

# The electron field emission properties of ion beam synthesised metal-dielectric nanocomposite layers on silicon substrates

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## Abstract

The search for cold electron field emission materials, with low threshold fields, compatible with existing integrated circuit fabrication has continued to attract a significant research interest over the past several years. This is primarily driven by their potential applications in vacuum microelectronic devices and flat panel displays. In this paper, the preparation of Ag-SiO<sub>2</sub> nanocomposite layers on Si substrates by Ag implantation into thermally oxidized SiO<sub>2</sub> layers is reported. The electron field emission (FE) properties of these nanocomposite layers were studied and correlated with results using other characterisation techniques, including atomic force microscopy, Rutherford backscattering spectroscopy, X-ray diffraction and transmission electron microscopy. The experimental results indicate that these nanocomposite layers have good FE properties with threshold fields as low as 13 V/μm. The FE mechanisms of these layers are discussed in term of an electrical inhomogeneity effect.

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## 1. Introduction

The saturation velocity of electrons in solids is typically limited to 10<sup>5</sup> ms<sup>-1</sup> by the lattice scattering, while the electron velocity in vacuum can approach the speed of light, 3 × 10<sup>8</sup> ms<sup>-1</sup>. Hence, vacuum electronic devices are attractive for high speed and high frequency applications [1]. In conventional vacuum electronic devices, electrons are liberated by thermionic emission from hot filaments. However, hot filaments are large and much energy is wasted in heating up the filament. Replacing the thermionic cathode by a cold cathode can reduce the size of the device and also improve the power efficiency. For cold cathodes, electrons are liberated by tunnelling from the cathode material at room temperature, under an intense electric field, which is termed electron field emission (FE). Furthermore, the global market for flat panel displays (FPD) will be worth 70 billion US dollar by 2010 [2] and cold electron FE materials, with low threshold fields, are seen to as potential candidates for FE-based displays.

Metallic-dielectric nanocomposites such as resin-carbon coatings [3], graphitic clusters embedded in amorphous carbon (a-C) films [4–6], metal doped a-C films [7,8] and metal

implanted SiC layers [9], have attracted much attention from researchers as novel cold field emission materials. This is due to their excellent FE properties and very low threshold fields for electron emission, typically smaller than 20 V/μm, when compared with several thousand volts per micrometre for the flat metallic cathodes. This is attributed to a local electric field enhancement by virtue of the electrical inhomogeneity between nanosized conductive clusters and the insulating dielectric matrix.

In this work, we synthesise Ag-SiO<sub>2</sub> nanocomposite layers by Ag implantation on SiO<sub>2</sub> layers, grown by thermal oxidation of Si substrates. We show that the FE properties of these layers are very good, and that the threshold fields achieved can be as low as 13 V/μm with appropriate implantation dose of Ag ions. The FE properties of these samples are discussed in terms of the electrical inhomogeneity effects due to the isolated nanosized conductive Ag clusters embedded in the electrically insulating SiO<sub>2</sub> matrix.

SiO<sub>2</sub> is used as the host matrix because it is chemically stable and its fabrication process by thermal oxidation is efficient. Moreover, the etching process of SiO<sub>2</sub> is well characterised and fast, hence, it is convenient to create differently patterned FE devices. Ag is chosen because it does not react chemically with the SiO<sub>2</sub> matrix [10] and because the formation of nano-sized pure Ag clusters in silica is possible with small Ag doses (1 × 10<sup>15</sup> cm<sup>-2</sup>) [11]. Moreover, Ag has excellent elec-

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trical and thermal conductivities, which are beneficial in FE device applications. Using the  $\text{SiO}_2$  as the insulating matrix and subsequently creating the conducting clusters by metal ion implantation has the added benefit that the whole fabrication process is compatible with existing IC technology. Therefore, using these Ag- $\text{SiO}_2$  nanocomposite layers as the cathode material for vacuum microelectronic devices take advantage of the possibility for integrating the devices with other circuit elements on a single chip.

## 2. Experimental

$\text{SiO}_2$  layers were grown on n-type (1 0 0) Si wafers with a resistivity  $\leq 0.05 \Omega \text{ cm}$ , using dry thermal oxidation at  $1000^\circ\text{C}$  for 2 h. The thickness of these layers were checked by ellipsometry and determined to be  $120 \pm 10 \text{ nm}$  over a 4 in. wafer. The resulting layers were subsequently implanted with 110 keV  $\text{Ag}^+$  ions at room temperature, using a 200 kV ion implanter. According to static SRIM simulation [12], the ion range of 110 keV  $\text{Ag}^+$  ions in a 110 nm thick  $\text{SiO}_2$  layer on a Si substrate is found to be 58 nm. The doses ranged from  $1 \times 10^{16}$  to  $5 \times 10^{16} \text{ cm}^{-2}$  and were confirmed by Rutherford backscattering spectrometry (RBS) measurements with a 1.56 MeV  $^4\text{He}^+$  beam.

The FE properties of the samples were studied at room temperature, in a high vacuum chamber with the base pressure better than  $5 \times 10^{-4} \text{ Pa}$ . The current–electric field ( $I$ – $F$ ) characteristics were measured using a “sphere-to-plane” electrode configuration, with a 5 mm diameter stainless-steel ball anode. The anode was mounted on a Vacuum Generator HPT translator, which allowed FE maps to be carried out without the need for the sample to be returned to atmosphere. The applied electric field was obtained by dividing the applied voltage by the gap distance (typically,  $40 \mu\text{m}$ ). The threshold field  $F_{\text{th}}$ , is defined as the field strength where the emission current reaches 1 nA. The measurement was taken in five different regions for each sample, with the high voltage stepped up and down five times to check the repeatability and homogeneity. The surface morphology was studied using atomic force microscopy (AFM). The microstructure characterization and phase determination were done using a Philips CM200 transmission electron microscope (TEM), at an acceleration voltage of 200 kV, and with glancing incidence X-ray diffraction (XRD) with a Cu  $K\alpha$  source.

## 3. Results and discussion

The XRD spectra of these Ag implanted  $\text{SiO}_2$  layers are shown in Fig. 1. For the two higher dose samples ( $3$  and  $5 \times 10^{16} \text{ cm}^{-2}$ ), four peaks corresponding to (1 1 1), (2 0 0), (2 2 0) and (3 1 1) planes of FCC silver are clearly observed in the XRD spectrum, indicating that the implanted silver atoms are in pure metal form. The average size of the Ag nano-clusters,  $d$ , is determined to be 10.2 and 14.1 nm in diameter, for the samples with doses of 3 and  $5 \times 10^{16} \text{ cm}^{-2}$ , respectively, from the full width half maximum of the Ag (1 1 1) peak, using the Scherrer formula [13]. However, a background signal is

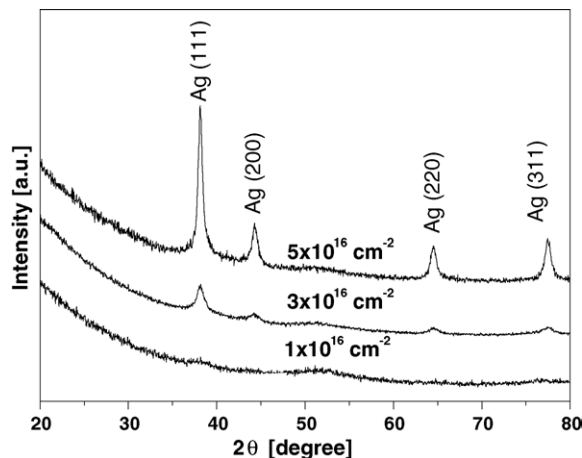


Fig. 1. Glancing incidence XRD spectra of samples implanted with various Ag doses.

obtained for the lowest dose sample ( $1 \times 10^{16} \text{ cm}^{-2}$ ), and hence, XRD fails to indicate the state of the implanted Ag ions in the sample.

In order to identify the state of the implanted Ag ions and to determine the microstructure of the lowest dose sample, cross-sectional transmission electron microscopy (X-TEM) measurements were performed and the results are shown in Figs. 2 and 3. As seen from the X-TEM micrograph, in Fig. 2(a), an Ag- $\text{SiO}_2$  nanocomposite layer, with a thickness of 130 nm, is formed after Ag ion implantation with a dose of  $1 \times 10^{16} \text{ cm}^{-2}$  at 110 keV. The clusters are identified as crystalline Ag nanoparticles since the selected area electron diffraction pattern can be indexed as the FCC silver. The Ag clusters with a maximum size of 11 nm in diameter, are located at a depth of 50 nm beneath the surface, which is close to the ion range predicted by SRIM. In addition, clusters with sizes of 1–2 nm are distributed in the whole implanted layer. The average size of the Ag clusters,  $d$ , is determined to be 4.8 nm and their statistical distribution is shown in Fig. 2(b). The high-resolution X-TEM micrographs of the sample taken at the top, the middle and the bottom of the layer, are shown in Fig. 3. Near to the surface, the size of the Ag clusters is  $\sim 2 \text{ nm}$ , which is smaller than the average value of 4.8 nm for the whole layer as shown in Fig. 3a. As the depth from surface increases, the average size and concentration of the Ag clusters increases. Ag clusters with sizes of around 6–8 nm, are observed in the middle of the layer. In the bottom region, the Ag cluster size decreases to  $\sim 2.5 \text{ nm}$  and the concentration is much lower than that in the middle layer. Moreover, near to the interface between the implanted layer and the Si substrate, a 1.25 nm thick layer of Ag precipitate is also observed.

The electron FE characteristics of the Ag implanted  $\text{SiO}_2$  layers are shown in Fig. 4. The FE characteristic of a ‘bare’  $\text{SiO}_2$  layer is also included, to show the effect of the introduction of Ag nano-clusters in the  $\text{SiO}_2$  layers. From the  $I$ – $F$  characteristics shown in Fig. 4(a), the  $F_{\text{th}}$  of the ‘bare’  $\text{SiO}_2$  layer decreases from 204 to 44 V/ $\mu\text{m}$ , after the Ag implantation to a dose of  $1 \times 10^{16} \text{ cm}^{-2}$ . The  $F_{\text{th}}$  has further decreased to 20 V/ $\mu\text{m}$  when the dose of Ag was increased to  $3 \times 10^{16} \text{ cm}^{-2}$ , and attained

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