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# Effect of UV irradiation on magnetic behavior of reduced graphene oxide decorated with nickel nanostructure

formation by the UV light.

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ARTICLE INFO	ABSTRACT		
A R T I C L E I N F O Keywords: Graphene Composite Magnetism UV-irradiation	Reduced graphene oxide (RGO) decorated with nickel has been synthesized via an in-situ reduction of graphene oxide (GO) and nickel nitrate using NaOH and hydrazine. The starting materials Ni (NO <sub>3</sub> ) <sub>2</sub> and GO were taken in two different ratios and the products formed were designated as RGNi <sub>2</sub> and RGNi <sub>1</sub> . The formation of the composite was confirmed by the appearance of X-ray diffraction peaks at 44.5°, 51.9°, 76.5° corresponding to Ni and at 24.8° and 43.2° for RGO. The RGNi <sub>2</sub> was irradiated with UV light ( $\lambda$ =254 nm) for different durations (2, 6, 12, 24 and 48 h). Intensity ratio of d and g-bands (I <sub>d</sub> /I <sub>g</sub> ) of Raman spectra increases from 1.18 to 1.47 over the duration of irradiation period (2–48 h). The magnetization measurements using the vibrating sample magnetometer (VSM) of these samples reveal their ferromagnetic behavior. The calculated saturation magnetization (M <sub>S</sub> ) value of Ni, RGNi <sub>1</sub> and RGNi <sub>2</sub> is 47.86, 30.56 and 8.25 emu/g respectively and the corresponding coercivity (H <sub>C</sub> ) value is found to be 181, 227 and 296 Oe. The M <sub>S</sub> of RGNi <sub>2</sub> is found to increase to		

#### 1. Introduction

Graphene, a two dimensional array of sp<sup>2</sup>-hybridized carbon atoms has been a topic of interest for more than past six decade since Wallace calculated its electronic structure [1]. However, recently the interest has renewed due to the discovery of new physics [2]. Graphene holds a lot of potential in basic research as well as technological applications due to the fact that, it possesses large surface area, exceptional electrical, mechanical, thermal, optical and magnetic properties. Defects in graphene such as vacancies, disorders, non-hexagonal rings and doped impurities affect various physical, chemical and magnetic properties [3]. The scattering centers are created by these defects, which affect the carrier mobility and electrical conductivity of graphene. Apart from causing deteriorating effects, these defects give rise to some fascinating phenomenon in graphene such as Kondo effect in which local magnetic moments at the defects interact with the conducting electrons at low temperature [4,5]. The defects in graphene have been extensively studied theoretically and experimentally [6]. In most of the experiments high-energy electron beams, alpha radiation, ion bombardment and photo irradiation is generally used [4,5,7,8]. In comparison to all these irradiation sources, photo irradiation is least employed. Photo irradiation such as UV irradiation has a great

potential for inducing defects in graphene by creating electron hole pairs [9].

10.65 emu/g after 48 h of irradiation. This enhancement in the  $M_s(\sim 23\%)$  with irradiation may be due to defect

Magnetic nanoparticles are of great interest owing to their large number of applications in magnetic fluids [10], data storage [11], biotechnology, biomedicine [12], catalysis [13,14], magnetic resonance imaging [15,16]. Recently, RGO sheets decorated by magnetic nanoparticles were used as a strong absorbent in waste water purification and magneto-photothermal therapy of cancer [17,18] Dispersion of magnetic nanoparticles on reduced graphene oxide sheets carves an efficient route for catalytic, magnetic, adsorbing and electrode material [22,23]. Composites of magnetic nanoparticles such as  $Fe_3O_4$ ,  $ZnFe_2O_4$ ,  $CoFe_2O_4$ , Co with graphene have been recently reported [24–30]. Ni nanoparticles are also one of the important magnetic materials having applications as catalyst, filler of nano inks and magnetic fluids [31–34]. The effect of UV irradiation on the various properties of graphene-based materials is very important subject which has recently attracted the attention of researchers [19–21].

To the best of our knowledge effect of photo-irradiation on the reduced graphene oxide and nickel nanoparticle (NP) composite is rarely studied. In the present study, the RGO-Ni NP composites were synthesized and photo-irradiated with the UV-light ( $\lambda$ =254 nm) for different time durations. Their structural and magnetic properties were

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studied by X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), Transmission electron microscopy (TEM), Raman spectroscopy and vibrating sample magnetometer (VSM). The aim of the present study is to investigate photo-induced modification in the structure of reduced graphene oxide-Ni nanocomposite. Systematic study on effect of increasing UV dose on the magnetic properties of the composites is reported.

#### 2. Experimental

#### 2.1. Preparation of reduced graphene oxide

All the chemicals used for the synthesis were of analytical reagent grade and were purchased from Sigma Aldrich. At first the graphene oxide was synthesized after oxidizing graphite with KMnO4 in the presence of sulphuric acid using well-reported Hummer's method [35]. Oxidation helps in exfoliating the sheets by increasing the interlayer spacing. The solid product was separated by centrifugation, after washing repeatedly with acetone, and dried in ambient at 65 °C for 12 h. To synthesize quantum sheets of RGO, we started with reducing the graphene oxide (GO) by hydrazine [36]. The hydrazine has been found to be the best reducing agent in producing very thin graphene like sheets [37]. The larger particles of graphite, which did not exfoliate, were removed by centrifuging (in de-ionized water) at 4500 rpm for 10 min, and the product so formed was suitable for further studies. The obtained product was again diluted with de-ionized water and sonicated in an ultrasonic bath for 30 min. Hydrazine was added and sonication continued for a further 210 min. The total synthesis time was 4 h and the sample is designated as reduced graphene oxide (RGO). The lightweight black products floated on the solution and were washed thoroughly with distilled water and alcohol and dried under vacuum at 370 °C for 2 h.

#### 2.2. Synthesis of Ni nanoparticles on RGO nanosheets

The typical procedure for the synthesis of RGO-Ni nanocomposite is as follows: 35.0 mg of GO sheets, 70 mg (5 mmol) of Ni (NO<sub>3</sub>)<sub>2</sub>. 6H<sub>2</sub>O, were dispersed in 50 mL of ethylene glycol (EG) with ultra sonication for 1 h (weight % ratio of Ni (NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O: GO=2:1). Next, we added 25 mL of hydrazine hydrate and 1.0 M NaOH solution in sequence. The solution was heated with rapid stirring at a temperature of 60 °C; nickel-RGO nanocomposite was formed after about 1 h. This sample was designated as RGNi1. Using the same procedure, another sample having weight % ratio of Ni (NO3)2·6H2O: GO=1:1 was prepared and this sample was designated as RGNi<sub>2</sub>. These samples were preserved and used for further experimentation. Wu et al. have explained the formation of Ni nanoparticles with ethylene glycol and NaOH. These authors have reported that no particles were formed in ethylene glycol without adding sufficient amount of hydrazine at 25-60 °C, which reveals the particles, were reduced by hydrazine instead of ethylene glycol. Also addition of NaOH solution favors formation of pure Ni nanoparticles in ethylene glycol. Instead of ethylene glycol water was also tried as the possible solvent, but the resulting product was impure [38].

#### 2.3. Characterization

The crystalline phase of as-synthesized samples was characterized using powder X-Ray diffraction (XRD, Bruker D8 Advance diffractometer) with CuK<sub> $\alpha$ </sub> radiation ( $\lambda$ =1.5406 Å). Surface morphology and particle size of the composite was studied using FESEM (Hitachi, S-4800 operating at 30 kV and 10 microA). Raman spectra were recorded at room temperature using a DXR Raman microscope (ThermoFisher Scientific) with 514.5 nm excitation source from an Ar<sup>+</sup> laser. The magnetic measurements were carried out with VSM, Lake Shore7037. A switchable UVGL 58 lamp emitting UV radiations of either wave-

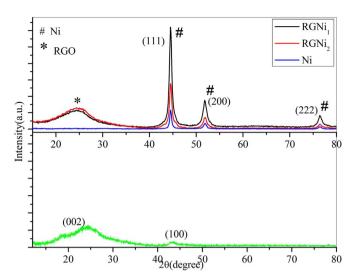


Fig. 1. The XRD patterns of (top panel) Ni, RGNi1, RGNi2; (bottom panel) RGO.

length 254 nm or 365 nm was used to expose the samples at room temperature. In the present study, samples were exposed only to 254 nm UV radiations. One minute of exposure by 254 nm radiations from this lamp at a distance of 3 in. from the sample corresponds to an irradiation of 55.8  $mJ/cm^2$ .

#### 3. Results and discussion

## 3.1. X-ray diffraction and field emission scanning electron microscopy (FESEM)

Fig. 1 shows X-ray diffraction (XRD) patterns of Ni NPs, Ni NPs-RGO composites (RGNi<sub>1</sub> & RGNi<sub>2</sub>) and RGO. The reduction of graphene oxide leads to the formation of highly reduced graphene which is indicated by appearance of peak at 24.4° and a weak peak at 43.2° [36]. Both RGNi<sub>1</sub>and RGNi<sub>2</sub> composites show peaks at 20=44.5°, 51.9°, 76.5° corresponding to (111), (200) and (222) planes of fcc Ni (JCPDS 01–1260) respectively and a weak signature of reduced graphene oxide is also visible at 24.8° which differs slightly (0.4°) from RGO peak may be due to difference in the degree of reduction in both the compounds and the fact that layers of reduced graphene oxide in the composites are more closely spaced. No peak corresponding to the NiO was detected. The average crystallite size (D) calculated using Debye Scherer formula, for the most intense peak (at around 20=44.5°) is listed in Table 1. The average particle size of Ni in the composite is found to be less than 20 nm.

Further insight into the structure and surface morphology of the samples was obtained by using field emission scanning electron microscope. FESEM images of as prepared a) Ni NPs, b) RGNi<sub>1</sub>, c) RGNi<sub>2</sub> and d) RGO are shown in Fig. 2. The Fig. 2a shows irregular morphology of Ni nanoparticles with needle like projections on them. As expected high agglomeration among the nanoparticles is visible due to reduced surface energy and the dipolar attraction between magnetic NPs [39,40]. In RGNi<sub>1</sub> and RGNi<sub>2</sub> sample the Ni NPs are seen decorated on reduced graphene oxide sheet. Arrows in the images

Table 1 FWHM, lattice parameter,  $(2\Theta)$  position of (111) plane and particle size (nm) of Ni, RGNi<sub>1</sub>, RGNi<sub>2</sub>.

Sample	FWHM	Lattice parameter (d)Å	Position (20)	Particle size (nm)
Ni	0.50	2.033	44.52	17
RGNi <sub>1</sub>	0.68	2.033	44.51	12
RGNi <sub>2</sub>	0.77	2.033	44.52	11

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