μm fabricated on a silica glass (spectrosil 2000) of dimensions 30 mm (L) × 20 mm (W) × 1 mm (T). The sample was prepared by the fs-laser ablation (fs laser energy 50 μJ, wavelength 800 nm, estimated laser peak intensity ~10^{13} W/cm^2, pulse width 100 fs, and pulse repetition rate 1 kHz) of a tellurite glass target of molar composition 79.5TeO_2-10ZnO-10Na_2O-0.5Er_2O_3. The structural characterization was carried out using X-ray diffraction (Philips PANalytical X'pert Diffractometer) and high-resolution transmission electron microscopy (HRTEM) (FEI Tecnai F20 200 kV FEGTEM) techniques. For the in-situ high-temperature XRD, the sample ×1 was diced into smaller dimensions of 15 mm × 15 mm to fit in the XRD stage. The sample holder attached to the hot stage was covered with a platinum foil, prior to placing the sample in, to protect the holder from any damages, in the event of glass melting at high temperatures. The sample was heated in steps from 300 °C to 1000 °C with a step size of 25 °C. At each temperature, the sample was held for 30 min and the XRD measurements were performed across the 2θ range 10–80° (scan rate: 0.05°/second). After the

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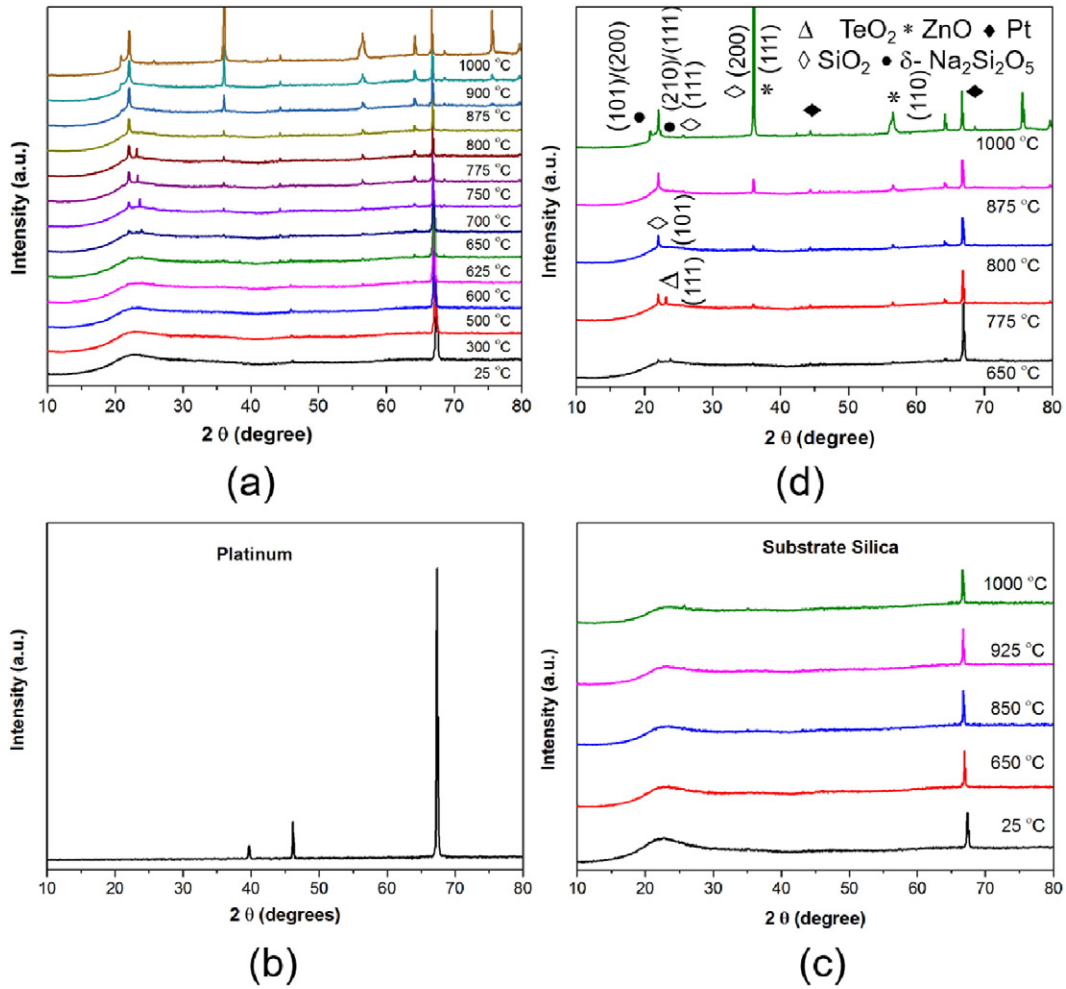

Fig. 1. X-ray diffraction patterns for (a) EDTS as fabricated (25 °C), and in-situ high temperature data between 300 °C and 1000 °C indicating the progressive crystalline phase development of EDTS, (b) platinum foil at room temperature, (c) substrate silica in the temperature range 25 °C to 1000 °C, and (d) selected XRD patterns of EDTS with indexing of the likely peaks corresponding to $Na_2Si_2O_5$, SiO_2, TeO_2 and ZnO in the temperature range 650 °C to 1000 °C.

experiment, the sample was allowed to undergo a natural cooling back to the room temperature. The post-annealing XRD scan was performed to ensure that the phase change produced in the material are permanent, even after the cooling. The X'pert High Score Plus software package was used to index the peaks detected in the experimental diffraction patterns. The XRD patterns of a pure silica substrate and the platinum foil were also recorded under the same experimental conditions to authenticate the XRD peaks obtained from EDTS. For the in-situ high-temperature TEM imaging and selected area electron diffraction (SAED) analysis, a section of the sample ×1 was extracted using the standard FIB TEM sample preparation method [20] and attached to a TEM grid. This grid was subsequently examined by TEM using a Gatan Model 628 single tilt heating holder. TEM bright field images and SAED data were recorded using a Gatan Orius SC600 CCD camera. The temperature cycle used for this experiment was kept the same as that in the XRD analysis.

Prior to the high-temperature studies, the room temperature (25 °C) XRD, HRTEM images and SAED patterns were also obtained from the sample to identify the basic structure of EDTS as-fabricated. This assorted analysis confirmed the amorphous phase of EDTS. Further, the evaluations were initiated with the in-situ high-temperature XRD experiments and the patterns collected on the set temperatures are presented in Fig. 1a. At room temperature, three peaks corresponding to the 2θ angle of 39.68°, 46.12°, and 67.38° were observed. These peaks correspond to platinum (Pt, ICDD reference code: 04-002-0887), which was confirmed by the XRD pattern collected from the platinum foil alone, as shown in Fig. 1b. The XRD pattern collected from the substrate silica glass is presented in Fig. 1c, under the same experimental conditions, which shows a low-intensity band peaked at 25.79° at 1000 °C and a stable peak at 67.38° corresponding to the background platinum foil. This analysis proved the absence of any crystals of detectable size and the amorphous phase of the base silica up to the maximum temperature employed in this study. Hence, it is asserted that all the peaks (excluding the Pt peaks) appearing in the XRD pattern presented in Fig. 1a are those characteristic of EDTS at elevated temperatures.

The absence of any significant XRD peak at room temperature validates the amorphous structure of EDTS as fabricated. It is evident from the XRD patterns that the composite material starts to lose its amorphous nature at temperatures above 600 °C. The diffraction peaks appearing with the increased temperatures from 600 °C onwards indicate the progressive crystalline phases in EDTS. However, analysing a mixture of new crystalline phases and a perfect match for all the

Fig. 2. HRTEM cross-sectional images and SAED patterns of EDTS captured at various temperatures presenting the evolution of crystalline phases in EDTS. HRTEM micrographs of EDTS at (a) 300 °C, (b) 600 °C, (c) 700 °C, and (d) post experiment, cooled to room temperature. Selected area diffraction pattern collected from the middle of EDTS at (e) as fabricated, (f) 650 °C, (g) 750 °C, and (h) after cooling the sample to room temperature.

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