



Effect of Thermal Growth on Structural And optical Properties

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Abstract: Ag nano-chip like structures has been prepared by chemical method at different annealing temperature. The X-ray diffraction results showed that face-centered cubic crystalline of Ag nano-chip like structure with grain size in the range of 35 – 50 nm has been obtained. The effect of temperature (100, 150 & 200) ° C on the size of silver nano-chip like structure was investigated. FESEM images show the formation of Ag nano-chip like structure with grain size ranging from 35 to 50 nm. The Raman spectra & optical absorption exhibited peaks corresponding to the Silver. Thus, we were able to develop a method for synthesis of nano-chip like structure based on the annealing temperature.

Key Word: Silver nano-chip like structure, FESEM, Raman, structural property and optical property.

Introduction

In recent years, to synthesis the metal nanoparticles have many ways that can be classified as both chemical or physical methods and biological synthesis. The chemical methods are the polyol process [1], chemical reduction, the thermal decomposition of metal, the alcohol reduction process and electrochemical synthesis [2, 3]. Physical methods include chemical vapor deposition, pulsed laser ablation, plasma, sonochemical reduction and supercritical fluids [4]. For biological synthesis various organisms are used for nanoparticles synthesis, because of its effectiveness and flexible biological factors [5, 6]. At present, various types of metal inorganic nanoparticles zinc, copper, titanium, magnesium, selenium, gold, iron and silver have been synthesized using various techniques [7]. Their possible applications in subsequent technology development are due to their physical and chemical properties [8]. Silver has been used in medicine to gastronomic items because of their disinfecting effect [9]. The unique optoelectronic and physicochemical properties, silver nanoparticles have attracted remarkable attention in recent decades.

Here we report, the silver nano-chip like structure were synthesized by a simple chemical precipitation method and then the effect of annealing temperature on the synthesis of silver nano-chip like structure was examined.

Experiment

Silver nano-chip like structures is synthesized by the chemical method with an inorganic base and without any external capping agents. 0.5 M of Silver nitrate (AgNO₃), 0.5 M of glucose and 0.5 M of Sodium hydroxide (NaOH) were separately dissolved in water and stirred. Initially the aqueous solution of AgNO₃ and glucose were added together then 0.5 M of Sodium hydroxide added drop wise with the above said mixer of aqueous solution at room temperature. The colour of the solution changed to yellow immediately when NaOH is added and then stirring was continued, colour changed to grey colour indicating the formation of silver (Ag) nanoparticles. The resultant solutions were well dissolved by using magnetic stirrer for 1 hour at room

temperature. The co-precipitated solutions were centrifuged at 1000 rpm for 30 min to separate undesired agglomerates and washed with de-ionized water and ethanol. After centrifugation, the samples were dried for one hour at 100°C to eliminate residual gas, water impurities and to obtain pure crystalline Ag nanoparticles. The prepared Ag nanoparticles at 100 °C are annealed at different temperature of 150 °C and 200 °C in argon atmosphere.

X-ray diffraction (XRD) has been studied using PANalytical X-ray diffractometer and surface morphology and composition of synthesized Ag nanoparticles were characterized by Field emission scanning electron microscope (FESEM) with EDX in CARLZEISS SIGMA version. Raman spectra of the samples have been recorded using HORIBA JOBIN YUON HR (800) spectrometer. The dried powder was subjected to Fourier Transform Infrared spectroscopy (FTIR) analysis has been recorded using SHIMKADZU IRAffinity – 1 instrument. A JASCO-UV–Vis-NIR Spectrophotometer (JASCO V570) was used for the spectrometric analysis to conform Ag nanoparticles formation.

Result and Discussion

The XRD patterns of the synthesized silver nanoparticles are shown in Figure.1 at different annealed temperature i.e., (100, 150 & 200) °C. In all the patterns, the four diffraction peaks obtained at 2θ (degrees) of 37.97°, 44.16°, 64.35° and 77.29° are respectively indexed as the (111), (200), (220) and (311) planes of Silver. All the four diffraction peaks in the 2θ range completely corresponds to the face-centered cubic (FCC) crystalline nature of Silver with lattice constants $a = 4.068\text{\AA}$ and are in good agreement with those on the database of JCPDS card No. 87-0719. The sharpness of the diffraction peaks suggests that the product is well crystallized. The average crystallite size of silver is calculated using Debye Scherrer's equation

$$D = \frac{K\lambda}{\beta \cos\theta}$$

where, D is the grain size, K is a constant taken to be 0.94, λ is the wavelength of the x-ray radiation, β is the full width at half maximum and θ is the angle of diffraction. The mean sizes of obtained silver nano-chip like structure at different annealing temperature were found to be 35, 42 and 50 nm for (100, 150 & 200) °C respectively. These observations indicate that the particle size of Ag nano-chip like structure increase with increasing annealing temperature and the crystallite size improvement is responsible for the sharpness in the diffraction intensity peaks from (a) to (c) in Figure 1.

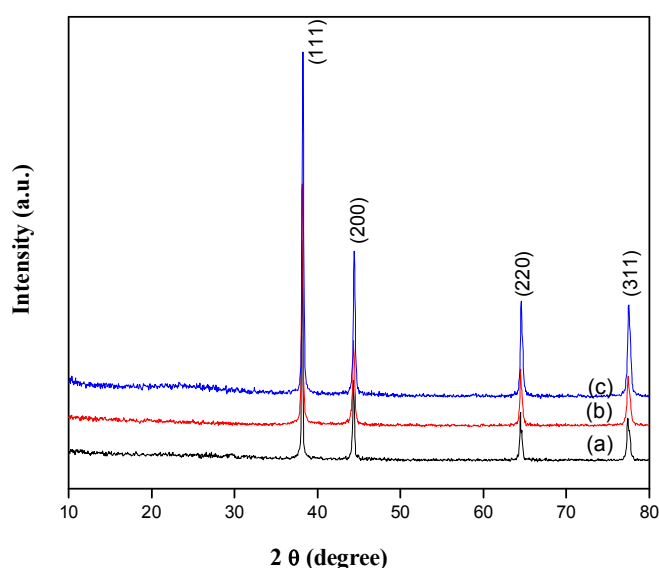


Figure 1. X-ray Diffraction pattern of Ag nano-chip like structure (a) 100°C (b) 150°C and (c) 200°C annealed samples

The size and shape of Ag nano-chip like structure has been studied using field emission scanning electron microscopy (FESEM). Figure 2 (a-c) shows the FESEM image of Ag nano-chip like structure annealed at different temperatures of (100, 150 & 200) °C is recorded. The FESEM investigations of all samples revealed that the crystallites are of nanometer in size. The formation of Ag nano-chip like structure as well as their morphological dimensions in the FESEM analysis demonstrated that the size at different annealing temperature are 35, 42 and 50 nm with inter-particle distance. Thus, it shows the particles size increasing due to the effect of annealing temperature through the FESEM image. The purity of the prepared sample was determined by energy dispersive X-ray spectrum (EDX) of the Ag nano-chip like structure. As seen in figure 3 the purity of Ag nano-chip like structure is about 83%. Also from figure 2 it can be concluded that the synthesized Ag nano-chip like structure have cylinder morphology.

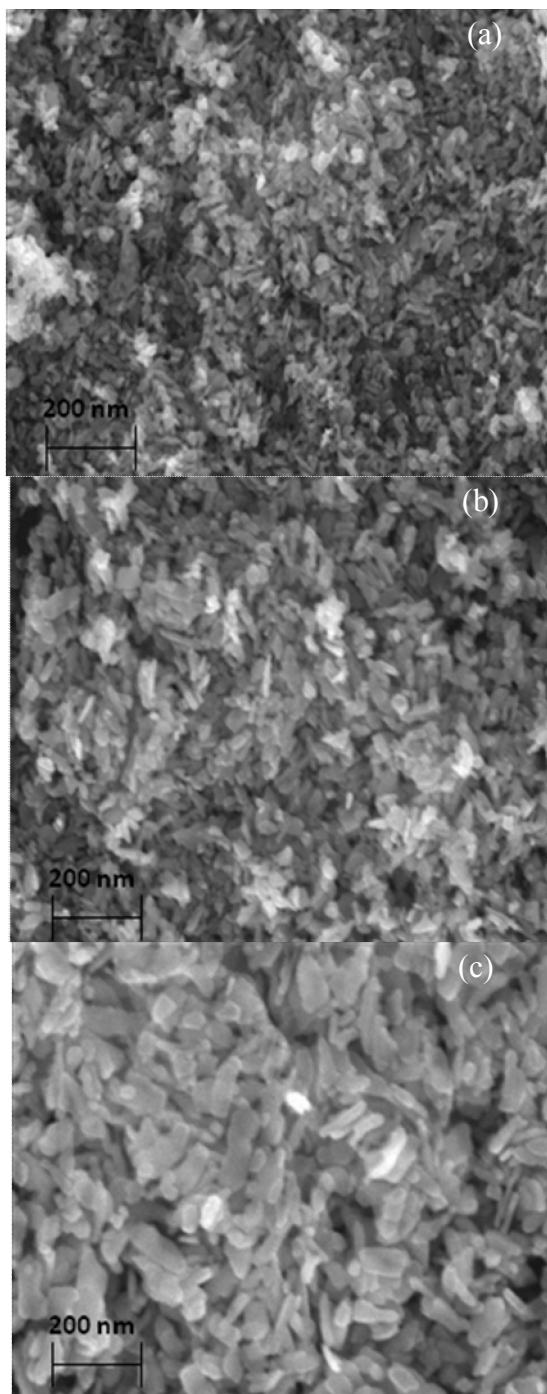


Figure 2. SEM images of Ag nano-chip like structure (a) 100°C (b) 150°C and (c) 200°C annealed samples

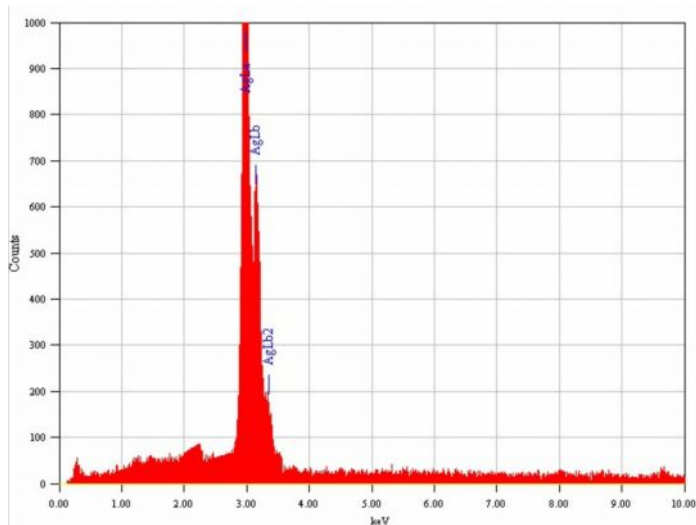


Figure 3 EDAX of Ag nano-chip like structure

Figure 4 shows the spectra of Ag nano-chip like structure prepared at different annealing temperature of (100, 150 & 200) °C. The results of FTIR analysis of this study show different stretches of bonds at different peaks; i.e., absorbance bands in the region scanning from 450 to 3422 cm^{-1} . The band seen at 3422 cm^{-1} and 1616 cm^{-1} were very broad and strong at the annealed temperature of 200 °C (Fig. 4c) while in figure 4a & 4b (100 & 150) °C, there was broad and not very strong band due to the less particle size and these two bands was assigned to hydroxyl (OH) group. The bands corresponding to CH_2 asymmetric stretching vibration occurs at around 2928 cm^{-1} was narrow and very strong for the pH 6.0 (fig. 4c). A weak band at 907 cm^{-1} was aliphatic group and weak absorption bands at 2781 cm^{-1} was represent the aldehyde group bending vibrations shown only at annealed temperature of 200 °C (fig. 4c) but not shown in other. A prominent and very sharp peak is observed at 1404 and 1315 cm^{-1} which was concluded to be due to the (NO_3^-) nitrate ions when compared with the FTIR spectrum for silver nitrate, as shown in figure 4. The weak peak seen at 760 cm^{-1} corresponds to the anti symmetric stretching vibration. The weak absorption band seen at 498 cm^{-1} and the narrow and strong at 451 cm^{-1} are corresponds to the silver nanoparticles in all the annealed temperature (Figure 4a to 4c). Almost all these above said The FTIR spectrum peaks are match with those for the silver nano-chip like structure exist.

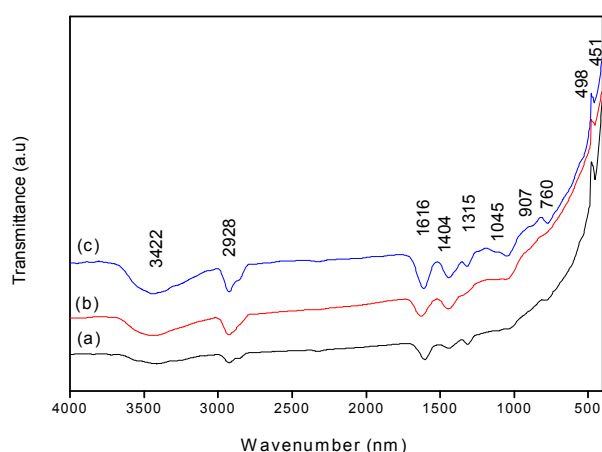


Figure 4 FTIR of Ag nano-chip like structure (a) 100°C (b) 150°C and (c) 200°C annealed samples

Figure 5 shows the SERS – Surface Enhancement Raman Spectroscopy in order to study the behavior of Ag nano-chip like structure at different annealing temperature. In that there was plentiful peaks which demonstrating that there was a remarkable Raman enhancement effect on the Ag nano-chip like structure. The peaks around 239, 710, 815, 930, 1050, 1282, 1400 and 1460 cm^{-1} were enhanced to the Ag nano-chip like structure by the bending of CH_2 , NO_3^- , CH . Comparing the Raman peaks of figure 5a to 5c the intensity was increasing from lower annealing temperature to higher annealing temperature. This shows that the particle size

was increasing with the increasing of annealing temperature. These results suggested that the Ag nano-chip like structure have a good enhancement effect. The results show that the formed Ag nanoparticles have a stable highly enhanced Raman signal for all the different annealing temperatures of (100, 150 & 200) °C.

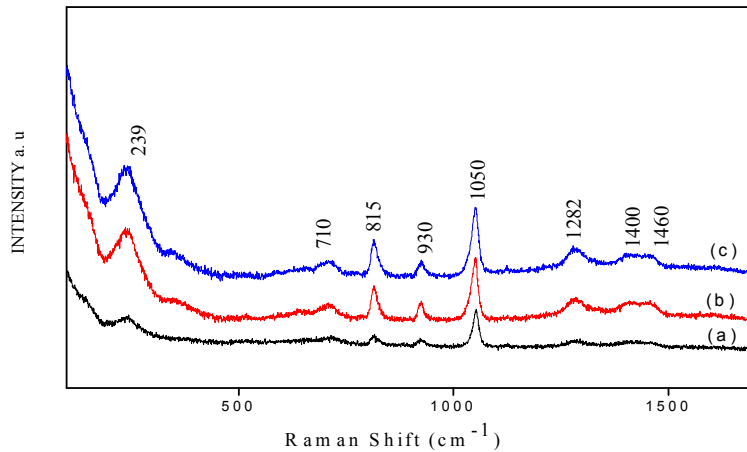


Figure 5 Raman Spectrum of Ag nano-chip like structure (a) 100°C (b) 150°C and (c) 200°C annealed samples

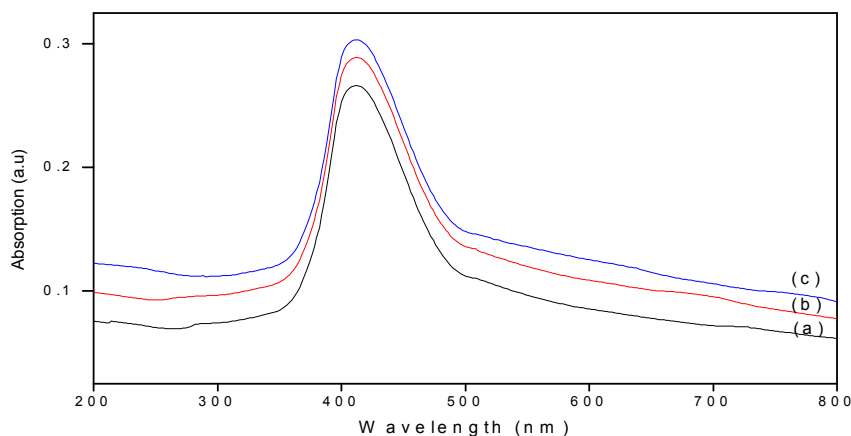


Figure 6 UV - spectrum of Ag nano-chip like structure (a) 100°C (b) 150°C and (c) 200°C annealed samples

Figure 6 shows the optical absorption spectra of Ag nano-chip like structure grown at different annealing temperature. In the UV-Vis absorption spectrum the Ag nano-chip like structure show a surface absorption spectrum, a strong, broad peak located at 412 nm. The absorption edge of Ag nano-chip like structure increases with increasing the annealing temperature. Therefore, nano confinement was increasing with the increasing of absorption edge. The crystallite grows during the modulation of annealing temperature and also the band gets broaden corresponding with annealing temperature due to the transition from small sized to bulk-sized particles. The optical band gap energy is calculated using the following equation

$$\alpha h\nu = A(E_g - h\nu)^n$$

where α is the absorption co-efficient, t is the frequency of the incident radiation, A is a constant, E_g is the band gap of the material, h is the Plank's constant and n is equal to $\frac{1}{2}$ for direct allowed transition. The band gap energy has been calculated and was found to be 3.01 eV for Ag nano-chip like structure grown at different annealing temperatures of (100, 150 & 200) °C respectively. Thus, there was no change in the band gap but there was shift in absorption while the change in different annealing temperatures. The size is also nearly consistent with the size obtained from XRD observations (Table 1).

Table 1 Relates Ag nano-chip like structure size with annealing temperature

Annealing Temperature (°C)	Nano Size from XRD (nm)	Band gap energy (eV)
100	35	3.01
150	42	
200	50	

Conclusions

The Silver nano-chip like structures have been prepared by chemical precipitation method at different annealing temperatures of (100, 150 & 200) °C. The different sized and shaped nano-chip like structure formed while changing the annealing temperature of the prepared particles. X-ray diffraction pattern of the prepared samples reveal the formation of Ag nano-chip like structure with grain size lying in the range of 35–50 nm. The average grain size of Ag nano-chip like structure from FESEM was found to lie in the range of 35–50 nm. It was found that annealing temperature has a significant influence on both size and crystallinity of the nano-chip like structure. The Raman spectra exhibited peaks corresponding to the Silver. The band gap of Ag nano-chip like structure is found to constant and increase in the absorption edge is observed to appear with increasing annealing temperature.

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