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Silver nanoparticles/polydimethylsiloxane hybrid materials and their optical limiting property

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ABSTRACT

To exploit the application potential of Ag nanoparticles (AgNPs) in optical limiting materials, it is significant to develop AgNPs doped polymeric hybrid materials. In this paper, spherical AgNPs were prepared using the seeding growth method. An additional silica coating was employed to obtain the Ag@SiO_2 core-shell materials. Then Ag@SiO_2 was doped into polydimethylsiloxane(PDMS) rubbers to investigate their optical limiting property to laser at 532 nm. The results showed that the Ag@SiO_2 doped PDMS rubber exhibited good optical limiting effect, which should be attributed to the nonlinear optical absorption and refraction. The AgNPs-PDMS hybrid materials would be very promising in optical limiting application area.

1. Introduction

Noble metal nanoparticles, especially Au and Ag nanoparticles, have attracted much attention for their excellent properties and application in many important areas [1,2]. Such metal nanoparticles possess strong surface resonance band in the visible to near-infrared regions. The ultrafast nonlinear optical response time facilitates their application in optical communication, all-optical switches and nonlinear optics [3,4]. Therein, optical limiting materials are very important for the manipulation of optical beams in the passive method. Silver nanoparticles with certain size and morphology are significant to exploit optical limiting materials because bulk silver exhibits the highest electrical (or thermal) conductivity among all metals.

It was reported that silver-containing nanocrystalline particles in polymer-stabilized suspensions of high linear transmittance had strong limiting effect to lasers at 532 nm. The optical limiting responses of the suspensions were significantly better than those of the benchmark materials fullerene and metallophthalocyanine in solution [5]. Large optical limiting effect from silver-dendrimer nanocomposites in aqueous solution was also reported and the optical limiting performance compared well to the results obtained with other organic structures. The mechanism governing the optical limiting was attributed to nonlinear scattering [6]. A novel [60]fullerene-silver nanocomposite was prepared by in situ reduction of silver ions encapsulated in a monofunctionalized methano-[60]fullerene derivative with reverse micelle-like structure. The experimental results demonstrated that the optical limiting behavior of the nanocomposite in organic solvent was better than that of C60 and the methano-[60]fullerene derivative [7,8]. The better optical limiting performance was attributed to the excited state interaction between the [60]fullerene and silver nanoparticles. Silver nanoparticles prepared by a focused femtosecond laser irradiation in $AgNO_3$ solution in the presence of TiO_2 sol exhibited strong self-focused effect and significant optical limiting property [9]. Recently, well-monodispersed silver nanopentagons were prepared and the nonlinear optical properties were investigated by open aperture Z-scan technique. The silver nanopentagons exhibited intensity-dependent transformation from saturable absorption (SA) to reverse saturable absorption (RSA), and the transformation from SA to RSA was found to be highly dependent on the shape and morphology of the silver nanoparticles [10].

To exploit the application potential of Ag nanoparticles (AgNPs) in optical limiting materials, it is significant to develop AgNPs doped nonlinear polymeric materials due to their relatively low cost, narrow dispersion in the refraction index, virtually endless possibilities of structure modification and good processability [11]. Among the various kinds of polymers, polydimethylsioxane (PDMS) elastomer is of particular interest due to its many useful properties such as distinguished flexibility, low toxicity, chemical inertness, good thermal stability and transparency in the visible region [12,13]. As soft matrix for optical limiting materials, PDMS rubbers can provide excellent chemical and thermal stability in the course of operation. In this article, spherical AgNPs were prepared using the seeding growth method. An additional

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silica coating was employed to obtain the $Ag@SiO_2$ core-shell materials, which could change their solubility, or decrease their propensity to aggregate. Then $Ag@SiO_2$ was doped into PDMS rubbers to investigate their optical limiting property to laser at 532 nm. The results showed that the $Ag@SiO_2$ doped PDMS rubber exhibited good optical limiting effect, and the optical limiting performance was attributed to the nonlinear optical absorption and refraction.

2. Experimental section

2.1. Preparation of AgNPs

AgNPs were prepared using the seeding growth method [14]. First, 100 mL solution containing 0.1 mM $AgNO_3$ and 0.3 mM sodium citrate was heated to boiling, then 1 mL of 50 mM $NaBH_4$ was added to the boiling solution under vigorous stirring. The solution was further boiled for 40 min and then left to cool to get the Ag seed solution. Then 20 mL of 50 mM cetyltrimethylammonium bromide (CTAB) solution was mixed with 0.5 mL of 10 mM silver nitrate at 27 °C, to which 1 mL fresh ascorbic acid solution of 100 mM was added to obtain growth solution. Finally, 3 mL of seed solution was added to the growth solution with gentle stirring, followed by dropwise addition of 100 µL of 1 M NaOH. The mixture was further stirred for 6 h. The AgNPs colloids were centrifuged, and resuspended in 2 mM CTAB solution to decrease the content of free CTAB.

2.2. Preparation of $Ag@SiO_2$ colloids

$Ag@SiO_2$ colloids were prepared by the literature method [15,16]. 50 µL of 1.08 mM 3-mercaptopropyl trimethoxysilane (MPS) solution in ethanol was added into 10 mL of AgNPs colloids. Ammonium hydroxide was used to adjust the pH of the solution to 9.35. Then 20 µL tetraethyl orthosilicate (TEOS) in 20 µL ethanol was added to the resulting solution and allowed to stand for 12 h. The $Ag@SiO_2$ colloids were transferred into ethanol by repeated centrifugation and ultrasonic dispersion.

2.3. Preparation of AgNPs-PDMS elastomer sheets

$Ag@SiO_2$ doped PDMS elastomers were prepared by adding a certain amount of $Ag@SiO_2$ colloids in ethanol into mixture of PDMS prepolymer and curing agent (10:1 weight ratio, SYLGARD 184 from Dow Corning) under stirring. The mixture was poured in a glass container with diameter of 25 mm, vacuum degassed and slow vulcanized at 65 °C for 10–12 h.

2.4. Instruments and optical property measurement

UV–vis absorption spectra were obtained using a PERSEE TU-1901 spectrophotometer (Beijing Purkinje General Instrument Co., Ltd). Transmission electron microscopy (TEM) was carried out on a JEOL JEM-2100 electron microscope. Zeta potential measurement was made on a Malvern Nano ZS90 Zetasizer. Thermogravimetric differential thermal measurements (TG-DTA) were performed using a Netzsch STA 449 C thermal analyzer (Selb, Bavaria, Germany) by heating the rubber samples from 286 to 1073 K under air atmosphere at a heating rate of 10 K/min. The optical limiting property of AgNPs /PDMS rubber was measured with Continuum Surelite Laser, which provided 5.0 ns laser pulses at 532 nm with a repetition rate of 10 Hz [3]. The laser pulses are spatially Gaussian, and the beam diameter is approximately 7 mm. The energy of incident laser was adjusted by a polarization analyzer. The beam was split into two beams by the beam splitter, and the reflected beam was used to monitor the incident energy. A lens with a focal length of 100 mm was used to focus the incident beam on the surface of the rubber sheet samples and the beam waist at the focus was 9 µm. The laser was operated in the single shot mode, and the incident and transmitted laser pulses under same pulse irradiation were monitored with two FieldMaxII-P laser energy meters (J-10MB-LE energy sensor, Coherent Inc. USA) respectively. For measuring the nonlinear optical properties, Z-scan experiments were conducted on the rubber sample according to the literature [17]. The laser pulse power used in each Z-scan experiment was constant at 21.6 GW/cm^2. A focused laser beam was used for irradiating the sample, which was moved along the beam (taken as the z-axis). The scan started from a distance far away from the focus (negative z) to the right (positive z) through the focal point ($z=0$). For each z position the corresponding transmission was measured. The transmittance through the sample was studied as a function of the sample position z. For comparison, the same experiment was performed for $Ag@SiO_2$ solution housed in quartz cell with a path 1 mm long.

3. Results and discussion

3.1. Preparation of AgNPs and $Ag@SiO_2$

Spherical AgNPs were prepared according to the seeding growth method for core-shell particles. Silver seeds were first prepared by the reduction of $AgNO_3$ with $NaBH_4$ under the protection of sodium citrate. Then AgNPs were obtained by reduction of $AgNO_3$ with ascorbic acid in the presence of seed solution in CTAB solution. Fig. 1 shows the UV–vis absorption spectra of Ag seeds and AgNPs solution. For Ag seeds solution, only a surface plasmon band appears at 400 nm, which can be attributed to plasmon resonance of nanosized Ag seeds. The plasmon band red shifts to 435 nm as the Ag seeds grew into AgNPs. Zeta potential was measured for the solution of Ag seeds and AgNPs. The Zeta potential of Ag seeds is −10.6 mV, whereas the Zeta potential of AgNPs is +56.1 mV. So, it is obvious that the surface of Ag seeds is protected by citrate anions, while the AgNPs are covered by CTAB bilayer.

The key step during the synthesis of the AgNPs-PDMS nanocomposite hybrids is the mixing of the prepolymer with the nanoparticles colloids in ethanol, so the AgNPs prepared in water should be first transferred into organic phase (ethanol). The surface property of the AgNPs must be properly engineered to avoid the irreversible aggregation of AgNPs in the course of transfer. Silica coating was proved to be an effective method to change the surface character of metal nanoparticles [15,18,19]. Herein, silica coating was adopted to prepare $Ag@SiO_2$. The silica coating was carried out according to the Stöber-Fink-Bohn method with MPS as coupling reactant to deposite silica on the surface of the AgNPs. The UV–vis absorption spectrum of $Ag@SiO_2$ is given in Fig. 1. The surface plasmon resonance band appears at 448 nm, and the redshift relative to AgNPs is due to the refractive index increase of the surrounding media. TEM analysis of the AgNPs before and after silica coating was performed. As shown in Fig. 2, the AgNPs have good

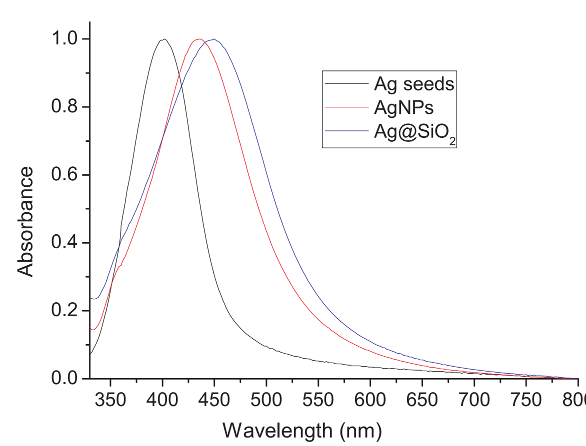

Fig. 1. Normalized UV–vis absorption spectra of Ag seeds, AgNPs and $Ag@SiO_2$ sol.

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