



Enhanced Raman spectroscopy analysis for contamination detection on microelectronic devices using gold nanoclusters grown by DC magnetron sputtering

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Received date:

7 March 2023

Revised date

22 June 2023

Accepted date:

26 June 2023

Keywords:

Surface-enhanced Raman scattering;
Sputtering;
Gold nanoclusters;
Microstructure;
Failure analysis

Abstract

Surface-enhanced Raman spectroscopy (SERS) is one of the most powerful analytical techniques for the identification of molecules in the microelectronics industry for failure analysis protocols. In this work, dry-processed gold nanoclusters were prepared by magnetron sputtering deposition to promote the enhancement of the Raman signal from selected common polymers found in the hard disk drive as surface contamination. The optimized sputtering conditions were applied for SERS on polycarbonate (PC), polyethylene terephthalate (PET), polypropylene (PP), and high-density polyethylene (HDPE). The Raman spectrum showed the average Raman signal intensity gain at about 114%, 78%, 254%, and 226%, respectively. The SERS with gold nanoclusters, prepared by magnetron sputtering, demonstrates that this method is a clean, simple, highly performing analytical method for failure analysis and can be an alternative method over the use of colloidal gold nanoparticles for contamination investigation in industrial failure analysis procedures, where the sample cleanness during the analysis is critical, as in the microelectronic industry.

1. Introduction

In the microelectronics industry, failure analysis is a technical procedure to investigate the root causes of why a product, equipment, an unintentional mistake in the design or manufacture, or any hidden problems in a continuous process fail [1]. A hard disk drive (HDD) is a magnetic data storage device that contains both mechanical and electrical parts that work together during HDD operation. Inside the HDD, there are many important polymer components that, sometimes accidentally, can be the source of the contamination issue inside the HDD. These polymers can form smears or flakes on the surface of important devices and interfere with their operation. Raman spectroscopy is one of the important characterization techniques that have been used in failure analysis to detect and identify the type of contamination that occurs during the production process.

Normally, Raman spectroscopy is the conventional technique for detecting HDD failures caused by contamination on the HDD read-write head and media for most cases. However, the Raman spectroscopy technique still has some limitations. In some cases, the contaminations on the HDD component, especially the read-write head, are difficult or even can't be detected and identified due to the volume and size of the thin-smear contamination and the size of the specimen. As the sample or surface contamination is a very small

area and thin, this causes very weak Raman signals or is obscured by fluorescence noise. Fluorescence noise is a molecular phenomenon in which a substance radiates light energy almost instantly upon being struck with light from another source. One method to amplify the weak Raman signals is to employ surface-enhanced Raman spectroscopy or scattering (SERS).

SERS is a surface-sensitive technique for significantly enhancing Raman scattering from molecules adsorbing on rough metal surfaces [2,3]. SERS uses local electric-field enhancement by optical near-field effects such as localized surface plasmons (LSP) to enhance Raman scattering. SERS substrates are typically fabricated of plasmonic resonance-supporting materials, mainly gold and silver but also copper and aluminum [4]. Moreover, in the field of nanoscience and technology, metallic nanostructures' optical characteristics are extremely crucial. The local electromagnetic field (EM), which is enhanced by the localized surface plasmon resonance (LSPR) of metallic surfaces with nanoscale features, considerably enhances the signal from molecules adsorbed on the surface of plasmonic nanostructures [3,5]. Gold or silver are typical noble metals for which the surface preparation can be through electrochemical roughening, a metallic coating of a nanostructured substrate, or the deposition of metallic nanoparticles (often in colloidal form) [6]. The interaction between adsorbed molecules and the surface of plasmonic nanostructures is crucial to

the success of SERS [7]. SERS technique for each specified purpose significantly depends on the selection of the SERS substrates; for example, LSP in silver nanostructures typically suffers from rapid silver degradation in ambient atmospheric conditions [8]. However, silver gives a higher enhancement of the Raman signal than gold.

Commonly, the simplest method for the preparation of plasmonic silver and gold nanoparticles for SERS is in the form of a colloidal solution. However, these colloidal gold nanoparticles can introduce more contamination on the surface of samples due to the chemical stabilizer agents and the difficulty of controlling the gold nanoparticle distribution on the surface, which makes them unsuitable for identifying unknown surface contamination in the microelectronics industry.

In this work, we have fabricated the gold nanoclusters by DC magnetron sputtering technique, on top of the melted flake polymer specimens, which were chosen from the common contaminations found on the surface of the failed device or component in the micro-electronic industry. The sputtering conditions to achieve the deposition of gold nanoclusters have been optimized. These as-sputtered gold nanoclusters were utilized as an alternative to the colloidal gold nanoparticles without introducing more contamination to the surface of the test specimen. The Raman signal amplification from the localized surface plasmon resonance (LSPR) phenomenon from the gold nanoclusters-polymer interaction has been investigated. The controlled temperature base will be created to improve the Raman signal due to the plasmon effect. Improving and enhancing the quality of the Raman signal and the ability to identify thin-smear contamination in this research will shorten the time and develop the failure analysis method.

2. Experimental methods

Several contamination polymers that are commonly found in HDD sliders, i.e., polycarbonate (PC), polyethylene terephthalate (PET), polypropylene (PP), and high-density polyethylene (HDPE), were prepared as SERS test specimens on both glass slides and $\text{Al}_2\text{O}_3\text{-TiC}$ (AlTiC) substrates. The flakes of those polymers were spread on the substrates and melted on a hot plate. The gold nanoclusters were fabricated by using the BALZERS SCD 040 DC sputter deposition system in a vacuum environment. The sputtering process was done at a pressure of about 1 mTorr of argon (Ar) gas and a target-to-substrate (T-S) distance of 7 cm. During the sputtering process, the 2-inch diameter of a 99.99% pure, solid gold disc sputter target was sputtered using Ar^+ plasma to create the gold nanoclusters. These gold nanoclusters were deposited on top of the prepared polymer specimens at room temperature. The sputtering current and deposition time were controlled at the optimum conditions for the fabrication of gold nanoclusters for SERS specimens.

2.1 Sample preparation

The polymer samples were prepared on the glass slides by melting each type of polymer flake using a hot plate. The polymers were melted on a hot plate at temperatures of 265°C, 250°C, 150°C, and 140°C for PC, PET, PP, and HDPE, respectively. These polymers were chosen from the common contaminants found on the surfaces of failed devices

or components in the microelectronic industry. The Raman spectra were taken before and after the fabrication of gold nanoclusters on the melted polymer samples.

2.2 Microstructure and Raman spectroscopy measurements

The JEOL JSM7001F field emission scanning electron microscope (FE-SEM/EDS) was used to investigate the microstructure information, size, and distribution of the gold nanoclusters on the polymer SERS samples after the sputtering deposition. The information from SEM/EDS was used to identify the optimum conditions for gold sputtering parameters to achieve the growth of gold nanoclusters.

All the SERS experiments were carried out using a Thermo Fisher Scientific™ DXR3 Raman microscope, equipped with 532 nm laser wavelengths. The excitation laser has been operated at a power of 10 mW, 2 s of exposure time, and 2 accumulations. The spectrum grating was 900 lines/nm, the apertures were 50 μm pinhole, and the 10x microscopic objective lens was used throughout all the experiments. All the Raman spectra have been measured at least five times for reproducibility.

3. Results and discussion

3.1 Optimizing the gold nanoclusters sputtering condition.

To optimize the gold sputtering conditions to form gold nanoclusters on the prepared polymer samples. The Raman spectra of different sputtering conditions were measured as shown in Figures 1(a-b) for polyethylene terephthalate and polycarbonate, respectively. Varying the sputtering current in the sputtering process can control the number of ions (Ar^+) from the plasma that strikes the sputtering target. This determines the average number of gold atoms ejected from the target toward the substrate. The deposition time needs to be controlled to allow the deposition of gold nanoclusters on the specimen and not to reach the coalescence or film-forming stage where the layer(s) of gold thin film is formed. From the Raman spectra in Figures 1(a-b), it can be observed that the Raman signals of the specimen with sputtered gold nanoclusters were increased, compared to the specimen without gold nanoclusters. However, as layers of gold thin film started to form at about 15 mA of sputtered current and 60 s of deposition time, the Raman signal noticeably decreased. It was found that the suitable sputtering condition for SERS specimen fabrication, which yielded the best Raman intensity gain on both polyethylene terephthalate and polycarbonate, can be achieved at about 5 mA of sputtered current and 15 s of deposition time. This optimized sputter condition was applied in the preparation of SERS specimens for all interested polymers.

3.2 Morphology investigation of sputtered gold nanoclusters

The microstructure and surface morphology of the samples with gold nanoclusters deposited on top of the prepared polymer SERS specimens were observed under FE-SEM. The size and distribution of the gold nanoclusters are clearly seen in the electron micrograph, shown in Figure 2(a).

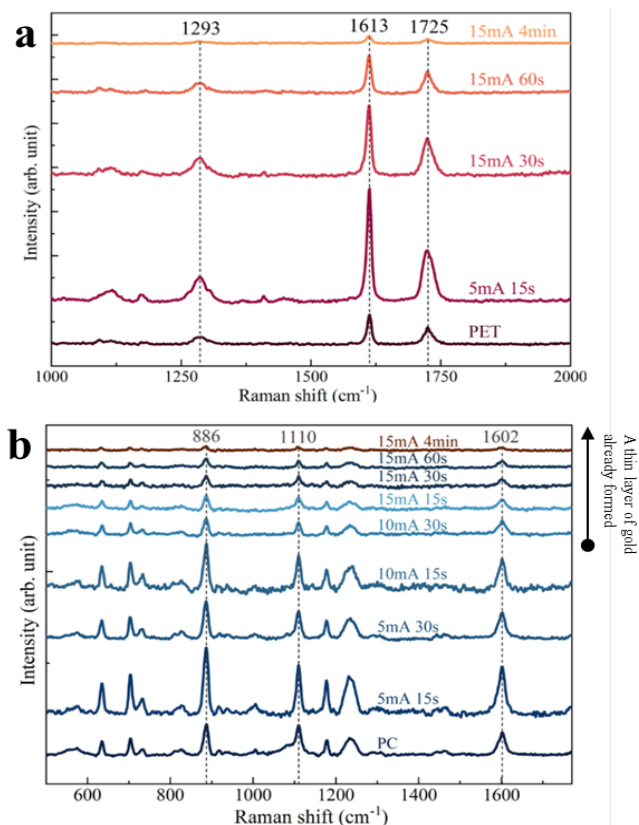


Figure 1. Raman spectra of (a) PET and (b) PC SERS specimens with the different sputtering conditions of gold nanoclusters. Note that, as the deposition time reached the stage where a thin layer of gold film was formed, the Raman signals decreased (indicated by the arrow).

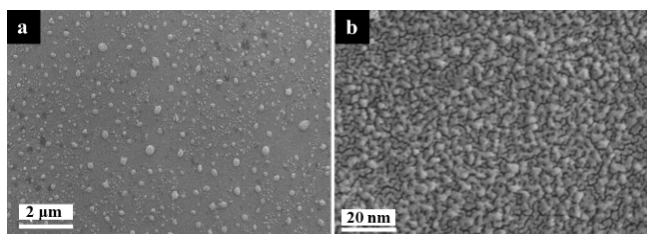


Figure 2. The electron micrographs of gold nanocluster formation (a) at the optimized sputtering condition ($I = 5 \text{ mA}$, $t = 15 \text{ s}$), and (b) at the condition in which the gold layer started to form ($I = 15 \text{ mA}$, $t = 30 \text{ s}$).

The size of gold particles was measured from the electron micrographs, obtained from SEM/EDS. The gold nanoclusters (AuNCs) fabricated at an ion current of 5 mA and a deposition time of 15 s have a cluster size between 50 nm to 200 nm. The variation in the cluster size can be attributed to the deposition process being carried out at room temperature. This results in the atoms and clusters landing on the substrate surface lacking surface kinetic energy, which is a common issue for thin solid film formation at low temperatures by the sputtering process [9].

Typically, the gold nanoclusters use the LSP to enhance the Raman signal in SERS. As the surface plasmon is contained in a nanoparticle that is the same size or smaller than the wavelength of light used to excite the plasmon, a confined surface plasmon is generated. The surface plasmon excitation results in the generation of a strong secondary field, which Raman scatters from the molecules, causing an electromagnetic

enhancement that enhances the Raman signal [10]. The Raman scattering is caused by the interaction of the substrate plasmon field from gold nanoclusters and the molecule field. Since the intensity of the electromagnetic field above the substrate falls with the distance normal to the surface there, SERS has a distance dependence [11]. SERS generates an electric field from gold nanoclusters, however, if the nanoclusters distribution becomes dense, the Raman signal is reduced and blocked, as seen in Figures 1(a-b), when the sputtering current or deposition time are increased beyond the optimized parameters. After the thin film growth mechanism reached the step where the layer(s) of gold thin film were formed, as shown in Figure 2(b), with a sputtering current of 15 mA for 30 s of deposition time. The Raman signal obviously decreased, as compared to the original signal.

Nanoparticles have been synthesized using a variety of chemical and physical methods. The chemical methods are based on colloidal solutions being formed by the reduction of the metal compound. Stabilizers are present in these solutions to stop the aggregation of nanoparticles, but the byproduct has a problem, so following the synthesis, purification actions must be performed [12]. One of the most affordable physical processes for creating pure materials and nanomaterials is sputtering. Nanoparticles prepared by direct sputtering contain fewer impurities than those prepared through chemical synthesis, and these nanoparticles are frequently also very stable over a long period of time [13].

3.3 Gold nanoclusters SERS of polycarbonate, polyethylene terephthalate, polypropylene, and high-density polyethylene

One of the main mechanisms affecting SERS with gold nanoclusters is the distance between each gold nanoclusters and the structure of the sample. The organic functional groups in PC are linked by carbonate groups and polar side groups, which causes PC to emit very weak Raman signals. The SERS spectrum of PC with and without deposited gold nanoclusters at Raman peak positions of about 886 cm^{-1} (C-O-C) [14], 1110 cm^{-1} (C-C) [15], and 1602 cm^{-1} (C=O) [16] is shown in Figure 3(a). The PC specimens with sputtered gold nanoclusters SERS, exhibit the maximum Raman intensity increasing by about 114%, as detailed in Table 1.

The comparison Raman spectrum of PET specimens with and without gold sputtered gold nanoclusters at Raman peak positions of about 1293 cm^{-1} (C(O)-O stretching), 1613 cm^{-1} (Ring mode 8a in Wilson's notation), and 1725 cm^{-1} (Stretching C=O vibration) [17] is illustrated in Figure 3(b). The PET sample with deposited gold nanoclusters as SERS shows an increasing intensity of about 78%, as seen in Table 1. Like PC, the PET has a polar group, which means that its Raman signals are particularly weak, therefore, the interaction between the PET and gold nanoclusters can help boost the Raman signal [18].

For PP, The SERS spectrum of PP with and without deposited gold nanoclusters at Raman peak positions of about 393 cm^{-1} , 803 cm^{-1} (C-C) [15], and 1453 cm^{-1} (CH_2 bending, CH_3 asymmetric bending) [19] is shown in Figure 3(c). Due to the non-polar polymer nature of PP, it typically provides quite a decent Raman signal. Therefore, with sputtered gold nanoclusters, PP can give a strong enhanced SERS signal [20]. The PP SERS specimen with sputtered gold nanoclusters SERS exhibits a Raman intensity increase of about 254%.

Polyethylene (PE) is a non-polar polymer that has a simple molecular structure without the ionic bonds found in other polymers [21]. Like generic PE, HDPE is also a non-polar polymer that normally provides a quite good Raman signal, similar to PP. The HDPE specimens with sputtered gold nanoclusters SERS, exhibit Raman intensity increasing by about 226%, with details of intensity at Raman peak positions of about 1054 cm^{-1} (C-C), 1123 cm^{-1} (C-C) [15], and 1289 cm^{-1} (CH₂) [22] shown in Figure 3(d) and Table 1.

Normally, the polarizability of a bond can indicate Raman activity. This measures how easily a bond will deform under the applied electric field. It essentially depends on how easily a temporary dipole can be created by displacing electrons in a bond. The polarizability is large when there is a large concentration of loosely held electrons in a bond, and the group or molecule will have a strong Raman signal. As a result,

rather than being sensitive to a particular functional group, Raman is typically more sensitive to the molecular framework of a molecule [23].

The close holding of electrons by electronegative atoms causes the Raman signals of polar molecules, such as polycarbonate, to generally be very weak [11]. Thus, metallic nanostructures' optical characteristics are extremely crucial. The local electromagnetic field enhancement is caused by the LSPR of metallic surfaces with nanoscale features being excited. This significantly boosts the signal from molecules adsorbed to the surface of plasmonic nanostructures [24]. The interaction of analyte molecules with a substrate surface caused the specific enhancement at each peak. The active metal surface's adsorption sites, adsorption orientation, and the adsorbed molecule's vibrational modes have an effect on this interaction [25].

Table 1. Raman signal enhancement using sputtered-gold nanoclusters SERS on PC, PET, PP, and HDPE.

Polymer	Raman shift (cm ⁻¹)	Average (%)	
		Intensity gain	Max intensity gain
PC	886	114	186
	1602	92	173
	1110	61	119
PET	1616	78	113
	1725	47	65
	1293	59	71
PP	1453	221	362
	803	254	355
	393	249	342
HDPE	1289	226	422
	1123	274	425
	1054	343	501

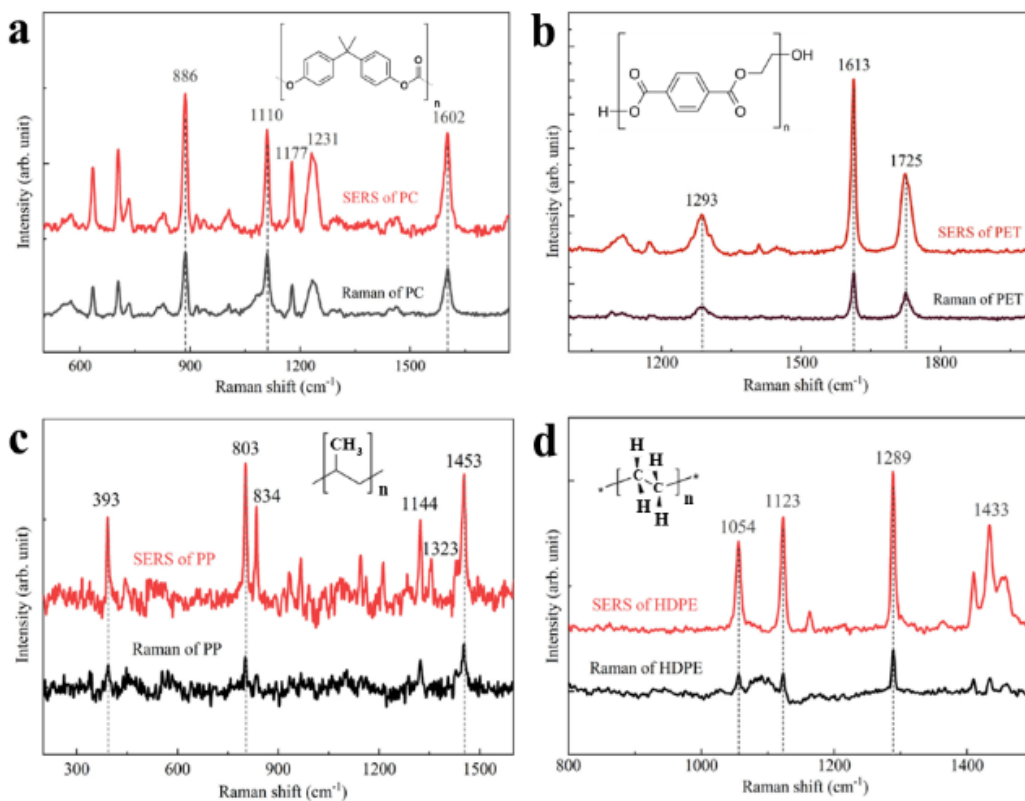


Figure 3. SERS spectra of (a) PC, (b) PET, (c) PP, and (d) HDPE with and without using a sputtered-gold nanocluster on top of the surface.

Backbone vibrations typically do not induce changes to the dipole moment, although polarizability is usually high because repeating units cancel out adjacent dipoles in long polymer chains. Therefore, Raman spectroscopy is particularly useful as it is highly sensitive to the structure and conformation of the polymer backbone [26]. Additionally, different polymers will lead to different boosts in SERS intensity.

Raman and IR spectroscopies can be used to analyze side chains and the backbone structure and conformation of polymers, respectively [27]. Polymers uniquely exhibit the phenomenon of chain orientation, and due to the adjacent dipoles being canceled out by repeating units, the backbone of long polymer chains vibrating results in considerable changes in polarizability but no change in the dipole moment, consequently, it can display the specific Raman signal [28]. Thus, the non-polar polymers are usually providing a good intensity Raman signal, with sputtered gold nanoclusters as SERS, their Raman signals can be easily amplified, as in the PP and HDPE cases.

4. Summary

Surface-enhanced Raman spectroscopy (SERS) is one of the most powerful analytical techniques for the identification of molecules, especially on the surface of microelectronic devices as a surface contamination analysis tool. The vibrations that modify the polarizability of a molecule are detected by Raman spectroscopy. This research work provided an alternative approach for a clean, fast, and economical method based on SERS for failure analysis. Each polymer has a different structure and different SERS activity because of the polar structure and the distance between functional groups and gold nanoclusters. With our optimized, sputtering conditions applied for SERS specimen preparation on polycarbonate (PC), polyethylene terephthalate (PET), polypropylene (PP), and high-density polyethylene (HDPE), it yielded maximum intensity gain for the Raman signal at about 186% for PC, 113% for PET, 362% for PP, and 501% for HDPE, respectively. Compared with the use of colloidal gold nanoparticles that contained stabilizer agents, the sputtered gold nanoclusters provide a clean, simple process that is suitable for failure analysis in the microelectronics industry.

Acknowledgements

The authors would like to thank the Interdisciplinary program in Nanoscience and Technology, Graduate School, Chulalongkorn University and Western Digital Storage Technologies (Thailand) Ltd. for their supports.

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