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Effect of Current Density on Porous Silicon (pSi) for Non-Invasive Dengue Detection SERS Substrates: Electrical and Structural Property

N. F. Ismail, A. R. M. Radzol*, L. N. Ismail, Khuan Y. Lee, A. Z. Zulhanip and N. S. Mohamad Hadis

***Abstract*—Surface-Enhanced Raman Spectroscopy (SERS) is an ultra-sensitive and vibration-specific spectroscopy technique. It enhances Raman scattering by adsorbing molecules to roughen metal surfaces or colloidal nanoparticles known as SERS substrate. Enhancement performance of SERS is highly dependent on the type of substrate. This study sets out to examine the factors influencing the surface formation of porous Silicon (pSi) structure that is intended for SERS substrate for non-invasive Dengue detection at the febrile stage. The current density (*J* mA/cm^2) in electrochemical etching process is one of such factors. Results of samples with different current density are documented in structural and electrical property. Field Emission Scanning Electron Microscopy (FESEM) images and surface structure of the SERS samples and cross-shapes porous structure are investigated. The current-voltage (I-V) property, conductivity versus resistivity property and sensitivity property of pSi structure are also examined. Results on structural property show that dimensions of the cross-shaped structures at different current density are visually almost the same. The averaged dimension ranges from 2.632 to 3.719 µm and depicts indeterminate trending with the current density. From the electrical property perspective, the I-V characteristic graphs of all samples show an exponentially rising trend, that bear similarity to the characteristic of diode. Besides, conductivity is found to increase with the current density. The ideal current density for producing porous structure is found to be that for the j-20 to j-28 samples, 20 to 28 mA/cm^2**

***Index Terms*—Porous Silicon (pSi), Surface-Enhanced Raman Spectroscopy (SERS), Fabrication.**

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N. F. Ismail, A. R. M. Radzol, L. N. Ismail, A. Z. Zulhanip and N. S. Mohamad Hadis are with School of Electrical Engineering, College of Engineering, Universiti Teknologi MARA, Cawangan Pulau Pinang, 13500, Permatang Pauh, Pulau Pinang, Malaysia.

Khuan Y. Lee is with School of Electrical Engineering, College of Engineering, Universiti Teknologi MARA, 40450, Shah Alam, Selangor Darul Ehsan, Malaysia.

A. R. M. Radzol, Khuan Y. Lee and L. N. Ismail are with the Computational Intelligence Detection RIG, Pharmaceutical and Lifesciences Communities of Research, Universiti Teknologi MARA, 40450, Shah Alam, Selangor Darul Ehsan, Malaysia (E-mail address: afafrozan944@uitm.edu.my)

*Corresponding author
Email address: afafrozan944@uitm.edu.my

I. INTRODUCTION

SERS is an application of plasmonic effect of metal nanostructures. Plasmonic is gaining prominence due to its ability to capture and modulate light of nanoscale structures. Plasmonic semiconductor enhances the efficiency of hot electrons and thus widens the range of spectrum response considerably, in particular within the visible and near-infrared region, owing to the formation of nanoscale structures [1], [2].

Surface plasmons concentrated on nanostructures with a particular frequency, or better known as localized surface plasmons (LSP), are considerably smaller than the wavelength of light, being confined to the nanostructures. The LPS of metals with nanoscale structure surface characteristic excitation produce local electromagnetic (EM) field enhancement. The amplified EM fields can be detected by a highly sensitive and powerful vibrational spectroscopy technique, known as SERS [3], [4]. Enhanced Raman scattering of SERS is associated with highly localized regions of intense local EM field, caused by surface plasmon resonance. SERS overcomes the inherent limitation of the very weak Raman scattering signals, which is particularly useful, where the sample concentration is low [4]. The LPS wavelength is influenced by the geometrical characteristics of nanoscale structures, chemical composition of metallic nanoparticles (NPs), distance between NPs on surface structures and the related materials, besides processes [3]. The alternating plasmonic wavelengths ranging from ultraviolet, visible, near-infrared to far-infrared obtain from various morphologies and structures formation occur in metals, semiconductors and two-dimensional materials. Theoretically, computational and experimental studies have confirmed that various light-matter interaction processes including fluorescence, Raman scattering, heat generation, photoacoustic effects, photocatalysis, non-linear optical conversion, and solar energy conversion can significantly enhance the order of magnitude within plasmonic structures [3], [5].

The interaction of adsorbed molecules with the surface of plasmonic nanostructures is critical to the success of SERS, with classic substrates of gold (Au), silver (Ag), or copper (Cu). Effective metal plasmons, Au and Ag, are frequently employed as SERS substrates, for their stable-in-air property meanwhile, Cu is more reactive. The LSP of all three metals spans across majority of the visible and near-infrared wavelength range where most Raman observations take place [6]. The nanomaterials contribute to charge transfer, which

increases the Raman signals through the form of electrons. Researchers have spent the last thirty years attempting to improve the SERS substrate structure to popularize its usage.

According to literature review, nanofabrication, colloidal synthesis and self-assembly are techniques most frequently researched [7], [8]. At the early stage of research, the focus is on enhancing the Raman signals with surface roughness of SERS substrates, characterized by plasmonic NPs on polymers or porous semiconductors [1], [9]–[14]. Then, limit of detection for chemical composition deposited on the surface is investigated [8], [14]. Lately, optimization of substrate formation is explored for its effective deployment in different sensing applications.

Porous silicon (pSi) has been extensively studied for use as label-free, low-cost optical biosensor [15]–[18]. The pSi structure is produced from crystalline Si through electrochemical etching process. Its surface structure formation depends on the preparation conditions. Furthermore, pSi features are adapted in tune with application for biomedical, solar cells and sensors et al [16]. For SERS, the main advantage of pSi lies in the high surface area produced from etching process, which results in substantial increase in Raman intensity [8], [9], [15]. In other words, the pSi structure enables the enhancement of Raman signal.

A wide range of approaches has been used to produce pSi structures. The microelectronic manufacturing method is used to specify and fabricate surface nanostructures. The production of nanoscale substrate from Si wafer is accomplished through several processes: the advanced lithography technique generates a nanoscale pattern, to be followed by material removal (etching) or material deposition. There are two main categories of fabrication technique. In the first category, the bottom-up technique which literally uses chemical synthesis as the main process for sample production. This method is well-known for its cost-effectiveness and convenience in implementation since the chemical composition can be modified for optimal performance. In the second category, the top-down technique involves the lithography process, such as optical and electron beam (E-beam) lithography (EBL), focused ion beam and nanoimprint lithography, which is advanced yet expensive. However, it produces substrates with exceptional functional uniformity and outstanding SERS signal reproducibility, both of which are desirable in contemporary SERS applications [19], [20].

For our work, the electrochemical etching process adapted from the top-down technique is adopted. Self-assembly is required in this process, which involves several chemical and physical processes in the SERS formation. The technique is chosen owing to its cost-effectiveness. Besides, optimal sample production is possible with control over variables such as size and shape of porosity formation; chemical composition and percent concentration for an aqueous solution of ethanol (C_2H_5OH) and hydrofluoric (HF) acid to perform porous structure. The electrical conductivity of solid platform is another factor with substantial impact on the performance of SERS substrate. Intensity of Raman signal improves with materials of superior conductivity [21]. In this study, pSi structure is chosen as the solid platform for SERS substrate to be developed for detection of DENV-NS1 protein.

Dengue virus (DENV) is a mosquito-borne illness found in tropical and subtropical regions [22], [23]. In recent years, its rapid transmission has become a serious global public health concern, mostly in urban and semi-urban areas [24]. DENV 1-4 are the four antigenically different serotypes of DENV believed to bring about Dengue diseases [23], [24]. Yellow fever, Japanese encephalitis virus (JEV), West Nile virus (WNV), St Louis encephalitis virus belong to the Flavivirus/Flaviviridae genera [25]. Significant genetic diversity amongst the virus serotype results in phylogenetically different genotypes. The virion consists of three structural proteins, a lipoprotein envelope and seven non-structural proteins. The non-structural protein 1 (NS1) has diagnostic and pathological importance [22]. Dengue infection is determined by viral genomic RNA, antigens or antibodies that it triggers. The DENV NS1 protein is produced from Dengue-infected cells and appears early in the plasma. Therefore, NS1-based antigen detection assays are developed for detection of the DENV NS1 protein [23], [26].

Precise clinical diagnosis of dengue is challenging because its symptoms are diverse. As a result, laboratory or point-of-care diagnostics must be utilized in combination with clinical evaluation [26]. Several methods are for diagnosing DENV infection. The traditional method has been virus isolation. Then, there are molecule based reverse-transcription polymerase chain reaction (RT-PCR), nucleic acid hybridization, NS1 antigen-captured enzyme linked immunosorbent assays (ELISAs) and rapid diagnostic test (RDTs), which is the most recent [26].

Virus isolation works on patient blood samples collected 5 days after the onset of disease. The PCR-based technique identifies DENV during the acute phase of infection on the same day or the following day. The ability to identify viral RNA from the beginning of disease infection is an advantage of PCR-based methods. In addition, the techniques are sensitive, precise, time efficient and simple. However, the equipment for this technique is bulky and specialized, which demand the operation by well-trained staff. The antigen-capturing ELISAs is an example of serological diagnosis. The NS1-based ELISAs is a commercial development, which has revolutionized dengue diagnosis method. It uses simple and low-technology assays with high sensitivity and specificity. RDTs are test kits that can detect infected patient rapidly with reasonably reliable results [26].

Our work here intends to examine the performance of the structural and electrical property of pSi structure in response to different current density applied during the electrochemical etching process. Section II explains the framework for fabricating the SERS substrate for dengue detection. It elaborates on the methods used to characterize the surface structural and electrical property of the pSi structure. Section III discusses observations and findings obtained from the characterization.

II. Methodology

Fig. 1 illustrates the conceptual model for our research work [27]. Fig. 2 shows the steps involved from fabricating the pSi using electrochemical etching process up to the characterization of sample production, where the pSi structure is subjected to different values of the current density.

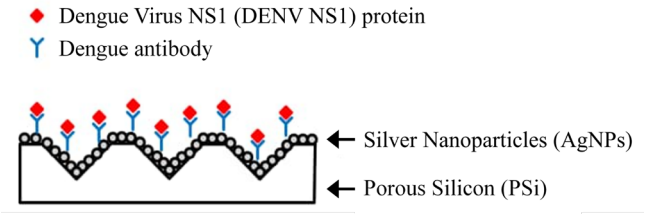

Fig. 1 Conceptual model of DENV-NS1 protein SERS substrate.

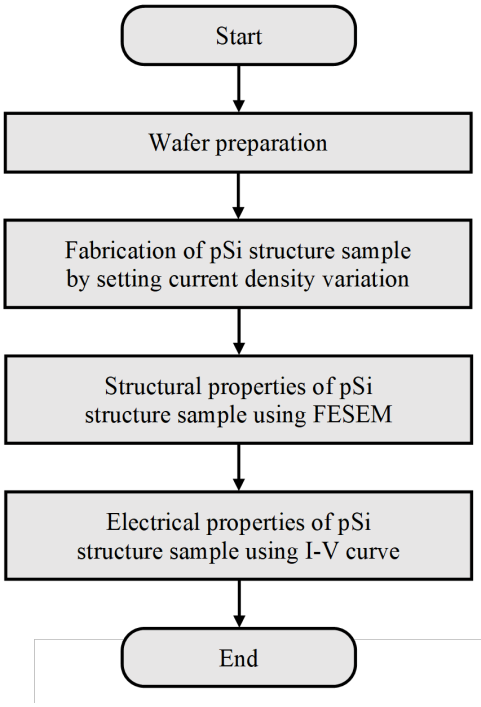

Fig. 2 Research methodology.

A. Porous Si structure formation

The procedure to fabricate pSi for SERS substrate base has been described in our prior work [27]. The experiment investigated surface of the pSi structure in response to different current density. Here, the duration of etching was fixed at 20 minutes. A DC power supply model GW INSTEK GPD-4303S was used to control the setting for voltage and current. The initial voltage was set to 10 V and current to 10 mA, which represents a current density (*J*) of 20 mA/cm^2. Equation (1) calculates the current density as follow,

$$J\left(\frac{mA}{cm^2}\right) = \frac{I\ (mA)}{Area\ (cm)} = \frac{x\ (mA)}{\pi\ (0.4)^2} \qquad (1)$$

J = Current density
I = Current setting at power supply (variable)
r = 0.4 (size of O-ring)

TABLE I
RANGE OF THE CURRENT VALUES SETTING REPRESENT THE DIFFERENT VALUES OF CURRENT DENSITY

Current, I (*mA*)	Current density, *J* (*mA/cm^2*)
10	20
12	24
14	28
16	32
18	36
20	40

The current was varied from between 10 to 20 mA to produce the different current density, *J* from 20 to 40 mA/cm^2. Table I tabulates the current density values between 20 until 40 mA/cm^2 at an interval of 4 mA/cm^2 and the corresponding current values. The different current density samples were labelled as j-20, j-24, j-28, j-32, j-36 and j-40 respectively.

B. Electrical and Structural Property of pSi structure

The electrical property of pSi structure was analyzed using Keithley 4200-SCS PK2 Parameter Analyzer. The voltage was swept from -10 to +10 V and applied to the pSi structure to produce the I-V characteristic.

The surface morphology and topography of the pSi structure was characterized by the field emission scanning electron microscopy (FESEM) (Model: JEOL JSM 7401F). The NanoScope Analysis software was used to analyse the surface roughness and the estimated pore depth of the samples with a scan area of 5 µm × 5 µm and 10 µm × 10 µm. The features of scan area selected for samples were discussed in [27].

The FESEM measures the cross-shape on the pSi structure surface at different current density. The current density is found to limit the parameter range selection in producing the best pSi structure to serve as base layer of the SERS substrate.

III. Results and Discussions

A. Structural Property of pSi structure at different current density

The structural property of pSi surface from top-view at different current density of the etching process are shown in Fig. 3. Microsized cross-shaped structures are observed. Visually, the dimensions of the cross-shaped structures at all current density values look almost the same. The averaged dimensions are tabulated in Table II. The sizes range from 2.632 to 3.719 µm with indeterminate trend with current density. The largest averaged dimension of cross-shaped pSi structure is obtained when the current density is 36 mA/cm^2.

Fig. 4 shows the FESEM characterization of the pSi sample from cross-sectional view. It is observed again indeterminate trend of triangle opening and depth with current density. The largest triangle opening is attained when the current density is 32 mA/cm^2, while the deepest depth measurement is scored at current density of 24 mA/cm^2. It is worth noting that, the measurement of triangle opening and depth becomes more

challenging as current density increases from 32 to 40 mA/cm^2. It is difficult to place the measurement marker properly as the boundary of the triangle opening and depth is ambiguous due to surface roughening as shown in Fig. 4 (d)–(f).

The structural property of pSi from FESEM images as current density varies do not give a determinate trend with dimension of the cross-shapes from the electrochemical fabrication. Hence, investigation on the electrical property is conducted instead to explore the effect of current density on the pSi structure.

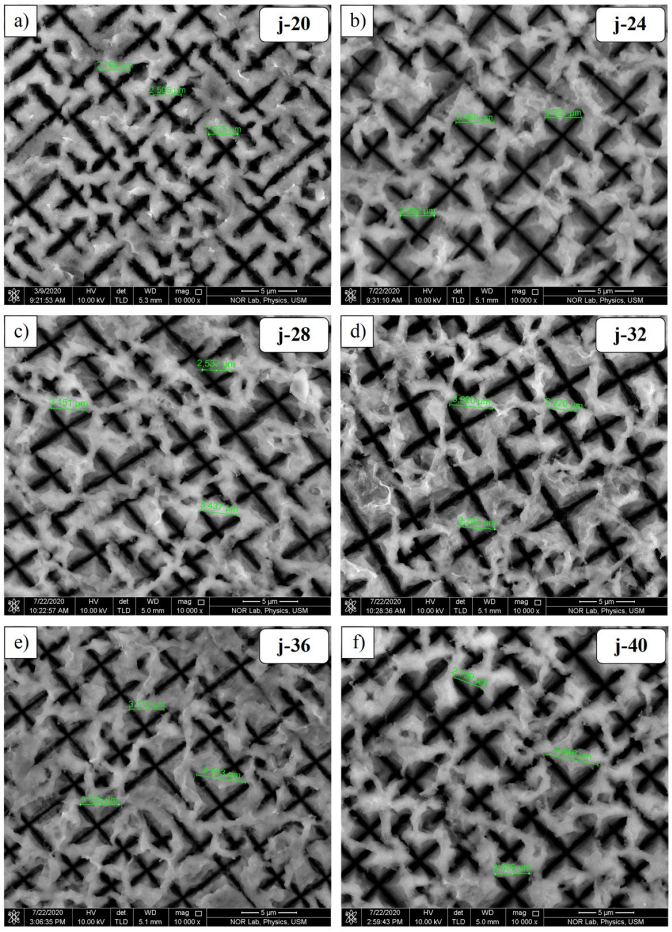

Fig. 3 FESEM characterization of pSi surface from top-view with 10000× magnification by setting different current density in the etching process (a) 20 mA/cm^2, (b) 24 mA/cm^2, (c) 28 mA/cm^2, (d) 32 mA/cm^2, (e) 36 mA/cm^2 and (f) 40 mA/cm^2.

TABLE II
DIMENSION OF CROSS-SHAPED POROUS SI AT DIFFERENT CURRENT DENSITY

Current density, J (mA/cm^2)	Top-view Average size (μm)	Cross sectional-view Average size (μm)	
		Triangle opening	Depth
20	2.632	3.684	7.502
24	3.303	4.423	11.003
28	3.042	3.932	8.480
32	3.161	4.688	7.791
36	3.719	4.468	6.238
40	3.558	4.053	6.563

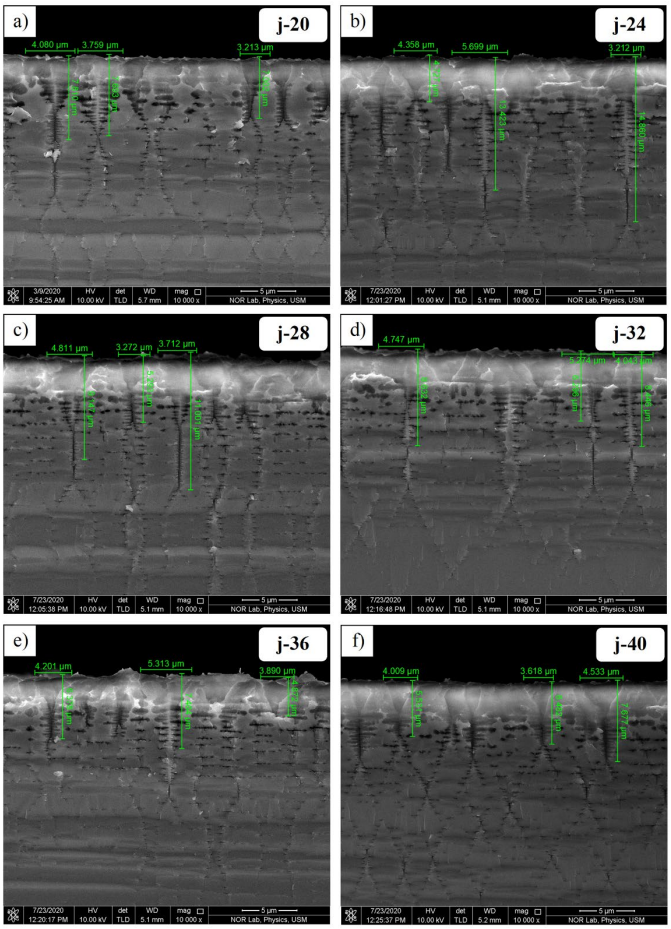

Fig. 4 FESEM characterization of pSi surface from cross sectional-view with 10000× magnification by setting different current density in the etching process (a) 20 mA/cm^2, (b) 24 mA/cm^2, (c) 28 mA/cm^2, (d) 32 mA/cm^2, (e) 36 mA/cm^2 and (f) 40 mA/cm^2.

B. Electrical Property of pSi structure at different current density

Fig. 5 (a) illustrates the I-V curves of the pSi samples at different current density. The applied voltage was swept from 0 to 10 V to observe the behaviour of the current. Six samples are collected at different current density values. With reference to the figure, j-20 to j-36 samples exhibit an exponentially increasing trend of current versus 0 to 10 V of voltage, which resembles the property of a diode. This is as expected since pSi is a semiconductor material. Meanwhile, j-40 shows a different behaviour where the current changes at slower rate starting from 6 V. Consequently, at 10 V, the current value for j-40 (~216.80 µA) is lower than j-36 (~240.18 µA). It is also observed, the current values increase as current density increases. The only exception is for j-40 when voltage increases from 8 to 10 V. Different I-V characteristic exhibited by j-40 is conjectured due to rougher structure as illustrated in Fig. 3 (f).

Besides, Fig. 5 (b) depicts the conductivity and resistivity versus different current density settings. Based on the graph, the conductivity is found the reciprocal of resistivity. These two graphs show that the pSi structure remains like a semiconductor material, in spite of the variation in current density, as the conductivity increases significantly while the resistivity decreases.

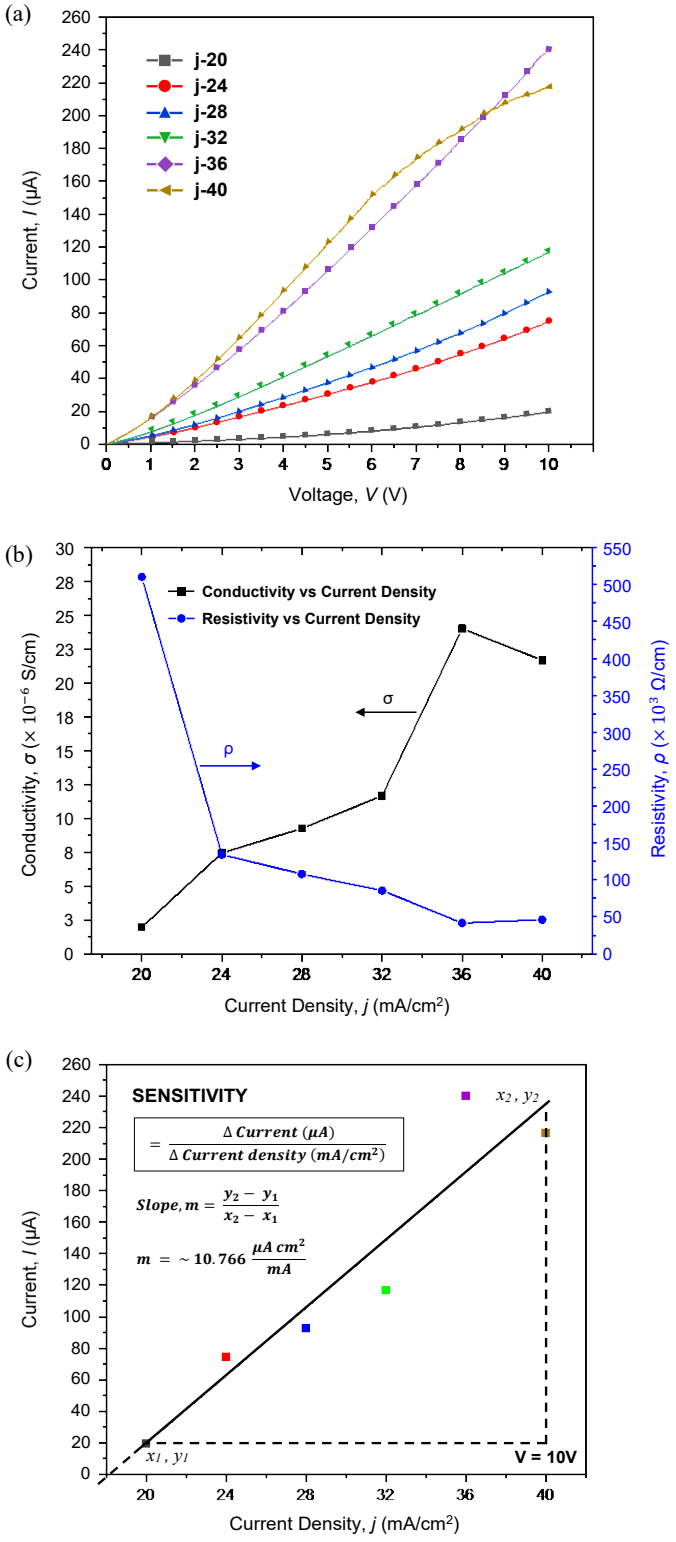

Fig. 5 Electrical Property for pSi structure. (a) I-V curve of the pSi structure at different current density, (b) Conductivity and resistivity property versus different current density setting (c) Sensitivity property of pSi at different current density.

The conductivity rises as current density increases from 20 to 36 mA/cm^2 and slightly drop for 40 mA/cm^2. Since the Si is doped with phosphorus dopant to become *n*-type semiconductor material, the amount of electrons is increased. This causes the conductivity of pSi to increase significantly. High conductivity means more electrons are generated and hence the current flow is intensified.

Fig. 5 (c) portrays the sensitivity property of the pSi structure with different current density settings. The sensitivity is measured from the slope of current versus current density. The slope measurement is extracted from the I-V curve as shown in Fig. 5 (a). The sensitivity measurement is taken from the slope, of the best-fit line for all the current density samples. The slope of current versus current density is ~10.766 μA cm^2/mA. From Fig. 5 (c), the j-20 sample is considered as the optimal current density for fabrication process, since it is located on the best-fit line. Also, it has the smallest triangle opening compared to the others (see Table II), thus making it the most sensitive. Furthermore, the j-24 and j-28 samples show current measurement next nearest to the optimal slope. The j-32 and j-40 samples display current measurement below the best-fit line while the j-36 sample is observed the farthest from the slope. Based on these sensitivity data, it can be inferred that the optimal current density is between that of the j-20 to j-28 samples, which produces porous structure with higher sensitivity.

IV. Conclusion

Our work here explores the effects of current density applied during electrochemical etching process on the structural and electrical property of pSi structure, intended for SERS substrates for non-invasive dengue detection. The effects are reported in Fig. 3, Fig. 4 and Fig. 5 and analyzed. From the FESEM images, the top-views show that dimensions of the porous cross-shaped surface structure for all current density appear almost the same. The averaged dimensions range from 2.632 to 3.719 μm and display an indeterminate trend with current density. The largest averaged dimension is obtained at a current density of 36 mA/cm^2. Hence, current density of 36 mA/cm^2 or less is found suitable to produce more uniform and porous cross-shaped surface structure. The cross-sectional view depicts an indeterminate trend of triangle opening and depth with current density. The largest triangle opening measurement is at a current density is 32 mA/cm^2, while the deepest depth measurement is at a current density of 24 mA/cm^2. The I-V characteristic of samples j-20 to j-36 exhibit an exponentially increasing pattern, which is similar to that of diode. The j-36 sample gives the highest current (~240.18 μA) compared to others at 10 V. The conductivity versus resistivity graph shows that the pSi structure retains its characteristics as a semiconductor material, despite variation in the current density. Conductivity increases as current density increases from j-20 to j-36; showing that SERS substrate can be produced with high conductivity and hence high current flow between these current density value setting. In addition to the above, from the sensitivity graph, it can be inferred that the optimal current density is that of the j-20 to j-28 samples, i.e. 20 to 28 mA/cm^2 which produce porous structure with highest sensitivity. The optimal current density setting will be applied to develop a SERS substrate with a good enhancement factor to detect low concentration NS1 protein from saliva of infected patients. For future work, the pSi structure surface coated with AgNPs will be

examined with a suitable Raman probe to study the Raman peak intensity dependent on the substrate structure and determine the enhancement for different structural layers in the SERS substrate.

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Noor Fadzilah Ismail was born in Kedah, Malaysia in 1996. She received B.E. (Hons) degree in Electrical & Electronics Engineering from Universiti Teknologi MARA, Cawangan Pulau Pinang, Permatang Pauh Campus, Malaysia in 2020. Since March 2020, she has been pursuing MSc degree in Electrical Engineering at Universiti Teknologi MARA, Malaysia. She became a research assistant under a Fundamental Research Grant Scheme (FRGS) and was engaged in the development of SERS substrates for bio-sensing application and Raman spectroscopy.

Afaf Rozan Mohd Radzol is a senior lecturer in School of Electrical Engineering, College of Engineering Universiti Teknologi MARA, Cawangan Pulau Pinang, Permatang Pauh Campus, Malaysia since 2004. She received her first degree in Electrical and Electronics from Universiti Tenaga Nasional, Malaysia in 2001. Later in 2004, she received MSc in Control Systems from University of Sheffield, UK. In 2018, she graduated in PhD in Electrical Engineering from Universiti Teknologi MARA, Malaysia. Her research interest includes artificial intelligence and signal processing in biomedical application and application of Raman spectroscopy in disease detection. Currently, she is doing her post doctorate in deep learning at Universiti Pertanian Malaysia.

Lyly Nyl Ismail was born in Kuala Lumpur, Malaysia, in 1978. She received the PhD. degree in Electronic Engineering specialized in nanocomposite material for electronic devices from Universiti Teknologi MARA, Malaysia in 2015. MSc degree in Microelectronic from Universiti Kebangsaan Malaysia in 2005 and B.E. degree in Electrical Engineering from Universiti Teknologi MARA, Malaysia in 2000. Currently, she is a senior Lecturer in School of Electrical Engineering, College of Engineering Universiti Teknologi MARA, Cawangan Pulau Pinang, Permatang Pauh Campus. She is the author of more than 60 articles, and more than 10 inventions. Her research interests include nanocomposite material for dielectric material for electronic devices and high voltage application.

Yoot Khuan LEE received her PhD in EE Eng (Biomedical) from Nottingham University, UK. She is now Prof at the School of Electrical Engineering, College of Engineering Universiti Teknologi MARA. She has authored more than 200 Scopus/WOS indexed technical publications in conferences and journals. Her research experience is in AI for Biomedical Engineeing, such as Analysis, compression and remote display of biomedical signals; Neonatal cry for physiologic status of baby; Neurofeedback for dyslexia children; Automated amperometric flow injection analysis system for human stress; Polysilicon based micro-cantilever biosensor for human stress; ChemFET biosensor for histamine; Salivary based disease detection from Raman spectra. She has won numerous International Awards of Excellence for her research products and technical papers. She is well recognized at the IEEE for her contribution and executive appointments with IEEE EAB and EMBS, and is awarded with numerous prestigious IEEE Awards.

Aida Zulia Zulhanip attained her B. Eng. (Hons) in Electrical Engineering (Electronics) from Universiti Teknologi MARA, Malaysia in 2007 and MSc in Electrical and Telecommunication Engineering from Universiti Teknologi Malaysia (UTM) in 2011. Her research interests involved are device fabrication and characterization, biosensor and audio engineering. She is currently working as senior lecturer at the School of Electrical Engineering, College of Engineering Universiti Teknologi MARA, Cawangan Pulau Pinang, Permatang Pauh Campus, Malaysia.

Nor Shahanim Mohamad Hadis was born in Kedah, Malaysia in 1981. She received her Bachelor (Hons) in Electrical Engineering from Universiti Teknologi MARA, Malaysia in 2005, MSc in Microelectronic from Universiti Kebangsaan Malaysia in 2006 and PhD in Nano-Material & Device Fabrication from University Sains Malaysia in 2018. Currently, she is working as a senior lecturer at the School of Electrical Engineering, College of Engineering Universiti Teknologi MARA, Cawangan Pulau Pinang, Permatang Pauh Campus, Malaysia since 2006. Her main interests are microelectronics device, device fabrication and characterization, biosensor, memristor and Internet of Things.