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Distribution and coverage of 40 nm gold nano-particles on aluminum and hafnium oxide using electrophoretic method and fabricated MOS structures

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ABSTRACT

In this paper, electrophoretic method (EPD) is used to deposit 40 nm gold nano-particles (GNPs) on atomic layer deposited (ALD) grown oxides. Ultra-High Resolution Scanning Electron Microscopy (HRSEM) has been used to study the distribution of gold nano-particles on both Al_2O_3 and HfO_2 thin films as a function of dipping time. The results show better distribution and mechanical adhesion of GNPs on top of HfO_2 compared to Al_2O_3. Additionally, metal-oxide-semiconductor (MOS) structures with GNPs embedded within the HfO_2 layers are fabricated. The distribution and coverage of the GNPs are analyzed using hysteresis loop of capacitance-voltage measurements at (1.0 MHz) and by quantifying the threshold voltage shift (ΔVt). Moreover, the distribution of the GNPs as function of dipping time is discussed in terms of the measured ΔVt. The results show an increase in ΔVt with longer dipping time followed by a drop in the ΔVt. Moreover, the Vt shift vs. dipping time indicates changes in the electrophoretic mobility behavior and the final distribution of GNPs on the surface of the HfO_2.

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1. Introduction

Methods to deposit nanoparticles in an ordered and controlled manner are vital for future use of nanoparticles in electrical and photonic applications. For instance, it has been shown that nanoparticles can improve the efficiency of solar cells by either plasmonic effect or photon down shifting [1,2]. In addition, it has been shown that nanoparticles in non-volatile memory devices can lower the operating voltage and enhance their performance characteristics such as: retention and endurance characteristics [3,4]. One promising method to introduce nanoparticles in a device structure is the Electrophoretic Deposition (EPD). It has been shown [5,6] that such method is well suited for fabricating a wide range of structures ranging from thin-films, wires and nano patterns of colloidal nanoparticles. EPD method offers superior mechanical adhesion strength of the fabricated thin film and is inherently a low cost deposition technique [7,8]. Typically, in the EPD method, a uniform electric field is used to achieve the deposition of the particles on the substrate. This method allows for controllability by the applied external field, and it requires no special substrate passivation on the colloidal nanoparticles [9,10]. Several studies have been done to correlate the amount of particles deposited during EPD with different influencing parameters [11,12]. Bouyer and Foissy [13] describe the EPD method as a two-step process. Step one is the application of an electric field, while step two is the movement of the particles in the suspension to one of the electrodes. This depends mainly on the bulk properties of the colloidal dispersion (bath conductivity, viscosity, particle concentration, size distribution, and surface charge density) and the actual field strength in the bath. After this the deposition step proceeds and particles accumulate selectively and lose their charge at the electrode. However, the process of deposition using EPD is complex and many parameters can influence the distribution of the NPs, hence the technique is still not well understood. Earlier we studied GNPs distribution deposited on crystalline silicon wafer using EPD method [14]. In this article, we study an electric field-assisted dip-coating deposition method of gold colloidal nanoparticles on oxides surface. The GNPs are initially suspended in an aqueous solution and deposited by EPD onto an Atomic layer Deposited (ALD) oxide

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surface. The adsorption of nanoparticles onto the Al_2O_3 or HfO_2 thin films is described and the generated yield of gold nanoparticles is investigated as a function of experimental parameters: applied voltage, electric field, and initial nanoparticle density existing in the solution. The dipping time is varied and High Resolution Field Emission Scanning Electron Microscopy (HR-FESEM) is used to confirm the thin film phases. The electrical properties of the fabricated MOS structures are measured and the results are explained based on the oxide thickness and dipping time.

2. Experimental details

2.1. EPD setup and characterization

Fig. 1(a) illustrates the process flow of depositing GNPs on ALD grown oxides surfaces using EPD. Similar process flow was discussed in [14]. The starting substrate is a *p*-type Si < 100 > with a resistivity of 2mΩ cm. The silicon electrodes are cleaned using 1:10 HF solution. A thin film of Al_2O_3 or HfO_2 is deposited using plasma enhanced Atomic Layer Deposition (ALD) at temperature of 200 °C and a pressure of 200 mTorr. The oxide thicknesses are measured using spectroscopic ellipsometry over a wavelength range (250–1800 nm) and the data fitting was carried out using (CompleteEASA [15]). After the ALD grown high-κ oxide deposition, electrophoretic of GNPs is carried out using moving electrodes that are dipped vertically into the colloidal particle solution as indicated in Fig. 1(b). One electrode consists of *p*-Si and a thin film of Al_2O_3 or HfO_2 and the other electrode is heavily doped *p*-type Si. The electrodes are connected to an outer voltage source charging them differently with an effective electric field of 20 V/cm. The cathode and the anode are spaced by 2 cm. The process consists of two steps: immersing the electrodes inside the colloidal particle solution and applying the electric field for a fixed period of time. The 40 nm citrate-stabilized gold nano-particles are commercially available from SIGMA ALDRICH and have an initial concentration of 7.15×10^{10} NP/ml [16]. A 2 ml of gold colloidal has been diluted with 50 ml of deionized water, giving a final solution that has a particle concentration of 2.86×10^9 NP/ml. The deposition occurs selectively at the anode, which indicates that the Au particles have a negative charge.

Ultra-High Resolution Field Emission Scanning Electron Microscopy (FE-SEM) with 10 keV accelerating voltage has been used to study the particle distribution, packing and morphology. Furthermore, the particles distribution and the statistics of their sizes are analyzed using Image J software [17]. The electrical properties, in particular the capacitance-voltage measurements, are carried out using Agilent B 1505 curve tracer and Signatone manual 1160 prober. The CV curves are measured under a high frequency of 1 MHz.

2.1.1. Structural properties of GNP

Fig. 2 shows a micrograph of the GNPs distribution and the particles coverage on the top of 5.5 nm thin film of Al_2O_3. The image clearly shows that a more densely packed nanoparticle cluster can be achieved for longer dipping times, and a thin film of GNPs was gradually transformed into multilayered structure. Although these particles are closely packed, they retain the properties of individual particles. The GNPs on the top of a 4.2 nm thin film of HfO_2 is also imaged for two different dipping times and is shown in Fig. 3. These micrographs show that the adhesion of GNPs on a top of HfO_2 is highly dense compared to the ones deposited on Al_2O_3 film for the same dipping time period. This might be explained in terms of surface termination and the chemical bonds of each oxide deposited using ALD, where TMA-Al $(CH_3)_3$ precursor is used for Al_2O_3 deposition, while TEMAH-Hf(N $(C_2H_5)(CH_3))_4$ precursor is assigned for HfO_2 deposition). Accordingly this makes it easier for GNPs to attach on the HfO_2 surface [18,19]. It should be noted that after

the organic cleaning of the samples with Isopropanol $(CH_3)_2CHOH$ and Acetone (CH_3COCH_3) in ultrasonic path could not remove the gold nano particles due to strong adsorption of the particles on the surface after applying an electric field. Fig. 4 shows the GNPs diameter distribution after applying a DC voltage of 40 V. Most particles are between 40nm–50 nm in diameter.

2.1.2. Electrical properties

The Capacitance-Voltage (*CV*) voltage measurements conducted on the samples after the EPD process with GNPs on the top of Al_2O_3/HfO_2 oxides at different dipping time is carried out to investigate the electrical properties of the uncapped structures prepared as shown in Fig. 1(a). The voltage was swept from −5 V (accumulation) to +5 V (inversion) under high-frequency of 1 MHz, as indicated in Fig. 5. Using these measurements, a higher capacitance value is observed for longer deposition times where the density of GNPs is higher (the number of nanoparticles in 1 μm^2) as confirmed by SEM. Since HfO_2 has higher κ value (~22) than Al_2O_3 (~9), the Capacitance-Voltage curve at 30 min for HfO_2 has a higher value than the Al_2O_3 at the same deposition time.

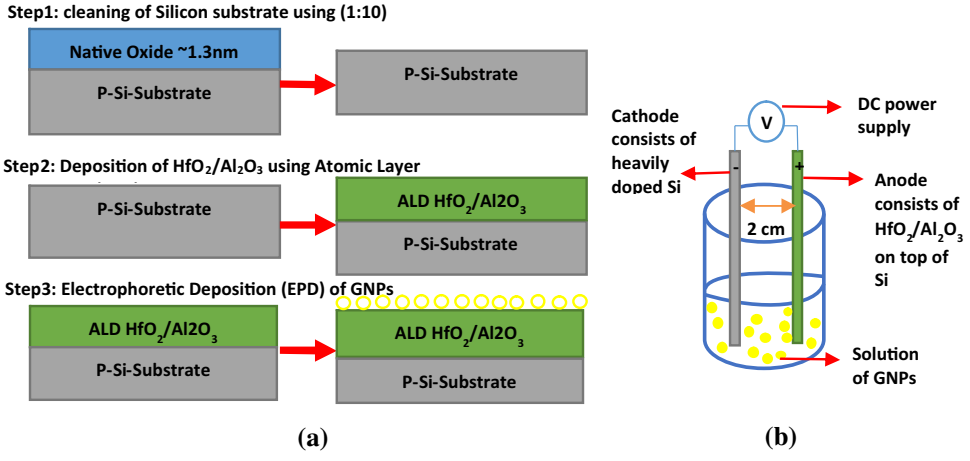

Fig. 1. Electrophoretic of gold nano particles (uncapped structure) (a) Schematic of the sample preparation processes (b) Cartoon of EPD set up.

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