

Influence of Oxygen ions irradiation on Polyaniline/Single Walled Carbon Nanotubes nanocomposite

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ARTICLE INFO

Article history:

Received 2 June 2016

Received in revised form 12 July 2016

Accepted 27 July 2016

Available online xxx

Keywords:

Nanocomposite

Swift Heavy Ion Irradiation

Atomic Force Microscopy

Fourier transformed infrared spectroscopy

X-Ray Diffraction

ABSTRACT

Influence of Oxygen ions (100 MeV) irradiation on Polyaniline (PANI)/Single Walled Carbon Nanotubes (SWNTs) nanocomposite was studied in the present investigation. PANI/SWNTs nanocomposite was synthesized by electrochemical Cyclic Voltammetry technique. Nanocomposite was exposed under SHI irradiation of Oxygen (100 MeV) ions for three different fluences such as 1×10^{10} ions/cm², 5×10^{10} ions/cm² and 1×10^{11} ions/cm². The SHI irradiated PANI/SWNTs nanocomposite was investigated by using morphological (AFM), structural (XRD) and spectroscopy (FTIR) characterization. AFM study exhibits effects of SHI irradiation on morphology of the nanocomposite and root mean square roughness of the nanocomposite is observed to be decreased as fluence was increased. The FTIR absorption spectrum exhibits formation of new functional sites with the increase in intensity of absorption peaks, due to SHI irradiation. X-Ray Diffraction studies show a gradual decrease in the crystalline nature of the nanocomposite upon irradiation.

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1. Introduction

After the revolutionary discovery and development of electrically conductive polymers (CPs) by Heeger, MacDiarmid and Shirakawa the scientific world has started to look forward these materials with potential applicability. Ease of synthesis, low cost, tunable doping/dedoping properties, modifiable electrical conductivity have proven CPs as challenging, competitive and reliable materials in the development of many application areas (Shirsat et al., 2009, 2007; Kharat et al., 2007; Gade et al., 2006). However, while dealing with the device application, polymer stability is one of the important key factors. Incorporation of carbon nanotubes (CNTs) in CPs could come up with extraordinary throughputs (Meng et al., 2009; Yao et al., 2010; Spitalskya et al., 2010; Sahoo et al., 2010). In this contest many research groups have investigated efficient role of CNTs exhibiting synergistic properties of CP/CNTs nanocomposite. CP/CNTs nanocomposite exhibits extraordinary properties such as high mechanical strength, electrical conductivity, rheological properties, thermal conductivity, thermal stability (Mohammad Moiruzzaman and Winey, 2006) etc. ultimately resulting in enhanced thermo-electric performance (Zhang and Song, 2009), sensing capability, electrochemical capacitive performance (Xiaolei et al., 2005; An et al., 2004; Nikzad et al., 2012; Waltman and Bargon Can., 1986) etc. of the devices. Elec-

trochemical synthesis is one of the easiest techniques adopted for the synthesis of nanocomposite. The resulting nanocomposite shows good adhesion and electrical contact to the electrode surface. The control over potential/current enables in deciding thickness, morphology and conductivity of the nanocomposite (Paramo-Garcia et al., 2012).

Swift Heavy Ion (SHI) irradiation plays significant role in tailoring intriguing properties of the nanocomposite. SHI irradiation, due to electronic and nuclear excitations would cause significant changes in the properties of materials which will be important for device applications. Different nanocomposites with various organic inorganic materials such as poly(vinylidene fluoride-hexafluoropropylene) (Kumar et al., 2010), ZnO/PMMA (Sharma et al., 2011), silica-metal (Pivin et al., 2009), ZnO-CuO (Kuriakose et al., 2015), Poly(vinylidene fluorides propylene/layered silicate) (Tiwari et al., 2009), copper/poly-methyl methacrylate (Kishore et al., 2013), PPy/SnO₂ (Sarmah and Kumar, 2010) were exposed to SHI irradiation for the investigation of many characteristic properties such as enhanced ionic conductivity, photo-luminescence, photocatalytic activities, piezoelectric β -phase transition etc. There are some reports on Polyaniline, Polyaniline nanofibres irradiated with different ions with various energies O⁷⁺ (90 MeV, 100 MeV, 80 MeV) (Kumar and Banerjee, 2013; Ali et al., 2013a; Chandra et al., 2009), Ni¹²⁺ (120 MeV, 160 MeV) (Hazarika et al., 2013; Ali et al., 2013b), C⁵⁺ (40 MeV) (Kumar et al.) at different fluences which results into remarkable structural, conformational and morphological changes. This has ultimately resulted into enhancement in their performance and properties such as conductivity, electro-

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chemical stability, sensitivity, crystallinity, solubility, porosity, density etc. (Ghosh et al., 2013). Sensors based on CPs have been widely studied to achieve lowest explosive levels of hazardous analysts such as various volatile organic compounds, heavy metal ions etc. (Kadam et al., 2010; Srivastava et al., 2006; Rao et al., 2007; Huang et al., 2003). Significant improvement in the sensing capability of the backbone is prominently decided by the surface morphology i.e. roughness of the same (Srivastava et al., 2006). Present investigation deals with the electrochemical synthesis of Polyaniline (PANI)/Single Walled Carbon Nanotubes (SWNTs) nanocomposite. Further, nanocomposite was exposed to SHI irradiation by 100 MeV Oxygen ions with different fluences such as 1×10^{10} ions/cm², 5×10^{10} ions/cm² and 1×10^{11} ions/cm² and influence of SHI irradiation was investigated by using AFM, XRD and FTIR.

2. Experimental details

A single compartment, three cell electrode assembly was used for the synthesis of PANI/SWNTs nanocomposite, using the Cyclic Voltammetry technique. Planar Stainless Steel (SS) substrate ($20 \times 10 \times 0.50$ mm) was used as working electrode, planar platinum ($20 \times 10 \times 0.25$ mm) was used as counter electrode and saturated Ag/AgCl was used as a reference electrode. In electrolyte preparation, COOH functionalized SWNTs (purchased from Nanoshel LLC -Wilmington, DE, USA) were suspended in laboratory grade Sodium dodecyl benzene sulphonate (SDBS). SWNTs suspension, ultra-sonicated for four hours and further after adding to the electrolyte solution (0.1 M aniline monomer and 0.2 M sulfuric acid – H₂SO₄) was kept stirred for twenty minutes. Nanocomposite was synthesized by applying dynamic potential in the range 0.1–1 V. The scan rate during deposition was of 0.1 V/s. A SHI irradiation experiment was carried out at the Inter University Accelerator Center (IUAC), New Delhi,

India utilizes 15 UD Tandem Pelletron Accelerator in Material Science beam line under high vacuum (5×10^{-6} Torr). PANI/SWNTs nanocomposites were subjected to 100 MeV Oxygen ion SHI irradiation with fluences 1×10^{10} ions/cm², 5×10^{10} ions/cm² and 1×10^{11} ions/cm². Morphological, structural and spectroscopic investigations were carried out by Atomic Force Microscope (AFM), X-Ray Diffraction (XRD) and Fourier Transformed Infrared Absorption Spectroscopy (FTIR). Topographic images recorded for surface morphology was obtained from Atomic Force Microscope (AFM) - PARK system – XE7. The Fourier Transformed Infrared Absorption Spectral investigation was carried out using Bruker - Alpha spectrophotometer.

3. Results and discussion

3.1. Synthesis of PANI/SWNTs nanocomposite

Electrochemical synthesis of PANI/SWNTs nanocomposite was carried out using a Cyclic Voltammetry technique. Fig. 1a shows the Voltammogram recorded during synthesis of nanocomposite. Voltammogram exhibits redox current peaks roughly near at 0.2 V and 0.6 V respectively, with the increase in the current intensity at potential region with increase in number of cycles. This indicates electroactive nature of nanocomposite, where aniline is used to solubilize SWNTs via formation of donor-acceptor complex (Wu and Lin, 2006). The formation mechanism of PANI/SWNTs nanocomposite with tubular nanostructure is believed to involve strong interaction between aniline monomer and functionalized SWNTs caused by the presence of π - π^* electron interaction between SWNTs and aniline monomer as well as the hydrogen bond interaction between the carboxyl groups of functionalized SWNTs and the amino groups of aniline monomers. This strong interaction attributes to the fact that the aniline monomer is adsorbed onto the surface of SWNTs which serve as a core and self-as-

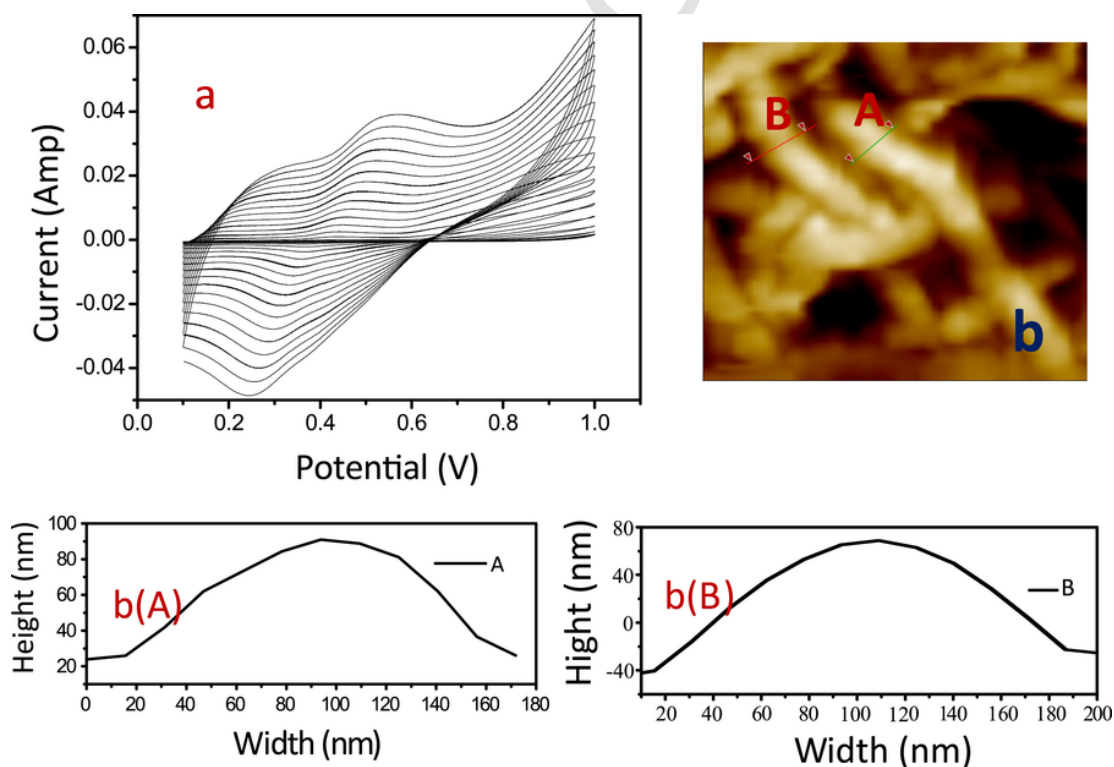


Fig. 1. a – Cyclic voltammogram recorded during electrochemical synthesis of PANI/SWNTs nanocomposite; b – line profiling of nanocomposite for width determination A (green line) and B (red line). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

sembly template during the formation of the tubular nanostructure (Tarushee and Devendra, Kumar). The wrapping of conducting polymer onto the surface of SWNTs was investigated by AFM, shown in following Fig. 1b (A and B). The line profiles (green-A, red-B) were taken for the determination of the width of the tubular nature of the nanocomposite. It is observed that it is having width ~ 160 nm. The resulting conducting polymer based nanostructure could greatly improve diffusion since it has a much greater surface area (Singh et al., 2010). Therefore, it could be utilized as a sensing platform for determination of different organic/inorganic analyte species.

3.2. Morphological investigations

The surface morphology of PANI/SWNTs nanocomposite was studied by ex situ AFM in non-contact mode. The area of $2 \times 2 \mu\text{m}^2$ was recorded for the topographic surface analysis and considered as the whole nature of the nanocomposite film. Fig. 2 shows the topographic images of pristine and 100 MeV Oxygen ions SHI irradiated PANI/SWNTs nanocomposite. In Fig. 2a–d shows the 2-dimensional (2D) topography of the pristine and SHI irradiated nanocomposite, corresponding Fourier Transformed (FT) images are shown in insets which depicts the obvious presence of a characteristic wavelength, which gets attenuated at 1×10^{10} ions/cm² fluence of the SHI irradiation. Each AFM image was analyzed in terms of surface average roughness i.e. root mean square roughness. The root mean square (RMS) roughness of the nanocomposite were evaluated based on data obtained from the scanned area of nanocomposite film. The observed values of roughness are found to get decrease with an increase in the fluence of SHI irradiation (tabulated in Table 1). The related smoothness is probably due to defect enhanced surface diffusion (Rao et al., 2007). Fig. 2i–iv shows the corresponding 3-dimensional (3D) view and line profile of the same shown in Fig. 3, providing the height dimensions of the pristine and SHI irradiated nanocomposite films. These morphological modifications illustrate the interaction of SHI irradiation with nanocomposite. The high energy ions penetrate deep into the material producing the disordered zone along its trajectories.

During interaction with material it loses its energy mainly by two ways, elastic process (nuclear energy loss) and inelastic process (electronic energy loss). In case of high energy range i.e. 10 MeV and above the electronic energy loss is dominant. And these electronic excitations bring out the modifications in the material (Kanjilal, 2001).

3.3. Spectroscopic investigations

The Fourier transformed infrared absorption spectroscopy was used for the study of 100 MeV Oxygen SHI irradiation induced effects on physical and chemical characteristics of PANI/SWNTs nanocomposite. The absorption spectra was recorded in the range $500\text{--}4000 \text{ cm}^{-1}$ and is as shown in Fig. 4. The FTIR spectra of SHI irradiated PANI/SWNTs nanocomposite, shows additional peaks which indicate that there are new functional groups associated with typical bands. Also, it was found that intensity decreases due to irradiation, which may be due to the formation of free radicals that leads to cross linking or degradation of the polymer (Kumar et al., 2012). The peak at around $\sim 1590 \text{ cm}^{-1}$ corresponds to the IR active phonon mode of the SWNTs and the peak at around $\sim 1730 \text{ cm}^{-1}$ corresponds to the stretching mode of the carboxylic acid groups attributed to the presence of carboxylic acid at the walls of SWNTs and responsible for the formation of nanocomposite (Kondawar et al., 2012). The formation of new bands $\sim 3000 \text{ cm}^{-1}$ and $\sim 3500 \text{ cm}^{-1}$ attributed to N–H and C–H stretching have been induced to increase in intensity. It may be concluded that this formation of new bands has brought about cross linking of polymer chain. Moreover, this has led to morphological changes upon irradiation. As it is well studied that conducting polymers are the electrochemically active materials, when there are small perturbations at their surface or in bulk can generate the changes in their electroactivity, and therefore this intriguing property is well probed in study through voltammetric or amperometric techniques (March et al., 2015). The SHI induced modifications will be inducing more electroactive nature of the nanocomposite. The formation of new functional groups in the nanocomposite will be beneficial for functionalization/modification by organic/inorganic compounds having

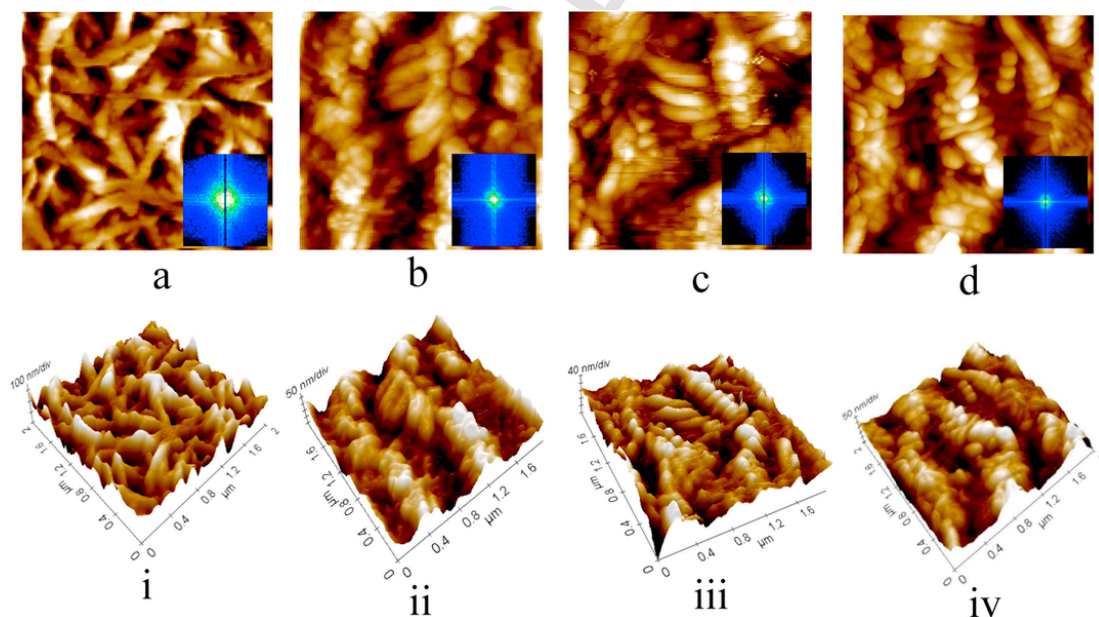
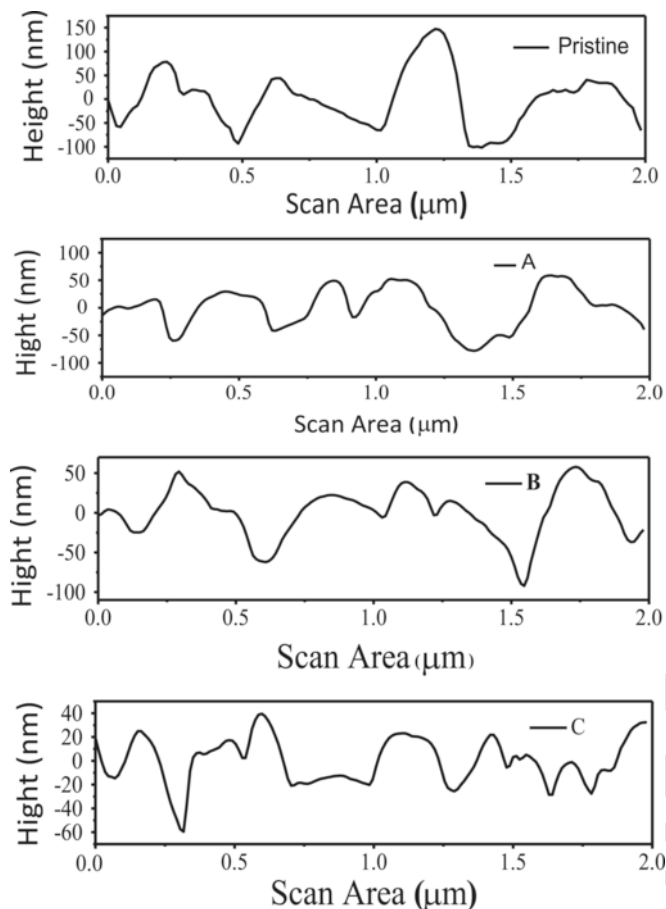


Fig. 2. Topographic images – with 2D and 3D view – of Pristine [a (i)], & SHI irradiated PANI/SWNTs nanocomposite with fluence 1×10^{11} ions/cm² [b (ii)], 5×10^{10} ions/cm² [c (iii)] 1×10^{10} ions/cm² and [d (iv)] insets (a–d) shows FT of same.

Table 1

The root mean square (RMS) roughness of the composite before and after irradiation.

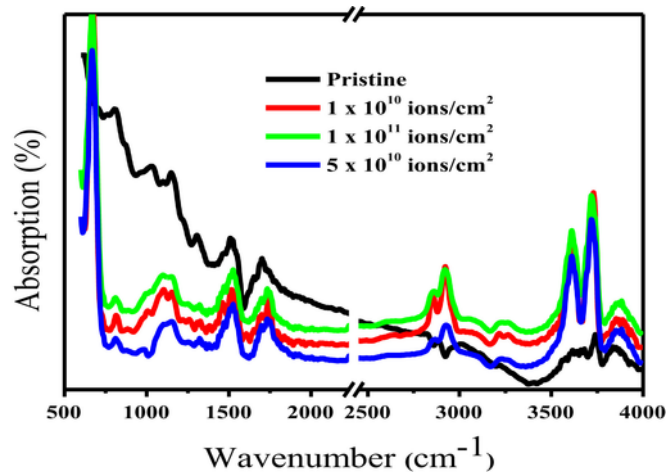
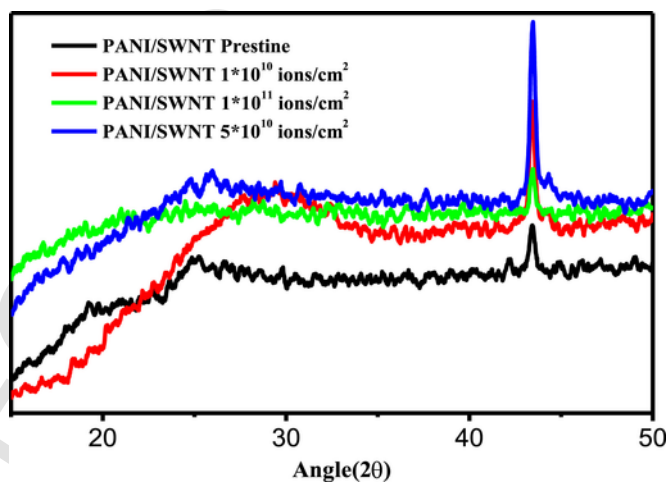
Sr. no	PANI/SWNTs nanocomposite before and after irradiation	RMS roughness (nm)
1.	Pristine	38.275
2.	Fluence of irradiation 1×10^{10} ions/cm ²	32.629
3.	5×10^{10} ions/cm ²	30.369
4.	1×10^{11} ions/cm ²	25.437

**Fig. 3.** Line profile of pristine and irradiated nanocomposite (A- 5×10^{10} ions/cm², B- 1×10^{10} ions/cm² & C- 1×10^{11} ions/cm²).

a ring like structures such as cheating ligands, which could benefit into significant enhancement of selectivity and sensitivity of the electrochemical sensor.

3.4. Structural investigations

The structural analysis of PANI/SWNTs nanocomposite before and after irradiation was carried out using X-Ray Diffraction (XRD) pattern. Fig. 5 shows the diffraction pattern of the nanocomposite before and after irradiation. As shown in the diffraction patterns of pristine and SHI irradiated nanocomposite exhibited broad scattering at 2θ values between 20° and 50° . The peaks observed at $2\theta \sim 25^\circ$ and 43° revealed the presence of PANI and SWNTs. The presence of peak $\sim 25^\circ$ attributed to the periodicity parallel to the polymer chain indicating semi crystalline nature (Abdiryim et al., 2012). In comparison,

**Fig. 4.** FTIR absorbance spectra of pristine and SHI irradiated PANI/SWNTs nanocomposite.**Fig. 5.** X-Ray Diffraction pattern of pristine and SHI irradiated PANI/SWNTs nanocomposite.

of pristine and SHI irradiated nanocomposite it has been observed that there is an increase in intensity of peaks upon irradiation and at the same time there is a gradual decrease in the peak at the Florence 1×10^{11} ions/cm² indicating the amorphous nature of the nanocomposite as a result of defect enhanced surface diffusion.

4. Conclusions

Electrochemical synthesis of PANI/SWNTs nanocomposite films was successfully carried out. Synthesized nanocomposite having tubular nanostructure found to have width ~ 160 nm. The influence of SHI irradiation of 100 MeV Oxygen ions on PANI/SWNTs nanocomposite is quite evident. Morphological, structural and spectroscopic investigations of pristine and SHI irradiated nanocomposite, reveals that defect enhanced surface diffusion has significant influence on the roughness of the nanocomposite. Increase in SHI irradiation of fluence decreases root mean square roughness of the nanocomposite, which results in diffusion of more electronic energy there by formation of new bands. This kind of platform will be more advantageous while modifications of the same, in the perspective of inculcation of selectivity of the sensor devices.

Acknowledgment

Authors are thankful to Inter University Accelerator Center (IUAC), New Delhi, India for providing 15 UD Pelletron facility (under UFR no. 55305) and DST - SERB, New Delhi (Project no.: SB/EMEQ-042/2013) for financial support.

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