



# Holographic testing of fluorescent photosensitive glass–ceramics

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## ARTICLE INFO

### Article history:

Received 15 April 2010

Received in revised form 4 October 2010

Accepted 6 October 2010

### Keywords:

Holography

Fluorescent photosensitive glass–ceramics

Recording

## ABSTRACT

In this paper we investigate the holographic recording properties of the fluorescent photosensitive glass–ceramics. The materials described here, stable up to 400 °C, represent a substantial advancement in the development of long lifetime media for holographic data storage. A detailed physical–chemical characterization of the proposed material is presented. It is shown that volume holograms were obtained after the recording process using a continuum high power laser in an off-axis setup.

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## 1. Introduction

Starting with Gabor's holographic principle, in our days many techniques were developed: holographic memories, digital holographic microscopy, computer generated holograms, holographic displays, and security holograms [1–4].

In recent years, the interest in new materials for holographic memory increased. The potential for high-speed/density volume holographic data storage has made the photosensitive materials an attractive subject [5–7]. Such high densities are attributable, in part, to the holographic storage media ability to store information in three dimensions. Thick holograms were recorded in media with thickness larger than the wavelength of the incident radiation. A problem appears when long life recording media are needed for archiving and back-ups. Organic materials (photographic emulsions, dichromated gelatin, photoresists, photothermoplastics, and photopolymers) failed to store data for decades. Photorefractive crystals [5] supply an expensive solution.

A new material, fluorescent photosensitive glass–ceramic is tested and the results are presented in this paper. The fluorescent photosensitive glass–ceramics employ fluorosilicate glass compositions including various photosensitizing metals and rare earths which impart photosensitive properties and fluorescent properties to the materials. After short recording exposure (microsecond regime) in a holographic setup, a thermal treatment was applied to control the

precipitation of fluoride nanocrystals, which contain the hologram recording.

This paper reports the demonstration of thick hologram storage in fluorescent photosensitive glass–ceramics.

## 2. Experimental procedure

### 2.1. Preparation

The fluorescent photosensitive glass–ceramics with a composition (in wt. %): 69SiO<sub>2</sub>–15.8 Na<sub>2</sub>O–4.8ZnO–6.3Al<sub>2</sub>O<sub>3</sub>–0.5Sm<sub>2</sub>O<sub>3</sub>–0.05CeO<sub>2</sub>–0.2Sb<sub>2</sub>O<sub>3</sub>–0.05SnO–2.3F<sup>–</sup>–0.01Ag<sup>+</sup>, were prepared by melt quenching from purity SiO<sub>2</sub>, NaCO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, Sm<sub>2</sub>O<sub>3</sub>, ZnO, sodium fluorosilicate, Sb<sub>2</sub>O<sub>3</sub>, SnO, CeO<sub>2</sub> and AgNO<sub>3</sub> raw materials. Photosensitivity property was induced by Ce<sup>3+</sup> and Ag<sup>+</sup> ions, while strong fluorescence was produced by Sm<sup>3+</sup> ions. Accurately weighted 100-g batches were thoroughly mixed and placed in alumina crucibles. The mixtures were melted at 1550 °C for 1 h. Then the melts were poured into preheated graphite molds and annealed at 400 °C. Discs of 1 mm thick and 12 mm diameter with optically flat surfaces were produced after cutting and polishing processes.

### 2.2. Characterization

DSC measurements were carried out by a Shimadzu DTA-50 Differential Thermal Analyzer. The crystalline phases of the samples were identified by X-ray diffraction. The X-ray powder diffraction data were collected on a D8 Discover Bruker AXS diffractometer using Cu Kα1 (λ = 1.54060 Å) radiation and a filter for Cu Kβ. Diffraction patterns were collected in the 2θ range from 10° to 100°, in steps of

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0.04°. Samples of fluorescent photosensitive glass–ceramics were analyzed using a Hitachi S-2600 SEM operating at 25 kV and optical absorption spectra were measured with a Jasco V-560 UV–VIS spectrophotometer.

### 3. Results and discussion

#### 3.1. Glass and glass–ceramics properties

In this article, we report on the preparation and characterization of a Sm fluorescent photosensitive glass–ceramics used for holographic testing. The material was obtained by the heat treatment of an irradiated precursor glass above the glass transition  $T_g$ , resulting in nanocrystals with  $\text{Sm}^{3+}$  ions.

The first test of controlled crystallization is to examine the high temperature crystallization behavior with differential scanning calorimetry (DSC). The DSC curve of a sample is presented in Fig. 1. The inflexion point corresponds to the glass transition  $T_g$ , situated at about 455 °C. The exothermic shoulder situated at 535 °C is attributed to  $\text{NaSmF}_4$  crystallization and the broad endothermic peak located at 660 °C corresponds to the melting of fluorescent photosensitive glass–ceramic components.

The fluorescent photosensitive glass–ceramics were obtained by thermal treatment of the glass, for 4 h at 520 °C in order to precipitate nanocrystals without loss of transparency. Fig. 2 shows the X-ray diffraction pattern of the material with 0.5 wt. %  $\text{Sm}_2\text{O}_3$  after heat treatment. A broad diffraction curve characteristic of the glass structure is shown for the as-annealed glass, whereas sharp peak characteristics of  $\text{NaSmF}_4$  crystals are clearly exhibited, indicating that nanocrystals were successfully precipitated during thermal treatment. The fcc cell parameter  $a$  of the samples have been computed in terms of the X-ray diffraction data. The value for  $a$  is 5.437 Å.

Fig. 3 illustrates the transmission spectra in the 200–900 nm wavelength range, obtained from fluorescent photosensitive glass–ceramics with two  $\text{Sm}_2\text{O}_3$  doping levels. Initial materials with 0.5 wt. % and 5.5 wt. %  $\text{Sm}_2\text{O}_3$  are transparent and present two absorption bands at 304 nm and 401 nm. The first band is attributed to  $\text{Ce}^{3+}$  ( $^2\text{F}_{5/2} \rightarrow ^2\text{D}_{5/2}$ ) [7] and the second one to  $\text{Sm}^{3+}$  ( $^6\text{H}_{5/2} \rightarrow ^4\text{L}_{13/2}$ ,  $^4\text{F}_{7/2}$ ,  $^6\text{P}_{3/2}$ ,  $^4\text{K}_{11/2}$ ,  $^4\text{M}_{21/2}$ , and  $^4\text{L}_{15/2}$  [8]). The absorption intensities are proportional with ion concentration. The sample with 5.5 wt. %  $\text{Sm}_2\text{O}_3$  presents an increased absorption at 401 nm vs. the sample with 0.5 wt. %  $\text{Sm}_2\text{O}_3$ .  $\text{Sm}^{3+}$  ions have an absorption line at 532 nm corresponding to the transition:  $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{3/2}$  [8]. This line is suitable for recording holograms with high power frequency doubled Nd:YAG lasers. The recording mechanism is detailed in Fig. 4. In the first phase, the precursor glass containing  $\text{Sm}^{3+}$  ions is irradiated by intense light such as a laser beam. The following phase consists in a heat treatment which stimulates the growth of  $\text{NaSmF}_4$  nanocrystals formed on Ag clusters as crystallization

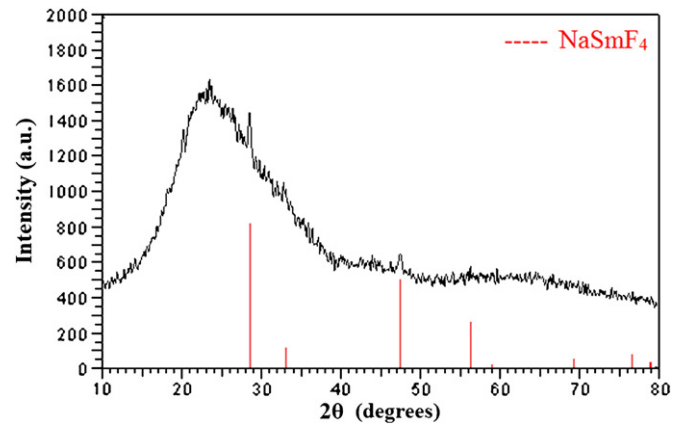


Fig. 2. X-ray diffraction pattern of fluorescent photosensitive glass–ceramics with 0.5 wt. %  $\text{Sm}_2\text{O}_3$  after thermal treatment.

seeds. The result consists of  $\text{NaSmF}_4$  nanocrystals located only in intense irradiation regions.

#### 3.2. Holographic recording by Sm fluorescent photosensitive glass–ceramics

Holographic method is used to record phase and amplitude information of the object wave in photosensitive materials (holographic plates, special glasses, photorefractive crystals, fluorescent photosensitive glass–ceramics, photopolymers, etc.) by a special procedure that permits the reconstruction of the object image. In the recording step, the object is illuminated by a laser beam and the diffracted wave  $O$  will interfere with the reference wave  $R$ . The pattern with intensity  $I$  is recorded in one of the photosensitive materials:

$$I = |R + O|^2 = |R|^2 + |O|^2 + R^*O + RO^* \quad (1)$$

where the asterisk denotes the complex conjugation.

In the reconstruction step, the recorded material is illuminated only by the reference wave. The mathematical expression of the reconstructed wavefront is:

$$U = RI = R|R|^2 + R|O|^2 + |R|^2O + R^2O^*. \quad (2)$$

The first two terms create a zero-order diffraction, the third term produces a virtual image located at the initial position of the object, and the fourth term produces a real image of the object situated on the opposite side of the hologram.

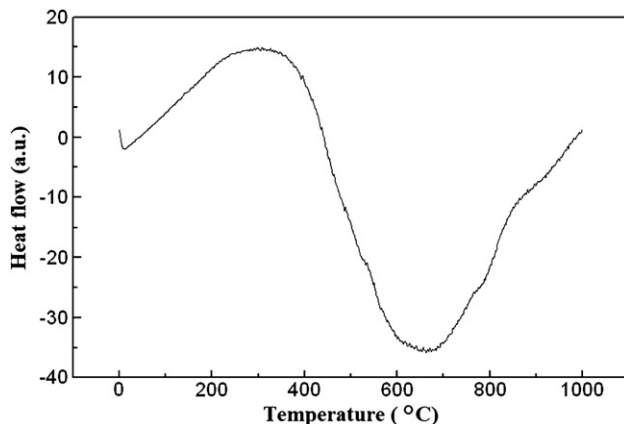


Fig. 1. DSC curve of a fluorescent photosensitive glass–ceramic.

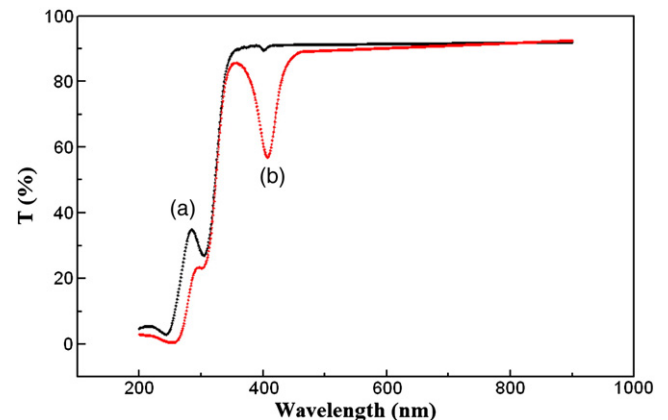


Fig. 3. Transmission spectra of fluorescent photosensitive glass–ceramics with (a) 0.5 wt. %  $\text{Sm}_2\text{O}_3$  and (b) 5.5 wt. %  $\text{Sm}_2\text{O}_3$ .

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