

# Synthesis of Ag nanoparticles on polymer surface: 150 keV Ar ion irradiation of Ag-PVC bilayer

Jai Prakash<sup>1\*</sup>, A. Tripathi<sup>2</sup>, G. B. V. S. Lakshmi<sup>2</sup>, V. Rigato<sup>3</sup>, Jalaj Tripathi<sup>1</sup>, D. K. Avasthi<sup>2</sup>

<sup>1</sup>Department of Chemistry, M M H College Ghaziabad-201001(Ch. Charan Singh University, Meerut), India

<sup>2</sup>Inter University Accelerator Centre (IUAC), Aruna Asaf Ali Marg, New Delhi 110067, India

<sup>3</sup>INFN Laboratori Nazionali di Legnaro, via Dell' Università 2, 35020 Legnaro, Padova, Italy

\*Corresponding author. E-mail: jai.gupta1983@gmail.com, jpg1983@gmail.com

Received: 16 March 2012, Revised: 22 July 2012 and Accepted: 26 July 2012

## ABSTRACT

Thin metal films of Ag (~10 nm) deposited on spin coated PVC film on quartz substrate, were irradiated with 150 keV Ar ions at fluences varying from  $5 \times 10^{15}$  to  $5 \times 10^{16}$  ions/cm<sup>2</sup> and characterized with Rutherford backscattering spectrometry (RBS), atomic force microscopy (AFM), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) techniques. RBS spectra show sputtering of the Ag film. As a result of ion irradiation, isolated Ag nanoparticles are formed on the surface. The size and size distribution of Ag nanoparticles are found to be dependent on ion fluence. Contact angle measurements were carried out to study the hydrophilic nature of the surface at varying fluences. Results are explained in the framework of sputtering from the surface due to dense collision cascade resulting from Ar ion and Ag/PVC film interaction. Copyright © 2013 VBRI press.

**Keywords:** Metal-polymer (Ag-PVC); Ion irradiation; Collision cascade; Ag nanoparticles; Contact angle measurement.



**Jai Prakash** is working as Post Doctoral researcher at IM2NP, Faculty of Science and Technology, University of Aix-Marseille, France. He received Ph.D degree from Ch. Charan Singh University, Meerut, India and worked in Materials Science under the project sponsored by Inter University Accelerator Centre (IUAC), New Delhi. He did his masters in Chemistry from CCSU. His research interests include ion implantation, swift heavy ions, thin films, synthesis and characterization of nanomaterials, carbon materials, etc.



**Valentino Rigato** has been working for more than 15 years at the National Institute of Nuclear Physics - National Laboratories of Legnaro (INFN-LNL, Italy), where he is responsible for research projects on advanced materials and interdisciplinary physics with ion beams. He is the scientific coordinator of the two Van de Graaff ion accelerators of LNL entirely dedicated to cross disciplinary experiments. His experience ranges from surface and thin films characterization and ion

surface interaction studies to thin films processes for the synthesis of nano-structured materials and multi-layers for mechanical, optical and X-ray applications.

## Introduction

Nanostructured noble metals have a strong potential for applications in a broad range of technologies, from nanoelectronics and photonics to sensing and catalysis [1,2]. Formation of noble metal nanoparticles in/on polymer matrix leading to the metal polymer nanocomposite is one of the interesting areas of nanotechnology because of their application in thin film technology [1-3]. Properties of the nanoparticles can be tailored by controlling their size, shape, and distribution. A number of techniques including both physical and chemical roots have been carried out to synthesize noble metal nanoparticles with controllable size, shape, and distribution [4-6].

Synthesis of metal nanoparticles on the surface and embedded in the matrix by thermal annealing (leading to diffusion of metal into polymer matrix) and ion beam irradiation is a promising method as reported in the literature [7,8]. As compared to the thermal annealing, for nanoparticle formation, using a thin metal film, ion beam irradiation provides a unique tool for the synthesis and engineering of nanostructures by depositing highly localized energy both in space (nanometers) and time (picoseconds) [3,9-11]. In case of ion beam with keV

energies, thermal spike is produced through the collision cascade resulting in a molten zone, subsequent sputtering of the target material, formation of holes and craters [12-14]. Therefore, ion beam irradiation seems to be promising size reduction tools and it has been shown that low energy ion bombardment of very thin films leads to the nanoparticles formation on the surface with controlled size and size distributions [5,15]. Hu et. al [9] have reported the formation of spherical and uniform Pt nanoparticles on SiO<sub>2</sub> surface as a result of 800 keV Kr ions irradiation of thin Pt film. Formation of self assembled Au nanoparticles with controlled size and distributions is reported using focused ion beam (FIB) irradiation (5 keV Ga<sup>+</sup>) of thin Au films on amorphous carbon films by Zhou et al [5].

In an earlier study [3], we have reported the synthesis of Au nanoparticles at the surface and embedded in carbonaceous matrix by 150 keV Ar ion irradiation of Au-PET bilayer system. In the present study, we report on the synthesis of Ag nanoparticles by 150 keV Ar irradiation of Ag-Polyvinylchloride (PVC) bilayer system. Such noble metal-polymer (Ag-PVC) composite have potential biomedical applications because of antibacterial activity of Ag metal and wide use of PVC as a polymer material in medical devices [16]. The size and size distribution of Ag nanoparticles are shown to be dependent on ion fluence. Contact angle measurements have been carried out to study the hydrophilic response of the bilayer system before and after irradiation at varying fluences.

## Experimental

### Materials and sample preparation

Thin Polyvinylchloride (PVC) films were deposited on quartz substrate using spin-coater. Plasticized PVC powder (from Merck, India) was dissolved in tetrahydrofuran (THF) [Analytical Reagent grade from Merck, India] and solution was kept for homogeneous dissolution on magnetic stirrer at 50 °C for 5 hrs. After clear solution was prepared, it was spin coated on quartz substrate at 1100 rpm. 10 nm thin Ag films were deposited on PVC films by electron beam evaporation at Inter University Accelerator Centre (IUAC), New Delhi. Deposition was performed in an ultra-high-vacuum chamber (UHV) with base pressure of ~10<sup>-6</sup> torr and film thickness was monitored using quartz crystal monitor.

### Ion beam parameters and ion irradiation

Ion beam irradiation was carried out with 150 keV Ar ions at varying fluences of 5×10<sup>15</sup>, 1×10<sup>16</sup>, and 5×10<sup>16</sup> ions/cm<sup>2</sup> with a current of 0.5 μA/cm<sup>2</sup> using the low-energy ion beam facility (LEIBF) at IUAC, New Delhi. Nuclear energy loss (S<sub>n</sub>) of 150 keV Ar ion in Ag and plasticized PVC is ~82 and ~35 eV/Å respectively and projected range is ~62 and ~200 nm respectively as calculated by the SRIM (stopping and range of ions in matter) code.

### Characterization techniques

Samples were characterized by Rutherford backscattering spectrometry (RBS) with 2MeV 4He<sup>+</sup> ions, using AN2000 Van de Graaff accelerator at INFN-LNL, Padova. He<sup>+</sup> ions

were impinged perpendicularly to the surface and backscattered ions were detected at scattering angle of 160° to beam direction. Surface morphologies were studied by atomic force microscopy (AFM) in the tapping mode with an antimony-doped silicon tip using Nanoscope IIIa SPM and field emission-scanning electron microscopy (FE-SEM) [MIRA\\ TESCAN] at IUAC, New Delhi. Hydrophilic nature of the surfaces were studied before and after irradiation by contact angle measurements with KRUSS easdrop contact angle measuring system (KRUSS GmbH, Hamburg 2005-2008) installed at IUAC, New Delhi.

## Results and discussion

Fig. 1 shows the SEM image and corresponding EDS spectra along with fitted RBS spectrum of the pristine Ag-PVC sample. RBS and EDS analyses show the presence of Ag and polymer components (C and Cl). Fitting of RBS spectra for Ag-PVC deposited on quartz substrate using RUMP simulation shows the estimated thickness ~10 and ~400 nm for Ag and spin coated PVC film, respectively.

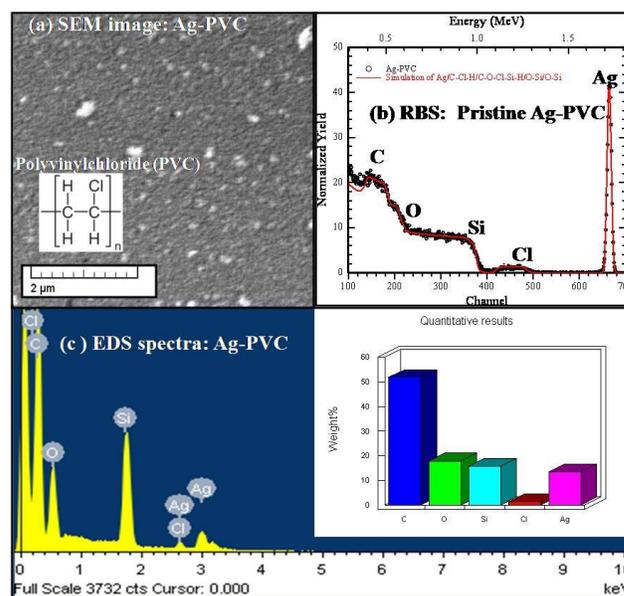


Fig. 1. Ag-PVC bilayer pristine sample on quartz (SiO<sub>2</sub>) substrate (a) SEM image (b) RBS fitted spectra using RUMP simulation code (c) EDS spectrum. RBS spectrum shows the presence of Ag and PVC polymer component (C and Cl), also confirmed by EDS spectra.

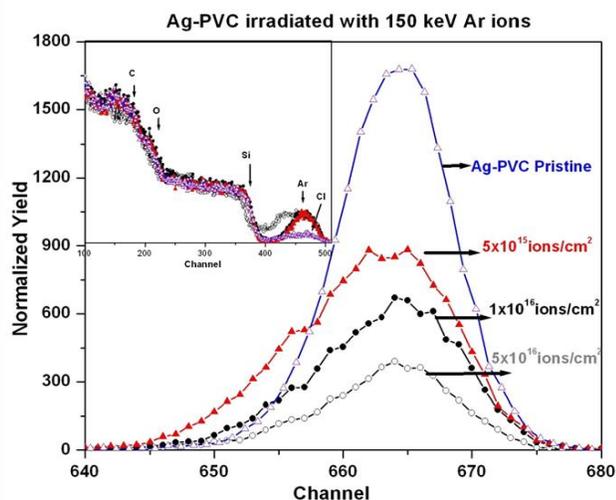
### Effect of ion irradiation on surface

Fig. 2 shows the Rutherford backscattering spectrometry (RBS) spectra of Ag-PVC bilayer system before (i.e., pristine) and after irradiation with 150 keV Ar ions at fluences 5×10<sup>15</sup>, 1×10<sup>16</sup>, and 5×10<sup>16</sup> ions/cm<sup>2</sup>. Comparison of the Ag peak intensity in RBS spectra of the pristine and irradiated samples, indicates the loss of Ag atoms i.e. sputtering of Ag, due to irradiation. Therefore, areal concentration (N<sub>a</sub>) of Ag atoms in the pristine and irradiated samples at the different fluences was calculated from integral counts of the RBS spectra as shown in Fig. 3. As the ion fluence increases, areal concentration of the Ag atoms decreases which confirms sputtering. Inset of Fig. 3 shows the Ag sputtering yield dependence on the Ar ion

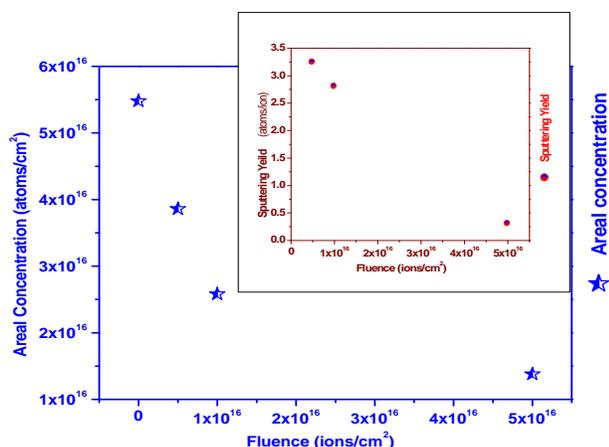
irradiation fluence. Sputtering yield ( $Y$ ) was calculated by using the formula [17]:

$$Y = \Delta N_i / \Delta \Phi$$

where,  $\Delta N_i$  is difference in areal concentration at two fluences whose difference is  $\Delta \Phi$ . Sputtering yield decreases with increasing ion fluence. At the initial fluence of  $5 \times 10^{15}$  ions/cm<sup>2</sup>, estimated sputtering yield is 3.24 atoms/ion and at the high fluence of  $5 \times 10^{16}$  ions/cm<sup>2</sup>, sputtering yield is 0.3 atoms/ion. The reason for decrease in sputtering yield is the reduction in area coverage by Ag on the surface with increasing ion fluence as also shown in AFM micrographs in Fig. 4.



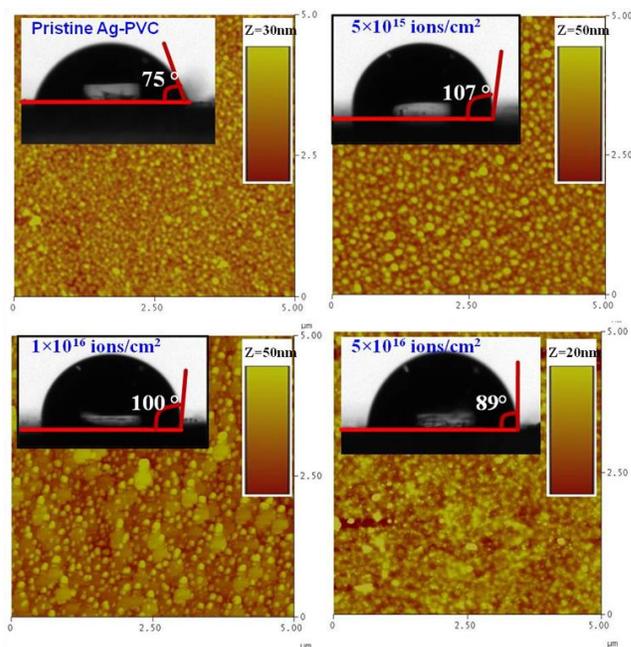
**Fig. 2.** RBS spectra of Ag-PVC pristine and irradiated films with 150 keV Ar ions at different fluence of  $5 \times 10^{15}$ ,  $1 \times 10^{16}$ , and  $5 \times 10^{16}$  ions/cm<sup>2</sup>. Decrease in Ag peak height and tailing in low energy edge indicate the ion beam induced sputtering of Ag and diffusion in matrix.



**Fig. 3.** Areal concentration of Ag in Ag-PVC pristine and irradiated samples with 150 keV Ar ions at different fluences of  $5 \times 10^{15}$ ,  $1 \times 10^{16}$ , and  $5 \times 10^{16}$  ions/cm<sup>2</sup>. Inset shows the sputtering yield of Ag at the different ion fluence. Areal concentration and sputtering yield of Ag decrease with increasing Ar ion fluence.

Contact angle measurements were performed on pristine and irradiation samples and it is found that the contact angle of the pristine and irradiation surfaces varies with the surface roughness of the samples as summarized in

**Table 1.** Fig. 4. Shows the AFM images and corresponding snapshots of contact angle measurements of the pristine and irradiated samples. It can be seen that pristine surface is not very smooth with the rms surface roughness of 5 nm and contact angle is 75°. After irradiation with 150 keV Ar ions at the fluence of  $5 \times 10^{15}$  ions/cm<sup>2</sup>, surface roughness is increased to 10.5 nm and contact angle is also increased to 107°. With increasing fluence, surface becomes smoother with smaller Ag nanoparticles (as shown in AFM images) due to sputtering as confirmed from RBS analysis and hence the contact angle also decreases as can be seen in Table 1.



**Fig. 4.** AFM micrographs ( $5 \mu \times 5 \mu$ ) and contact angle measurements of Ag-PVC pristine sample and sample irradiated with 150 keV Ar ions at the fluence of  $5 \times 10^{15}$ ,  $1 \times 10^{16}$ ,  $5 \times 10^{16}$  ions/cm<sup>2</sup>.

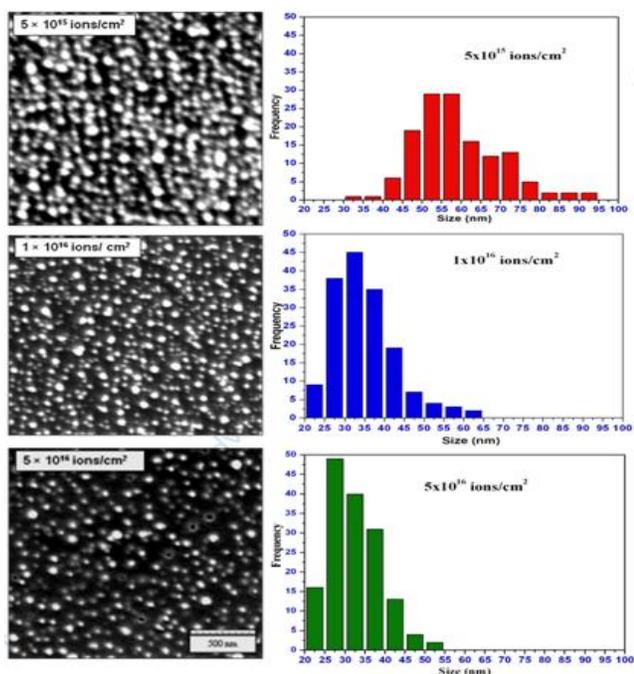
**Table 1.** Roughness and contact angle measurements on the pristine and irradiated samples.

Samples	Roughness (~nm)	Contact angle (°)
pristine	5	75
$5 \times 10^{15}$ ions/cm <sup>2</sup>	10.5	107
$1 \times 10^{16}$ ions/cm <sup>2</sup>	9	100
$5 \times 10^{16}$ ions/cm <sup>2</sup>	3	89

*Effect of ion fluence on size and size distribution of Ag nanoparticles*

Fig. 5 shows the SEM pictures of Ag-PVC samples irradiated with 150 keV Ar ions at different fluences,  $5 \times 10^{15}$ ,  $1 \times 10^{16}$ , and  $5 \times 10^{16}$  ions/cm<sup>2</sup>. SEM pictures show that with increase in ion fluence, there is reduction in metal coverage area on the surface and also in size of the Ag nanoparticles formed on the surface. Average size of the Ag

nanoparticles decreases with increasing ion fluence as shown in **Fig. 6** and at the high fluence of  $5 \times 10^{16}$  ions/cm<sup>2</sup>, isolated and spherical Ag nanoparticles are formed. Size distributions of the Ag nanoparticles at varying fluences has been plotted from the corresponding SEM images and also shown in **Fig. 5**, which indicate large variation in size distribution as a function of ion fluences. The size distribution becomes narrower with the increasing ion fluence. For the ion fluence of  $5 \times 10^{15}$  ions/cm<sup>2</sup>, most of the nanoparticles have a size of 50-60 nm with a broad distribution of  $\pm 20$  nm and about 4% of the particles have sizes between 80-95 nm. On the other hand, at ion fluence of  $1 \times 10^{16}$  ions/cm<sup>2</sup>, the size and size distribution tends to be sharper. The most probable size of the nanoparticles is 30-35 nm with distribution of  $\pm 10$  nm and about 5% of the particles are in the range of  $> 50$  nm. For the higher fluence of  $5 \times 10^{16}$  ions/cm<sup>2</sup>, the great majority of particles have a size of 25-30 nm and only about 2% of the particles are in the range  $> 45$  nm.

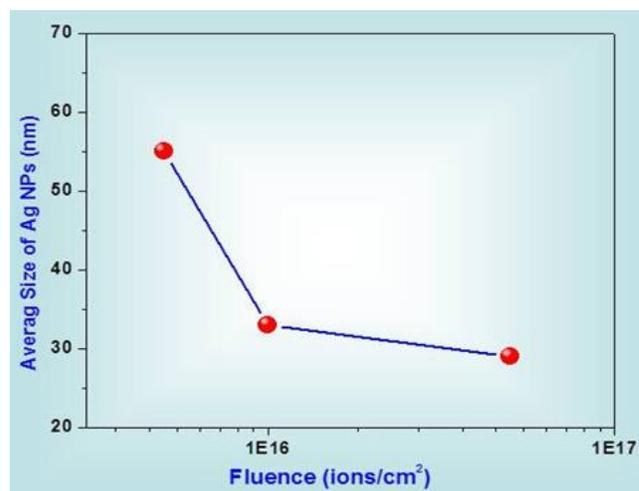


**Fig. 5.** SEM pictures and corresponding size distribution graph of Ag nanoparticles as observed on the surface of Ag-PVC samples after irradiation with 150 keV Ar ions at different fluences of  $5 \times 10^{15}$ ,  $1 \times 10^{16}$ , and  $5 \times 10^{16}$  ions/cm<sup>2</sup>. Size of the Ag nanoparticles decreases and size distribution becomes narrower with increasing ion fluence.

### Mechanism

Recently, we have reported the formation of Au nanoparticles by Ar ion induced thermal spike due to collision cascade, producing molten zones leading to dewetting of the Au film on PET through sputtering and crater formation [3]. Singh et al [18] have synthesized Au nanostructures on glass by low-energy ion irradiation (50 keV Si) of a thin Au film and explained on the basis of surface instability due to ion beam sputtering and surface diffusion. Paszti et. al [15] have observed the decrease in size of island Ag film on C and SiO<sub>2</sub> substrate with increasing ion fluence and formation of nanoparticles on the surface as a result of Ar ion sputtering and also

interface mixing as a secondary effect. Similarly, in the present case, it is considered that Ag nanoparticles are formed due to collision cascade induced molten track formation and sputtering induced by Ar ions. The size of the Ar ion induced molten zone in Ag target can be estimated on average by  $R_m^0 = (f_d^0 / \pi n_0 \epsilon_m)^{1/2}$  [3,10], where,  $f_d^0$  is the deposited energy per unit length by the ion along the ion path,  $n_0$  is the atomic density of metal, and  $\epsilon_m$  ( $\approx 3kT_m$ ) is the average energy of the atom at the melting point, where  $T_m$  is the melting temperature of the metal. For Ag,  $n_0 = 5.85 \times 10^{22}$  atoms/cm<sup>3</sup>,  $T_m = 1235$  K and for 150 keV Ar ions in Ag,  $E_{d=}^0 = 82$  eV/Å as estimated using SRIM code. The estimated average molten ion track size in Ag induced by 150 keV Ar ions in present case is  $\approx 4$  nm and estimated overlapping fluence is  $\sim 10^{13}$  ions/cm<sup>2</sup>. With continuing sputtering, particle size decreases with increasing ion fluence. Due to the dominance of sputtering effects, smaller Ag nanoparticles are sputtered away at higher fluence [5] leading to narrower size distribution.



**Fig. 6.** Average size of the Ag nanoparticles Vs increasing ion fluence. Average size of nanoparticles decreases with increasing ion fluence.

The contact angle measurement on the surface depends on surface properties like roughness and chemical composition of materials at the surface [19,20]. In the present case, the surface becomes polymer rich with isolated Ag nanoparticles and size of nanoparticles decreases with increasing ion fluence because of sputtering of Ag. Sputtering also affects roughness which reduces the contact angle. The irradiated polymer surface may contain free radicals and ions on the surface [21-23] which along with the relative coverage area of the two materials present on the surface influence the contact angle [19,20]. The decrease in contact angle of the surface is due to change in the surface composition and surface roughness.

### Conclusion

Isolated Ag nanoparticles were synthesized on the surface by 150 keV Ar ion irradiation of Ag-PVC bilayer films. Size of the Ag nanoparticles decreases with increasing ion fluence. The size distribution also decreases and becomes narrower with increasing ion fluence. Contact angle measurement of the surface before and after irradiation shows the surface roughness dependent and reveals that

surface tends to hydrophilic with decreasing size of Ag nanoparticles and increasing ion fluence. Results are explained in view of sputtering and crater formation in Ag film and disorder created in polymer, through molten zones due to thermal spike induced by 150 keV Ar ions.

### Acknowledgements

Author (JP) would like to acknowledge Council of Scientific and Industrial Research (CSIR), New Delhi for providing senior research fellowship (CSIR-SRF) and IUAC, New Delhi for providing LEIBF, all the experimental facilities and funding supports. Authors would also like to thank Dr. K. Asokan, Dr. Fouran Singh, Dr. Pravin Kumar, Mr. S. A. Khan, Mr. Pawan Kulriya, Mrs. Indira Sulania, Mr. Uday B. Singh, and Ms. Srashti Gupta for discussion and their help during the experiments and characterizations of the samples.

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