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ANALYSIS STUDY OF SINGLE GOLD NANOPARTICLE SYSTEM OF INTERDIGITATED DEVICE ELECTRODES (IDES) BY USING ENERGY-DISPERSIVE X-RAY (EDX)

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ABSTRACT

Rapid identification of viruses has been important implications for medical healthcare. This paper presents an analysis study of a single gold nanoparticle system of Interdigitated Device Electrodes (IDEs) by using Energy-Dispersive X-ray (EDX). The presently available methods of detection are mainly based on rapid immunoassay techniques that require trained manpower and are time consuming and expensive. However a biosensor facilitates a doctor to identify the specific virus quickly and prescribe an appropriate course of treatment. In this study, a nanobiosensor based on gold nanoparticles (GNPs) and oligonucleotide probe was designed for the visual detection of Human *Papillomavirus* (HPV). A simple and facile method for the depositing of gold nanoparticles (GNPs) colloid was described via a simple deposition method on Interdigitated device electrodes (IDEs). The GNP pattern deposition on IDE was investigated by using an SEM combined with an EDX. The study demonstrates the use 30 nm of gold nanoparticles functionalized with single stranded oligonucleotide (GNP- oligo probe) as visual detection probes for rapid and specific detection of HPV. By addition of gold nanoparticles, it enhances the signal and sensitivity of IDE for detection of HPV.

Keywords: gold nanoparticles, IDEs, nanodevice, EDX, human *papillomavirus,* DNA.

INTRODUCTION

Energy-dispersive X-ray spectroscopy (EDS, EDX, or XEDS), also called as energy dispersive X-ray analysis (EDXA) or energy dispersive X-ray microanalysis (EDXMA), is an analytical technique used for the elemental analysis or chemical characterization of a sample (Allen *et al*., 2012). It relies on an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray emission spectrum (Petkov, 2008). To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons, or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state electrons in discrete energy levels or electron shells bound to the nucleus.

The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray (Piryatinski *et al*., 2007). The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the X-rays are characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured (Yonehara *et al*., 2010).

Gold nanoparticles (GNPs) have received greatest interests because they have several kinds of intriguing properties (Li *et al*., 2010). GNPs, with the diameter of 1-100 nm, have high surface-to-volume ratio and high surface energy to provide a stable immobilization of a large amount of biomolecules retaining their bioactivity (Xing *et al*., 2010). GNPs have an ability to permit fast and direct electron transfer between a wide range of electroactive species and electrode materials (Milliron *et al*., 2003). In addition, the light-scattering properties and extremely large enhancement ability of the local electromagnetic field enables GNPs to be used as signal amplification tags in diverse biosensors (Liu *et al*., 2009).

GNPs are the nanostructures most widely used for DNA detection(Jelveh & Chithrani, 2011). Simple synthetic procedures are required in order to obtain GNPs with well-controlled diameters, shapes, and optical properties. AuNPs possess extremely high extinction coefficients, so slight aggregation may result in intense color changes (Jelveh and Chithrani, 2011). The large surface area of nanoparticles allows hundreds of capture probe DNAs to be loaded, while the three dimensional assembly of the probe lowers steric hindrance and favors target–probe hybridization (Stevens *et al*., 1999).

Gold Nanoparticles (GNPs) with unique size-dependant properties have drawn tremendous interest in various scientific and technical applications including electronics, chemical conversion, data storage, and sensors (Li *et al*., 2010). To fabricate functional devices using GNPs, it is essential to form well-defined ordered arrays of GNPs at the micro- or nano-scale (Nagender Reddy Panyala *et al*., 2009).

Thus, diverse methodologies including lithographic patterning using UV and electron beams, soft lithography (Suh *et al*., 2004), colloidal lithography, a self-assembled template method, inkjet printing, and other methods have been reported to pattern GNP on various types of substrates (Yang *et al*., 2006). In spite of these advances, a more accurate, lower-cost, and high-trough method has been required for practical applications.

The aim of this study was to study the morphology IDE coated with 30nm GNP by using SEM combined EDX analysis. IDE thin films with surface modification using GNPs were applied for biomolecule detection of target DNA hybridization.

MATERIALS AND METHODS

Material

The organosilane reagent (3-aminopropyl) triethoxysilane (APTES) was obtained from Sigma–Aldrich (St. Louis, MO, USA). APTES is silane coupling reagent with 3-aminopropyl group at one end, which terminates in a primary amine and silane reactive portion on another end with triethoxy group. The chemical formula of APTES is $C_9H_{23}NO_3Si$ with molecular weight of 221.37 g/mol. APTES is clear liquid with density of 0.946 g/ml at 25◦C.

SiO_2 cleaning

SiO_2 substrate devices were cleaned for 30 min in a piranha solution consisting of one-third hydrogen peroxide (30%) and two-thirds sulfuric acid (18 M), rinsed in distilled water, left for 10 min in boiling distilled water, dried under an argon flow, and used immediately. The substrates were then carefully rinsed with de-ionized (DI) water, and dried under a stream of nitrogen gas. The piranha solution (strong oxidizing agent) removes the organic contaminants and makes the surface more hydrophilic by hydroxylating the silicon surface.

IDE fabrication

The Interdigitated Device Electrodes (IDE) sensor was fabricated on a silicon-on-insulator (SOI) wafer with a 145nm buried oxide layer (Nadzirah *et al*. 2012). Silicon wafer was used as a main substrate in order to form silicon dioxide (SiO_2) as an insulation layer of an electrical device. The wafer was cleaned using buffered oxide etchant (BOE) to remove native oxide which had been naturally grow on it. Growth of SiO_2 using wet oxidation process provides thicker insulation layer and shorter time consuming compared to dry oxidation process (Adam *et al*., 2012). In order to transfer pattern from a mask on a wafer, photoresist was spin-coated on the growth SiO_2 wafer using spin-coater.

Photoresist is a light-sensitive material used to form a patterned coating on a surface. By using deep ultraviolet lithography, a 50 nm silicon layer was patterned and etched when UV-light was exposed on the phototresist, pattern from chrome mask was directly transfer onto the phototresist. After development process, aluminium metal was deposited using sputter-coater and acetone was used to strip the unwanted photoresist.

Gold nanoparticle colloid deposition

1µl of GNP (30nm) has been deposited on the active area of IDE for 1 h, at room temperature (Figure-1).

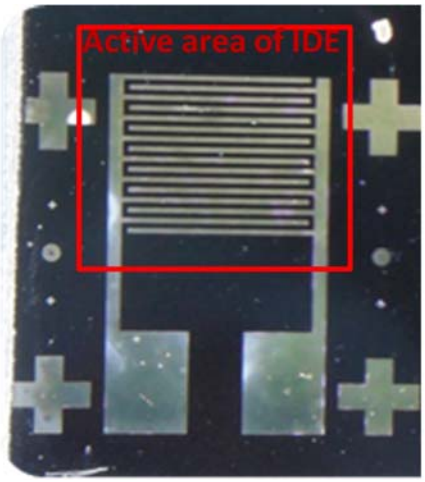

Figure-1. Active area of interdigitated device electrode (IDE).

Scanning electron microscopy (SEM) analysis

SEM was carried out at 10 kV acceleration volatege and a 9.8 mA emission current. The magnification was 2200 and working distance was 29.9mm. The SEM scan resolution was typically 640x480 pixels. Silicon dioxide thin films with 30nm GNP was analysed by using Scanning Electron Microscopy (SEM).

EDX analysis

Qualitative and semi-quantitative chemical analysis in the EDS mode were performed with a ThermoNORAN spectrometer model Quest and was applied to support the gold nanoparticles characterization. Schematic illustration of 30 nm GNPs linked to the top of an IDE thin film substrate was shown in Figure-2. Figure-3 showed schematic representation of an energy-dispersive spectrometer.

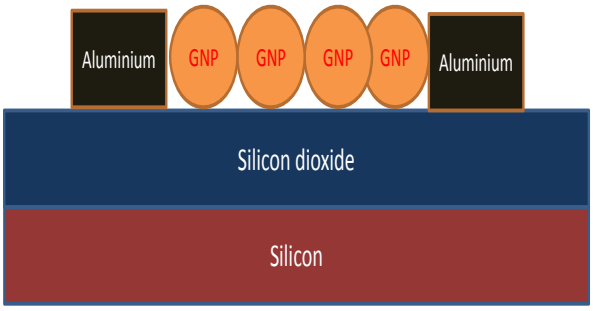

Figure-2. Schematic illustration of 30 nm GNPs linked to the top of an IDE thin film substrate.

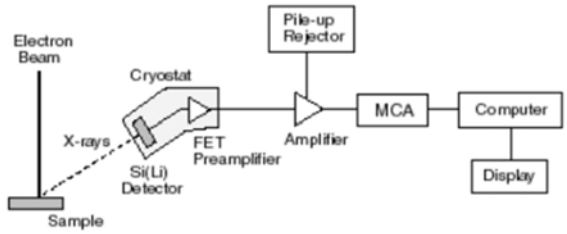

Figure-3. Schematic representation of an energy-dispersive spectrometer.

RESULTS AND DISCUSSIONS

Scanning electron microscopy (SEM) analysis

SEM image of gap between two fingers of IDE was shown in Figure-3. The size of gap is around 50 µm. The surface topology image was taken by using SEM as shown in Figure-4. Figure-4 showed the typical SEM image pattern of the gold nanoparticles with 30 nm size deposited on IDE thin film substrate in 5000x of magnificent. The SEM image shows surface compactness and some clustered nanoparticles. Scanning probe microscope experiments in tapping mode of operation was employed in order to determine the morphological structure of GNP deposit on IDE thin films. By using SEM, the images measured are only two dimensional.

The SEM micro-graph observation of the gold nanoparticle surface morphology was presented in Figure-4. In Figure-4, the particles are dominantly spherical in shape, with few triangular and hexagonal shapes, which are common features of gold nanoparticles (Song *et al*. 2009). The nanoparticles are all within the nanometer size and monodispersed. As expected, the GNP are agglomerated, the shapes of the particles are generally spherical and hexagonal.

Energy-dispersive x-ray spectroscopy (EDX) analysis

The GNP-embedded IDE patterns formed through a pattern mask were analyzed. The elemental composition of the synthesized gold nanoparticles was determined by energy dispersive x-ray (EDX) equipped with SEM. As shown in Figure-5, three elemental peaks assigned to the silicon (Si) and oxygen (O) of the SiO^2-deposited Si wafer were identified in the EDX spectra of the of the GNP pattern. Elemental peak of the gold (Au), in addition to the Si and O peaks, were observed in the EDX spectra correspond to the characteristic elemental constituent of the GNP patterns. 30 nm GNP was successfully formed during deposition on the IDE thin film.

Element composition of gold nanoparticles deposited on IDE thin films was shown in Table-1. Silicon shows 33.31% atomic percentages, gold nanoparticles show 0.03% while oxygen shows 66.66%. The EDX spectra show the weak peaks which may have been traced from water moisture and carbon adhesive used in the sample preparation prior taking the SEM micrograph for the EDX profile.

The elemental analysis of the EDX profile (Figure-5) confirmed the specific gold peaks from the sample. The result are in conformity with the earlier reports of gold nanoparticle found by other various synthesis methods (Ankamwar, 2010).

Table-1. Element composition of gold nanoparticles (GNP) deposited on IDE thin films.

Element	Weight (%)	Atomic (%)
Si K	2.48	33.31
Au M	0.02	0.03
O	2.83	66.66
Total	5.33	100

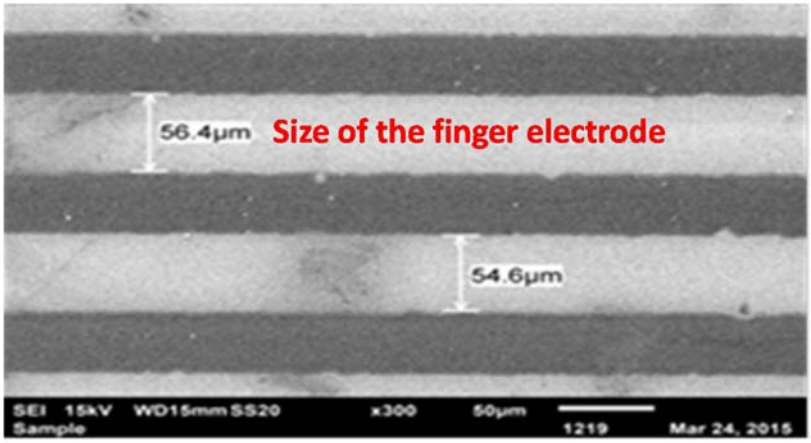

Figure-4. SEM image of gap between two fingers of IDE. The size of gap is around 50 µm.

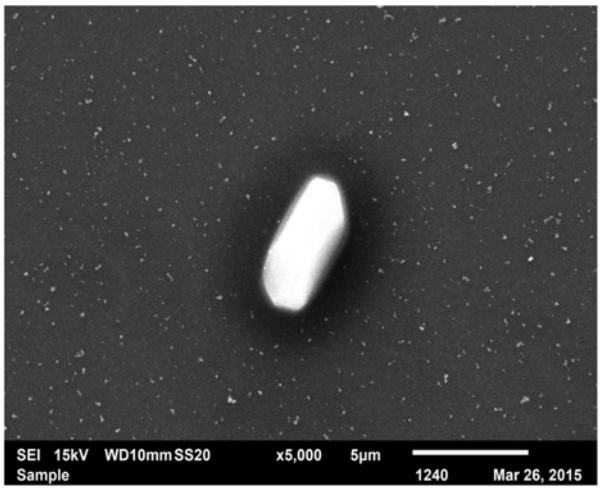

Figure-5. SEM image of gold nanoparticle (GNP) with 30nm size deposited on IDE thin film.

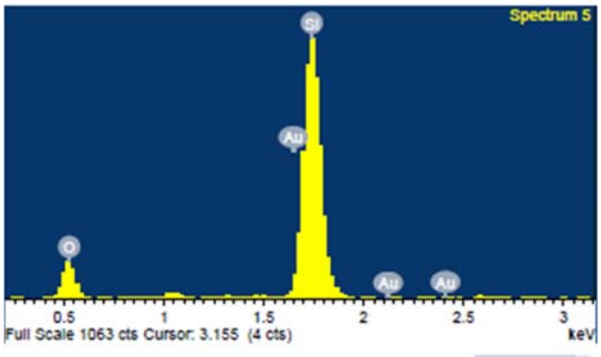

Figure-6. EDX spectra of gold nanoparticle (GNP) with 30nm size GNP 30nm deposited on IDE thin film.

CONCLUSIONS

The fabrication and characterization of gold nanoparticle (GNP) deposited on IDE based on SEM and EDX analysis was successfully demonstrated. The image taken from SEM shows the Interdigitated Electrodes (IDEs) on with GNPs, supported by EDX spectroscopy to verify the existence of GNPs binded to the electrode surface of IDEs. The unique and superior properties of GNPs have shown strong potential for the development of analytical systems with attractive and promising analytical behaviors. GNPs play in the process of biosensing, they provide much better analytical performances for biosensors in term of sensitivity, selectivity, reliability etc.

REFERENCES

Adam, T. *et al*. 2012. Material engineering for nano structure formation: Fabrication and characterization. In Procedia Engineering. pp. 361-368.

Allen, L.J. *et al*. 2012. Chemical mapping at atomic resolution using energy-dispersive x-ray spectroscopy. MRS Bulletin, 37, pp. 47-52.

Ankamwar, B., 2010. Biosynthesis of Gold Nanoparticles (Green-gold) Using Leaf Extract of Terminalia Catappa. E-Journal of Chemistry, 7, pp. 1334-1339.

Jelveh, S. and Chithrani, D.B., 2011. Gold nanostructures as a platform for combinational therapy in future cancer therapeutics. Cancers, 3, pp. 1081-1110.

Li, Y., Schluesener, H.J. and Xu, S., 2010. Gold nanoparticle-based biosensors. Gold Bulletin, 43, pp. 29-41.

Liu, J. *et al*. 2009. Nanomaterial-assisted signal enhancement of hybridization for DNA biosensors. Sensors, 9, pp. 7343-7364.

Milliron, D.J. *et al*. 2003. Electroactive surfactant designed to mediate electron transfer between CdSe nanocrystals and organic semiconductors. Advanced Materials, 15, pp. 58-61.

Nadzirah, S., Ahmad, S and Hashim, U. 2012. Titanium Dioxide Nanowires-Based Interdigitated Electrodes for Biomedical Application. (December), pp. 126-128.

Nagender Reddy Panyala, Eladia María Peña-Méndez and Josef Havel. 2009. Gold and nano-gold in medicine: overview, toxicology and perspectives. J Appl Biomed, 7, pp. 75-91.

Petkov, V., 2008. Nanostructure by high-energy X-ray diffraction. Materials Today, 11, pp.28–38.

Piryatinski, A. *et al*. 2007. Effect of quantum and dielectric confinement on the exciton-exciton interaction energy in type II core/shell semiconductor nanocrystals. Nano Letters, 7, pp.108-115.

Song, J.Y., Jang, H.K. and Kim, B.S. 2009. Biological synthesis of gold nanoparticles using Magnolia kobus and Diopyros kaki leaf extracts. Process Biochemistry, 44, pp. 1133-1138.

Stevens, P.W., Henry, M.R. and Kelso, D.M. 1999. DNA hybridization on microparticles: Determining capture-probe density and equilibrium dissociation constants. Nucleic Acids Research, 27, pp. 1719-1727.

Suh, K.Y. *et al.*, 2004. Soft lithographic patterning of hyaluronic acid on hydrophilic substrates using molding and printing. Advanced Materials, 16, p. 584–+.

Xing, Z.-C., Chang, Y. and Kang, I.-K. 2010. Immobilization of biomolecules on the surface of inorganic nanoparticles for biomedical applications. Science and Technology of Advanced Materials, 11, p. 014101.

Yang, S.M. *et al*. 2006. Nanomachining by colloidal lithography. Small, 2, pp. 458-475.

Yonehara, T., Yamaguchi, M. and Tsuji, K. 2010. X-ray fluorescence imaging with polycapillary X-ray optics. In Spectrochimica Acta - Part B Atomic Spectroscopy. pp. 441-444.