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Technical note

Study of surface morphology of ferrofluid deposited etched ion tracks in dielectric layers

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ABSTRACT

An AFM study is reported on swift heavy irradiated Si/SiO_2 substrates which have been etched by aqueous hydrofluoric acid solution leading to ion tracks in which ferrofluids have been deposited leading to **t**unable **e**lectronic **m**aterials with **p**ores in **o**xide on **s**ilicon (TEMPOS) structure. Two ferrofluids with different carrier fluids (aqueous and non-aqueous) have been deposited in the tracks. Atomic force microscopy has been used to study the empty as well as filled tracks. Since the ferrofluids contain iron oxide particles, there is a possibility of agglomeration of these particles inside and outside the tracks. Surface area and pore volume of the tracks have been measured by Brunauer-Emmett-Teller (BET) method. The track properties (empty and filled) as observed by AFM have been correlated with BET measurements.

1. Introduction

Swift heavy ions (SHI) loose energy via electronic excitation and ionization which can induce damage and structural modification in solids. Swift heavy ions travelling through a dielectric material will leave tracks of distorted material in their path. If the linear energy transfer of the ions is high enough, their tracks will be a few nanometers wide continuous channels of transformed material. In the low fluence regime, the damaged zone produced by SHI is localized within a diameter of few nm, while for high fluence, a few μm of damaged zone can arise due to SHI induced mass transport on the surface of irradiated zone. Therefore, to study the ion induced damage on the sample surface, the size of the observation window should be limited to an area of a few μm^2. In this respect, AFM offers an advantage over conventional techniques to study the SHI induced surface modifications. Many features of this type of materials have been reported previously (Lu et al., 2000; Singh et al., 2000; Srivastava et al., 2002; Carlotti et al., 2006; Martin et al., 2006, 2007; Carvalho et al., 2007, 2008). The etching followed by subsequent filling with appropriate filler creates **t**unable **e**lectronic **m**aterial with **p**ores in **o**xide on **s**ilicon (TEMPOS) structures. Studies on the TEMPOS structures are given in detail in references (Fink et al., 2003a,b, 2004, 2007; Fink and Chadderton, 2005; Sinha et al., 2004; Weidinger, 2004; Saroch et al., 2008).

The TEMPOS structure has been used to fabricate magnetic sensors by filling the ferrofluids in the ion tracks. In this paper, the AFM and BET studies of these structures is being reported. Ferrofluids, which are stable colloidal suspensions, have been used for various applications (Papell, 1965; Rosensweig and Kaiser, 1967; Reimers and Khalafalla, 1974; Rosensweig, 1985; Raj et al., 1995; Chen et al., 1997). It is known that the rheological, hydrodynamic, diffusional, magnetic and optical properties of a ferrofluid changes by a hundred times under an applied magnetic field of moderate strength (Papell, 1965).

Brunauer-Emmett-Teller (BET) method is recognized as a standard method for determining the surface area and pore volume of porous structures (Shelekhin et al., 1995; Clausen and Fabricius, 2000; Li et al., 2008). Gas adsorption is the most widely used and accurate technique for total surface area measurements. Gas molecules of known sizes are condensed onto the unknown sample surface. By completely covering the surface and opening of the pores with a condensed gas, the surface area analyser can characterise the surface, including irregularities and pore interiors down to an atomic level. Nitrogen is often the gas used as its molecular size is well established; it is inert and available of high purity. The technique requires a clean surface as the sample has to be taken to an elevated temperature under vacuum for which “out-gassing” is a necessary step. The “outgassed” sample under high vacuum in its sample tube is immersed in a coolant bath of liquid nitrogen at −195 °C. At this stage the sample is ready to attract gas molecules onto it when they are admitted to the sample tube. The amount of

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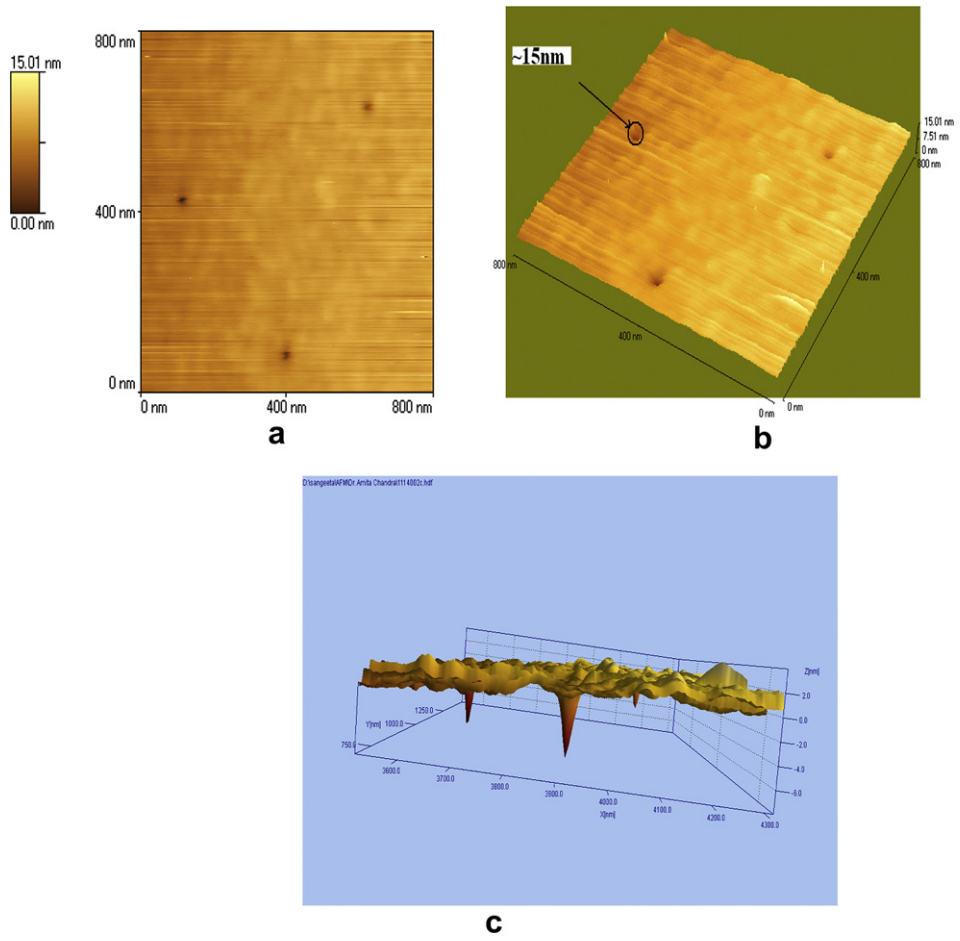

Fig. 1. AFM image of irradiated (350 MeV Au^{26+} ions beam of fluence $10^8/cm^2$) Si/SiO_2: (a) scan area 800 × 800 nm^2, (b) 3D image of the substrate and (c) view of conical tracks.

gas adsorbed and the resultant sample pressure are recorded. This data is subjected to a number of calculations to obtain surface area. In the present work, as the samples are porous, the deposition of ferrofluid changes the pore volume and surface area of TEMPOS structures. The above described BET method has been employed to correlate the pore volume and area with the AFM measurements.

2. Experimental

A dielectric layer (silicon dioxide of thickness 100 nm) on silicon substrate (thickness 375 μm) has been irradiated by 350 MeV Au^{26+} swift heavy ions. The electronic energy loss constitutes the ionization and excitation energy loss by the primary ion as well as by recoil atoms. As a result, the latent tracks are created. These latent tracks have been etched by 4% HF for 14 min to create parallel open tracks into which ferrofluids have been inserted. A layer (~4–6 μm thick) of ferrofluid has been deposited on the surface of the dielectric layer and into the etched ion tracks.

The ferrofluids have been synthesized by $FeCl_2$ and $FeCl_3$ using the methods described by Racuciu et al. (2005) for aqueous ferrofluid (particles size ~14 nm, water as carrier fluid) and Maity and Agrawal (2007) for non-aqueous ferrofluid (particles size ~10 nm, oil as carrier fluid), respectively.

The pore volume and surface area have been measured with Gemini-V2.00 instrument (Micromeritics Instrument Corp.) Both the parameters have been calculated by adsorption isotherm according to BET theory. Nitrogen has been used as adsorbate in it. Three samples, viz., (i) irradiated and unetched Si/SiO_2, (ii) irradiated and etched Si/SiO_2 and (iii) aqueous ferrofluid filled etched Si/SiO_2 have been selected for the above study. The non-aqueous ferrofluid filled Si/SiO_2 was not used so as to avoid oil contamination of the instrument.

3. Results

The AFM micrograph of irradiated dielectric layer on a substrate (silicon) is shown in Fig. 1(a), (area 800 × 800 nm^2). The imaging has been done in contact mode using Veeco instrument (2002). From Fig. 1(b), it is clear that the swift ions create tracks in the substrate. From Fig. 1(c) which is a 3D image of the substrate, the shape of tracks can be identified as conical. The track diameter is found to be ~15 nm and it increases in the case of overlapping of tracks up to ~25 nm. Table 1 gives the line analysis of this particular sample substrate which gives the depth of the conical tracks to be ~5–7 nm. The root mean square (rms) values of surface roughness evaluated from AFM data are 1.1972 nm and 1.1934 nm for whole and partial image, respectively.

Fig. 2(a) gives the schematic diagram of TEMPOS structure. The aqueous and non-aqueous magnetic nanoparticles (iron oxide) present in the ferrofluids, are coated with citric acid and oleic acid, respectively. When inserted in the tracks, the possibility of their agglomeration exists. Fig. 2(b) is the schematic of the distribution of magnetic nanoparticles on the TEMPOS surface and the possibilities of their agglomeration in the ion tracks. The tracks are of conical shape and there is a possibility of overlapping of tracks as seen in AFM images. In these overlapped tracks, the track diameter