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The synthesis of bimetallic gold plus nickel nanoparticles dispersed in a glass host and behavior-enhanced optical nonlinearities



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ABSTRACT

A sol-gel synthetic route toward the fabrication of bimetallic gold plus nickel nanoparticles (NPs) in sodium borosilicate glasses is presented, with controlled atmosphere heat treatments. Characterizations of the as-obtained glasses indicate that when exposed to variable atmospheres of oxygen (O_2) and oxygen (O_2) -hydrogen (H_2) , randomly distributed Au/NiO NPs and Au/Ni NPs are produced in the glass host, respectively. Subsequently, the Z-Scan technique is conducted to examine their nonlinear optical response with femtosecond pulses. The two glasses show superior optical nonlinearities comparable to that of the glasses incorporated with Au NPs. For the glass dispersed with Au/NiO NPs, the enhancement of the third-order nonlinear response mainly depends on a contribution of a stronger nonlinear refraction response. However, the dominant contribution to improve optical nonlinearities of the glass doped with Au/Ni NPs is the more intense nonlinear absorption effect.

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1. Introduction

The design and fabrication of new materials with outstanding thirdorder nonlinear optical performance has been extensively investigated, as it represents an active research field in nonlinear devices [1–4]. Owing to a an advantageous combination of metal nanoparticles (NPs) and a stable glass residence, metal/glass nanocomposite materials demonstrate attractive nonlinear optical features, making them an emerging class of extremely significant candidates for photonic applications [5–6].

Among the various dopants, the majority of studies on this topic have focused on monometallic species in recent years [7–9]. However, it's generally demonstrated that bimetallic NPs not only display the combined properties of their individual constituents but also exhibit rather superior properties which are distinctly different from those of the monometallic counterparts [10]. Therefore, it is intriguing to mount two different NPs in a dielectric glass matrix to study the possibility of tailoring the resulting nonlinear optical behavior by a facile processing route. Au NPs are known to exhibit large absorption coefficients and ultra-fast response, which hold promise in such applications as alloptical switches and optical limiters [11]. In this context, research on the third-order nonlinear response of Au NPs has been presently one of the most dynamic research issues [12–15]. Therefore, the design of Au NPs entrapped glass will be given priority. It is worth mentioning that the

study of the nonlinear optical properties of Au-based bimetallic NPs is far from sufficient. Additionally, research has shown that while associating noble metals to transition metals, the conduction electrons contribute to the improved localized surface plasmon resonance, and thereby exhibits enhanced nonlinear optical effects [16]. Hence, the magnetic@ noble metal Au-Ni system sparked our interest for this investigation. Furthermore, most of the studies on the Au-Ni system have aimed at exploring their magnetic properties, optical gas-sensor properties and catalytic activity. Few studies have been published on their optical properties, let alone the third-order nonlinear optical properties. Additionally, exploiting a new idea for facile and general synthesis of the configuration consisting of gold plus nickel NPs embedded in dielectric glass is scarce up to the present. As discussed above, realizing the fabrication of gold plus nickel NPs co-doped glass and exploring their nonlinear optical responses are worthy to the attempt.

Despite the prospective applications, it is difficult to disperse gold plus nickel NPs homogeneously in a transparent matrix. To our knowledge, several traditional synthetic approaches to the fabrication of glass nanocomposites have been studied, including melt-quenching, sol-gel methods, chemical vapor deposition and ion exchange. Melt-quenching method suffers from high temperature defect in `producing glass composites, which prohibits the incorporation of the desired non-crystalline phase. While, the necessary equipment for carrying out chemical vapor deposition and ion implantation are expensive. Fortunately, lowering temperature sol-gel method lacks these limitations. The sol-gel methods merits also include of the potentially higher purity, simplification of the processing procedure and diversification of doping

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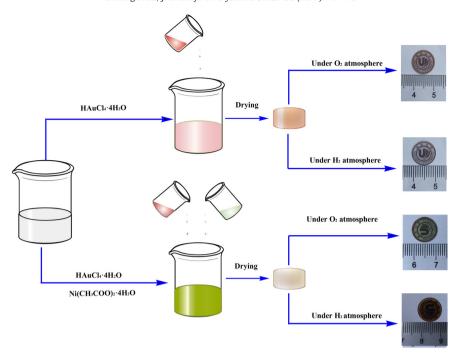


Fig. 1. Schematic of the synthesis for the production of as-obtained glasses.

species homogeneity. This method has been regarded as a template for preparing various materials such as ceramics, thin films and glass [17–19].

On the basis of the fact that particle size and composition can produce important changes for the nonlinear optical response, herein, we designed a method to fabricate sodium borosilicate glasses consisting of gold and nickel NPs employing the sol-gel route plus varying atmospheres of oxygen ($\rm O_2$) and hydrogen ($\rm H_2$). Microstructural analysis showed that, in our experiment, randomly dispersed Au/NiO NPs, Au/Ni NPs were produced in sodium borosilicate glasses under $\rm O_2$ and $\rm H_2$ atmospheres, respectively. Furthermore, we investigated the third-order nonlinear properties of the as-obtained glasses using ultrafast 190 fs laser pulses. The success of the fabrication of sodium borosilicate glass dispersed with Au/NiO NPs and Au/Ni NPs indicates the potential for synthesizing sodium borosilicate glass with other bimetallic nanoparticles. This method may have instructive significance on the progress of photodevices.

2. Experimental section

Gold plus nickel NPs co-doped glasses were fabricated through a solgel method plus controlled atmosphere of O2 and O2-H2. A schematic of the synthesis for the construction of as-obtained glasses is presented in Fig. 1. The sodium borosilicate glass chosen as the glass host was composed of $5Na_2O-20B_2O_3-75SiO_2$ (in wt%, ± 0.1 wt%). Gold salt HAuCl₄·4H₂O and nickel salt Ni(CH₃COO)₂·4H₂O acted as starting precursors for the production of bimetallic gold and nickel NPs and the initial weight ratio of Au and Ni was set to as 1:3. It should be noted that the O₂ atmosphere tends to produce smaller NPs while H₂ contributes to the formation of larger NPs, which has a noticeable impact on the transparency and nonlinear optical properties of glass. After several attempts, the optimal dosages of Au and Ni were determined to be 2% wt% and 1% wt% (± 0.1 wt%) in the glass host under O₂ and H₂ atmospheres, respectively. The fabrication of glass experienced an evolution of the sol-stiff gel-solid glass, among which the sol and stiff gel were prepared using a previously reported [20]. Hereafter, to the method of controlling the formation of the desired glasses from the stiff gel to the solid glass is illustrated as follows.

The conversion from a stiff gel to a glass was realized by performing the following steps. First, the stiff gel was thermally annealed in a continuously flowing oxygen atmosphere at the heating rate of 5 °C·h $^{-1}$ (± 0.1 °C·h $^{-1}$) from room temperature to 450 °C, and afterward prolonged for 10 h at 450 °C. The thermal annealing process promoted the removal of the residual organic solvent and the decomposition of the precursors. Then, the stiff gel was exposed to H2 atmosphere at 450 °C with a holding time of 10 h to produce metallic Au and Ni NPs (for the glass containing Au/NiO NPs, the atmosphere at this stage was replaced with O2). Finally, the above stiff gel was heated in a nitrogen atmosphere at a heating rate of 10 °C·h $^{-1}(\pm 0.1$ °C·h $^{-1})$ from 450 °C to 600 °C, and maintained for 2 h at 600 °C to achieve the final glasses. Hereafter, they are referred as Au/NiO and Au/Ni glasses, respectively. For comparison, the glasses containing Au NPs (named as Au glass) were synthesized by following the above thermal process in the absence of Ni.

The crystalline phases of the as-synthesized glasses were investigated by an X-ray diffractometer (Bruker, Germany) using Cu $K\alpha$ radiation.

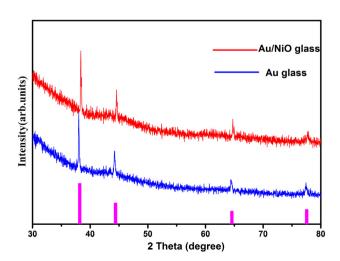


Fig. 2. XRD patterns of Au glass and Au/NiO glass under an $\rm O_2$ atmosphere. The JCPDS pattern of Au is also shown.

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