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Studies on the Characteristics of Nanostructures Produced by Sparking Discharge Process in the Ambient Atmosphere for Air Filtration Application

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Abstract: Among the various methods for the preparation of nanoparticles, a sparking process at atmospheric pressure is of interest because it is a simple method for producing nanoparticles ranging from a few nanometer-sized particles to agglomerated film structures. In this research, we studied the effects of metal electrode properties on nanoparticle sizes. The experiments were carried out by applying a high voltage to different metal sparking tips. The transfer of energies from positive ions and electron bombardments induced the melting and vaporization of electrode metals. Based on this research, we have developed a model to describe the formation of a nanoparticle film on the substrate, placed under the sparking gap, and the nanostructure produced by metal vapor on the sparking electrodes. The model provides a realistic tool that can be used for the design of a large-scale coating and the application of nanoparticles developed by this process for the filtration of PM2.5 mask fabric by air.

Keywords: sparking process; surface energy; nanoparticle nucleation; vapor deposition

1. Introduction

Nanoscale particles can have structural, thermal, electromagnetic, optical and mechanical properties that are significantly different from those of larger particles [1]. Particle properties are highly size-dependent and can be exploited in a variety of applications. Therefore, the control of nanoparticle size is very important and desirable, but it also represents a challenge. Nanoparticles can be prepared using various methods, such as laser ablation [2], ultrafine bubbles and pulsed ultrasound [3], corona discharge [4] and sparking processes [5–11]. The methods and conditions used to prepare nanoparticles strongly influence their size and shape [4,12]. The sparking process is of interest because it is performed in atmospheric air, is inexpensive and does not require a vacuum system. Moreover, this method is flexible regarding the material used, can be up-scaled, and is environmentally friendly as it does not produce any waste and does not require a chemical precursor. Although there have been several publications describing the production of nanoparticles using the sparking process in atmospheric pressure, the effects of the wire electrode properties on the nanoparticle size and formation pattern of the deposited films

have not been reported previously. During the sparking process, the applied voltage induces high-temperature arcing plasma in the air gap via the field ionization process. Electrons and ions in the plasma bombarded the two tip surfaces, resulting in the vaporization and liquefaction of the metal electrodes. In our previous work [5], the formation of nanoparticles was modeled using the Young–Laplace relation by considering the relative surface energies and different pressures inside and outside the molten layers on the electrode surfaces. For the low-pressure atmosphere, Tabrizi et al. [13] produced gold nanoparticles using the spark discharge at the pressure of 1–2.5 bar. They explained that the electrode materials were evaporated, and the nanoparticles were nucleated and agglomerated from the vapor. In this report, the effects of the electrode properties on the generated nanoparticles from both vaporized and molten metals were described and applied in forms of aerosol for disposable face mask testing of filtration efficiency.

2. Experimental

To study the size of nanoparticles, various metal wires were used as sparking tips. The metals used were zinc ($d = 0.38$ mm), aluminum, silver, gold, nickel, cobalt, titanium, vanadium and molybdenum (purity $\geq 99.5\%$, $d = 0.25$ mm, Advent Research Materials Ltd., Eynsham, Oxford, England). The sparking tips were placed horizontally, 5 mm above the center of the glass substrate, with a gap of 2 mm between them. Sparks were produced by the discharge of a 25 nF capacitor at 10 kV. Before the nanoparticles were formed on a glass slide substrate ($10 \times 10 \times 1$ mm³), the substrate was sonically cleaned in acetone, ethanol and distilled water and dried under nitrogen gas. To eliminate the effect of initial conditions, the tips were sparked 100 times before collected the nanoparticles. The spark was created once in an ambient air at atmospheric pressure to study the deposited particle sizes.

Scheme of sparking discharge apparatus is represented in Figure 1. The Supplementary Information depicts all of the sparking apparatus used in our research; from the first prototype that was used for the fabrication of nanoparticles for carbon nanotube growing, to this study. The principle of the work was described previously [14], in brief, our research technique consisted of a power supply that was connected to a 220 V alternative current source. The power supply unit consisted of an AC/DC converter and a controlled source of voltage and current, which was connected to a 7 KV DC boost step-up power module high-voltage generator, which was in turn connected to a capacitor that directs electrical power to a circuit breaker with changeable heads that hold metal wires. These wires were placed in a sealed container, through which gasses flowed, and in which a substrate for nanoparticle collection was placed.

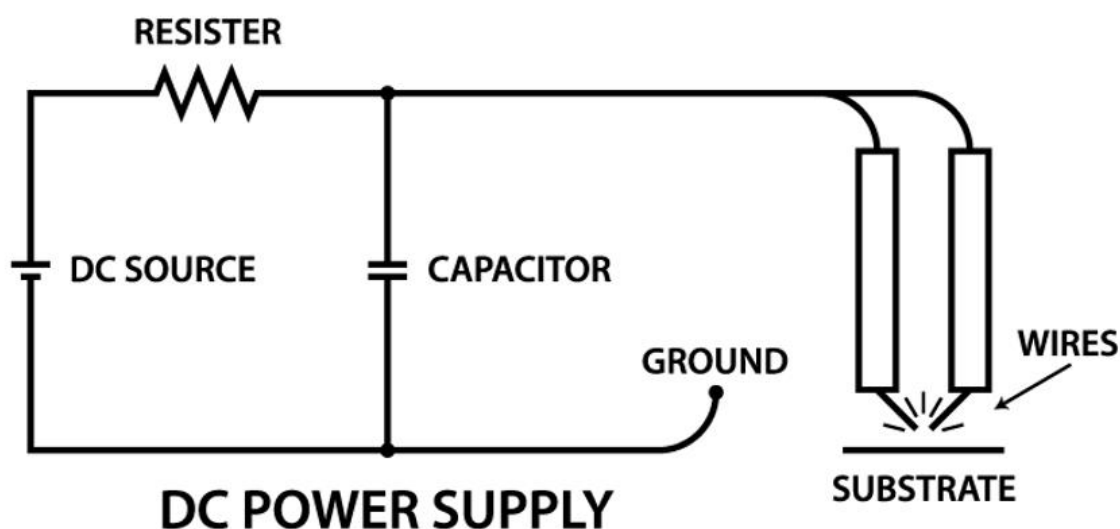


Figure 1. Conceptual representation of sparking machine apparatus. For pictures of the machine please see the Supplementary Information.

To study the particle sputtering patterns resulting from the sparking process (and therefore to study the directions in which the particles were expelled from the electrode wire's melting surface), gold and cobalt wires were used as the sparking electrodes, since they produce sparked nanoparticles of easily observable color. A voltage of 10 kV was applied across a 1 mm gap, causing the spark discharge. White paper substrate was placed 5 mm below the sparking gap and examined after sparking 1000 times.

The sparked electrodes and the deposited nanoparticles were characterized using scanning electron microscopy (SEM, JEOL JSM 6335F, Akishima, Japan). The Raman spectra were obtained with a 514.5 nm argon ion laser at a room temperature (Jobin Yvon Horiba T64000, Chiyoda-ku, Japan) to determine crystallinity of the films.

Surface characterization was done by using an atomic force microscope (AFM) in the tapping mode (Digital Instruments, Inc., Santa Barbara, CA) equipped with a standard Si tip and operated at a scan size of $1 \times 1 \mu\text{m}^2$ in air at room temperature. Section analysis was carried out to estimate sizes of the NDs (nanodots) using the Nanoscope IIIa 5.12 r3 (Veeco, New York, NY, USA) software. The primary particles size can be measured by sparking a very low surface coverage of nanoparticles on a substrate for AFM. After approximately one second of sparking onto the substrate, we noticed films with primary particles as an isolated island, consisting of primary particles that were deposited very far away from the other particles.

3. Results and Discussions

Nanoparticles produced using various sparking metals were imaged using AFM, as shown in Figure 2a. Because the lateral (horizontal) sizes of the nanoparticles estimated by AFM show a broadening effect due to a tip shape convolution [15], the nanoparticle sizes were determined by measuring their vertical heights. Due to the random nature of the nanoparticle agglomeration, some particles attached to others and created a secondary particle deposition on the substrate. To determine the primary size of the particles, only primary particles that appeared in the images were used. It can be clearly seen, that a low energy level is needed to melt the metals (Zn, Ag, Au), resulting in a higher density of the particles in the area under observation. The reason for this is that these metals require a lower energy to melt the electrode, and in turn, to melt a nano-droplet. The energy applied to the tips (E_{app}) is separated to the energy lost to the environment (E_{loss}) and the energy used to melt the metal tips, as described by Equation (1)

$$E_{app} = E_{loss} + m[c_p(T_m - T_r) + L_f] \quad (1)$$

where c_p is the specific heat capacity, m is the effective mass of the metal tip, T_m is the melting point, T_r is the room temperature and L_f is the heat of fusion. As shown in Table 1, the melting point of aluminum (933 K) is lower than that of silver (1235 K) and gold (1337 K), however, there a reduced amount of sparked aluminum observed. This is due to aluminum having a remarkably high heat fusion of $0.90 \text{ Jg}^{-1}\text{K}^{-1}$, whereas the high heat fusions of zinc, silver and gold are in the range of $0.13\text{--}0.39 \text{ Jg}^{-1}\text{K}^{-1}$. Moreover, aluminum is always covered with a microscopic thin layer of Al_2O_3 that is extremely hard and inert. Therefore, this oxide also reduces the creation of nanoparticles during the discharge. Figure 2b shows the height measurement of zinc nanoparticle using section analysis of Nanoscope III (Veeco, New York, NY, USA) software.

The relationship between the nanoparticle heights and the surface energy at melting point of the electrode metals is shown in Figure 3. It can be clearly seen, that the particle heights decreased as surface energy increased. Nevertheless, the heights of all primary particles were less than 4.5 nm, which corresponds with the transmission electron microscopy (TEM) images of sparked nanoparticles published elsewhere [10]. The relationship between the height of the particles and the surface energy is given by a linear formula $H = 1.6915 \gamma + 4.4783$, where H is the average particle height (nm) and γ is the

surface energy ($\text{J}\cdot\text{m}^{-2}$). This result strongly confirms that nanoparticles on the substrates were nucleated by the molten metal electrodes.

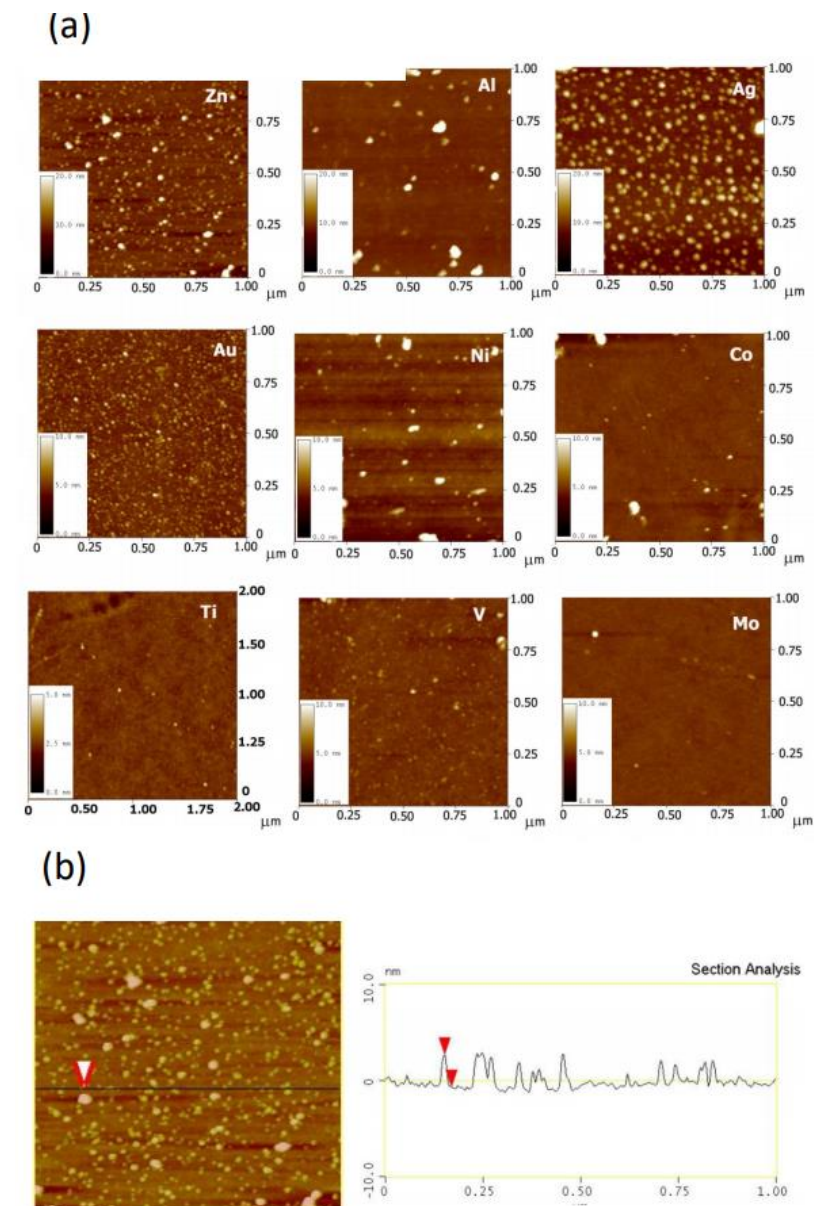


Figure 2. (a) Atomic force microscope (AFM) images of various metal nanoparticles prepared by single spark on glass substrates (b) Section analysis to measured height of zinc nanoparticles.

Table 1. Metal properties and experimental data [16].

Metals.	T_m (K)	σ_{LV} (Jm^{-1})	c_p ($\text{Jg}^{-1}\text{K}^{-1}$)	k ($\text{Wcm}^{-1}\text{K}^{-1}$)	L_f (Jg^{-1})	L_v (Jg^{-1})	Height (nm)
Zn	693	0.789	0.39	1.16	244	3,843	3.30
Al	933	0.871	0.9	2.37	830	22,569	3.15
Ag	1235	0.925	0.24	4.29	240	5,319	2.71
Au	1337	1.145	0.13	3.17	158	4,233	2.63
Ni	1718	1.796	0.44	0.91	623	13,228	2.02
Co	1811	1.881	0.42	1.00	599	13,944	1.50
Ti	1941	1.525	0.52	0.22	702	19,136	1.23
V	2183	1.855	0.49	0.31	908	19,652	1.08
Mo	2896	2.080	0.25	1.38	761	14,238	0.92

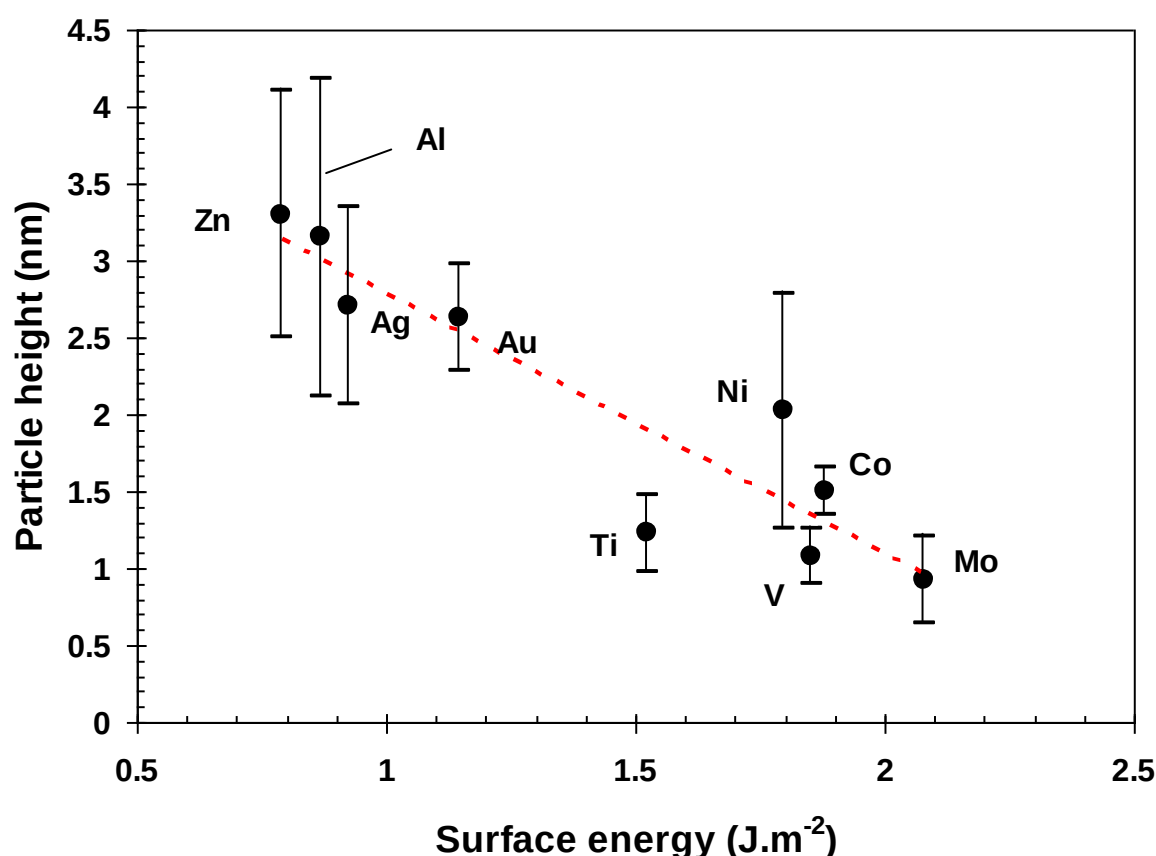


Figure 3. Plot of the height of sparked nanoparticles as a function of surface energy at melting temperature.

Figure 4a,d show sparked particle films of gold and cobalt, respectively, on white paper substrates. The film formed in two semicircle shapes with a gap of 1 mm between them. The semicircle on the right-hand side was formed under the cathode and is larger than the semicircle on the left that formed under the anode. This is caused by the process when, during the sparking, atmospheric gas breaks down and creates the positive ions and free electrons. The bombardment of the anode by the electrons and the cathode by the positive ions melts the electrode surfaces. The thermal energy expands, and a shock wave forms. The resulting high air pressure pushes the melted metal layer away from the electrode gap, and the molten metal therefore splashes away from the gap in a cone-shaped pattern, while the nanoparticles are nucleated. The cone shaped splashing produces the two semicircles on the substrate. The black color of the sparked cobalt film in Figure 4a indicates the presence of cobalt oxide. Furthermore, the sparked gold film is pink, which is a feature of gold nanoparticles [17].

Figure 4b,c show the cobalt anode and cathode electrode tips, respectively, after the sparking process. The erosion of the tip results in the round shape at both tip ends. The results are not the same for the gold anode and cathode tips, as shown in Figure 4e,f, respectively. The sparked gold tips are cone shaped. The cone angle of the anode tip is less than that of the cathode tip. The reduced cone angle of the anode tip corresponds with the smaller area of the particle film produced under the anode (Figure 4d). The shape of the anode is caused by the expansion of the plasma diameter at the anode surface [18]. In addition, the sides of the cobalt tips were covered by nanoporous films. It is believed that some of the metal evaporated as a gas phase and then deposited on the tip surfaces. The sides of the gold electrodes are different from those of the cobalt tips and no deposited nanoporous film was observed because the gold vapor could not oxidize in this setup.

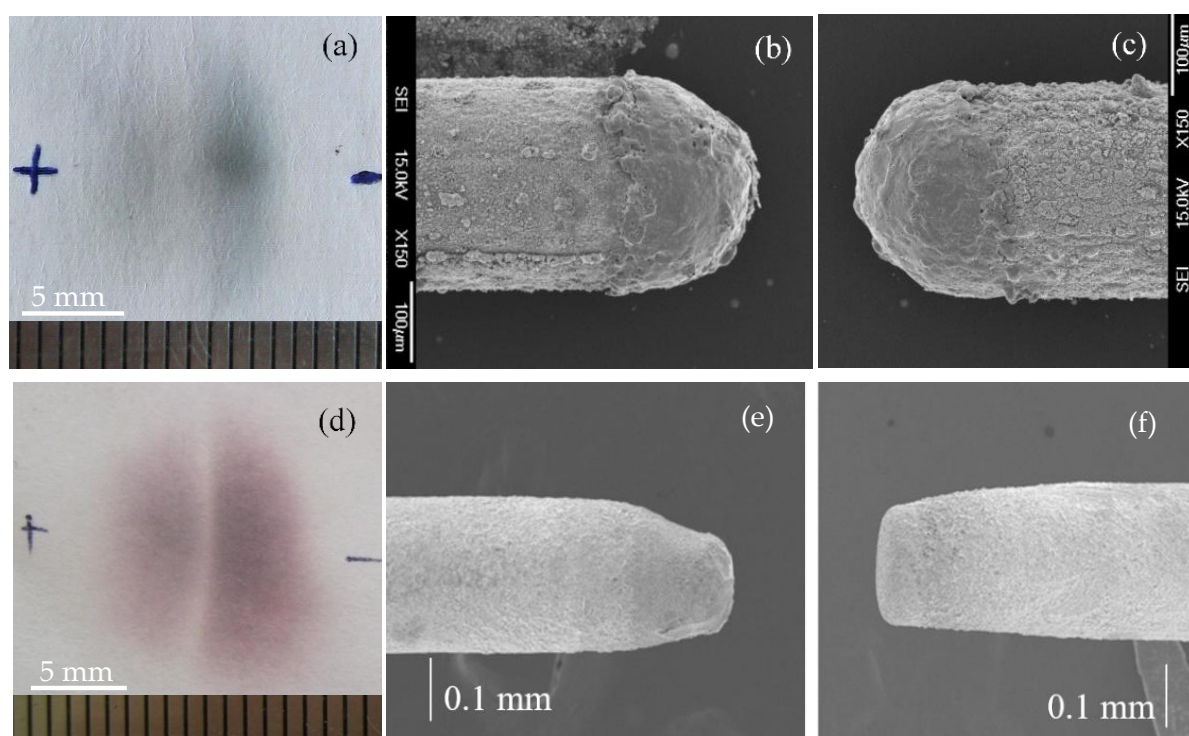


Figure 4. (a,d) Nanoparticle films on papers which were placed under the sparking gap of cobalt and gold, respectively. The electrode after 1000 cycles of spark: (b) Co-anode (c) Co-cathode (e) Au-anode and (f) Au-cathode.

More studies are needed in order to better understand the formation of particle coating on the electrode wires. In this study, titanium wires were used as sparked electrodes. The anode and cathode titanium wires were sparked 1000 times. The sparked electrode wires are shown in Figure 5a,b for the anode and cathode, respectively. In the pictures, the nanostructure films are seen to almost fully cover the anode surface. On the other hand, most of the surface area of the cathode is covered by the freeze splashing of molten metal droplets. As previously explained, positive ions have a larger mass and transfer more energy to the cathode compared to the energy transferred by electrons to the anode. Therefore, the cathode shows more surface damage than the anode. The molten electrode splashed and then froze very quickly due to the rapid cooling of the room atmosphere. Furthermore, the nanoporous film coverage observed on the tip is rather patchy. In addition, during the sparking, the glass substrate was placed 2 mm under the sparking gap to collect the nanoparticles from the metal droplets (the result shown in Figure 7).

Figure 5 shows high magnification SEM images displaying the tip surface located 200 μm , 400 μm and 600 μm from the tip end (the positions of the tips are shown in Figure 4). Nanostructures grown on the anode tip (Figure 5a,c,e for the position of 200 μm , 400 μm and 600 μm , respectively) are sponge-like structures composed of numerous irregular-shaped and randomly-oriented grains. It is believed that, the nanostructures are the deposition of titanium vapor. The difference in the size and shape of the nanostructures is explained by the influence of different substrate temperature [19,20]. On the cathode tip (Figure 5b,d,f for the position of 200 μm , 400 μm and 600 μm , respectively) no nanostructures can be observed, since the splashing of the melting titanium prevents the deposition of titanium vapor on the cathode tip surface. The results are the opposite for the cobalt, where the cathode tip is fully covered in cobalt oxide nanostructures, as shown in Figure 4c. Therefore, based on the results for gold, cobalt and titanium, it can be concluded that the nanoparticles were nucleated by the droplets of molten metals and deposited on the substrates under the sparking gap for all sparking metal electrodes. However, the nanostructure nucleation caused by the deposition of metal vapor on the sparking tip depends on the properties of each metal.

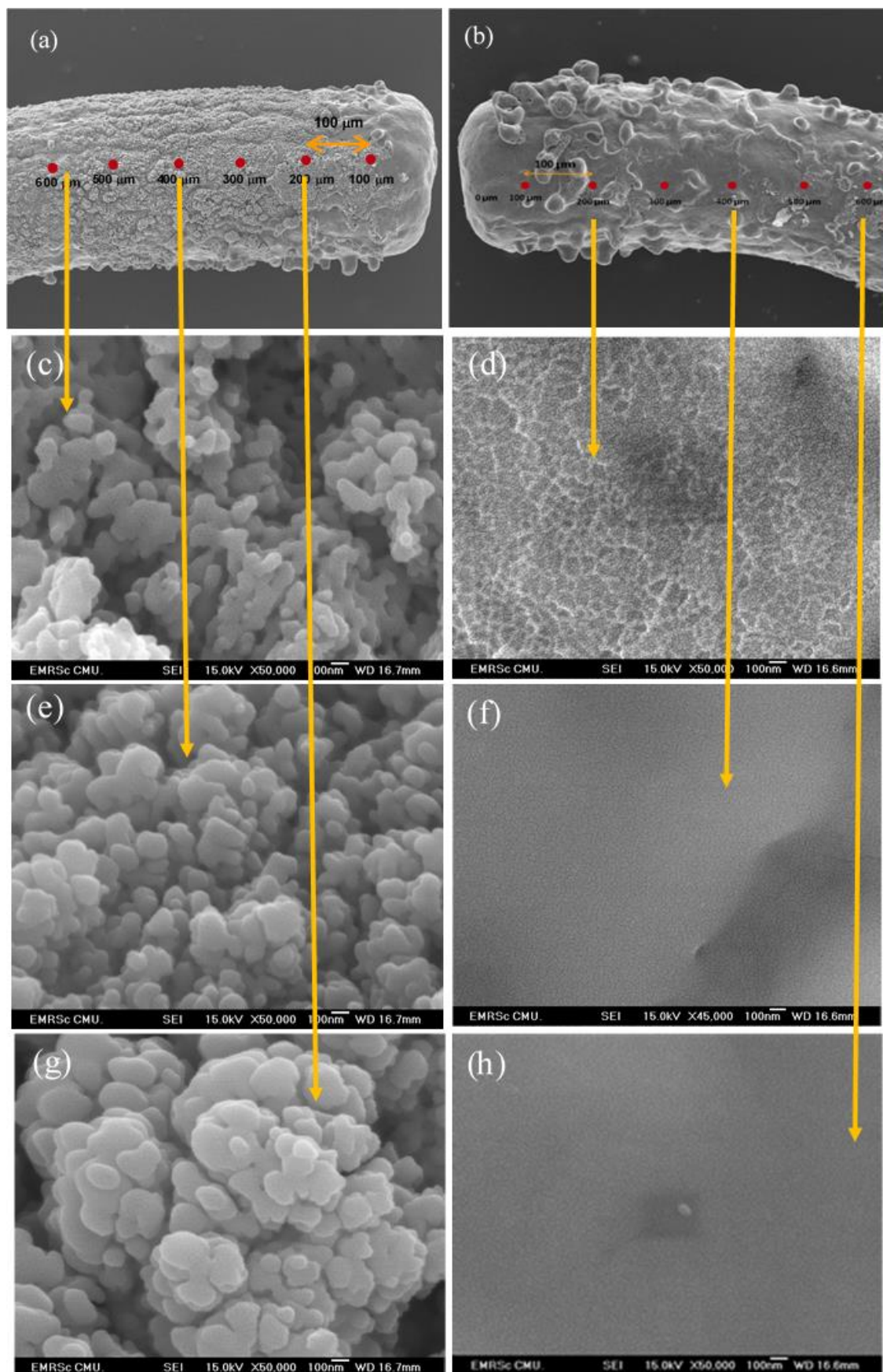


Figure 5. (a) Anode-Ti (b) Cathode-Ti after 1000 cycles of spark and surface structures on the sparked tips at the position of 200 μm (c,d), 400 μm (e,f) and 600 μm (g,h) from the tip ends for anode and cathode titanium tips, respectively.

Figure 6a,b illustrate the SEM images and EDS line scan result of the sparked anode and cathode titanium tips, respectively. At the tip of the anode electrode, a large titanium ratio and a small amount of oxide cover were found. Given that there was no oxygen found at the aperture created by the focus ion beam (FIB), we can conclude that the oxygen was only on the surface. The oxygen content increased in the nanoporous film area due to the formation of titanium oxide in that area. As was the case with the anode, the oxygen content also increased in the cluster of nanoporous material on the cathode.

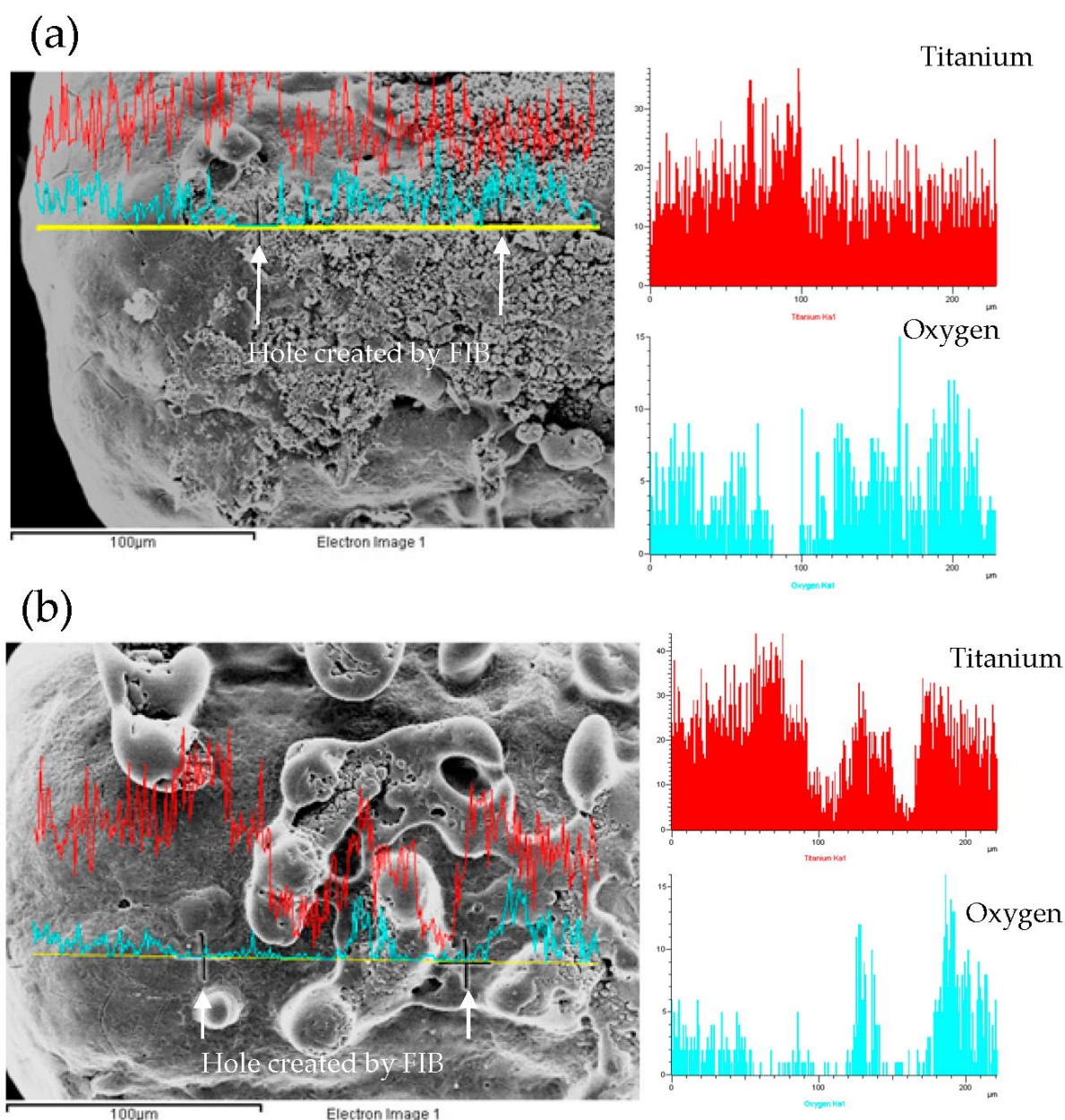


Figure 6. SEM–EDX line scans of sparked titanium tip (a) anode (b) cathode.

Figure 7 shows the SEM image of the sparked titanium particles that were deposited on the glass substrate, 5 mm under the sparking gap of titanium tips presented in Figure 5. The films were an agglomeration of nanoparticles with an approximate size of 20 nm. The size of the nanoparticles was almost ten times smaller than the size of the grain that occurred due to the vapor deposition on the tips electrodes. The crystallinity of the sparked films was initially amorphous and then gradually transitioned into the anatase phase of TiO_2 during the annealing at 400 °C for 1–5 h, as shown in Raman spectra in Figure 8.

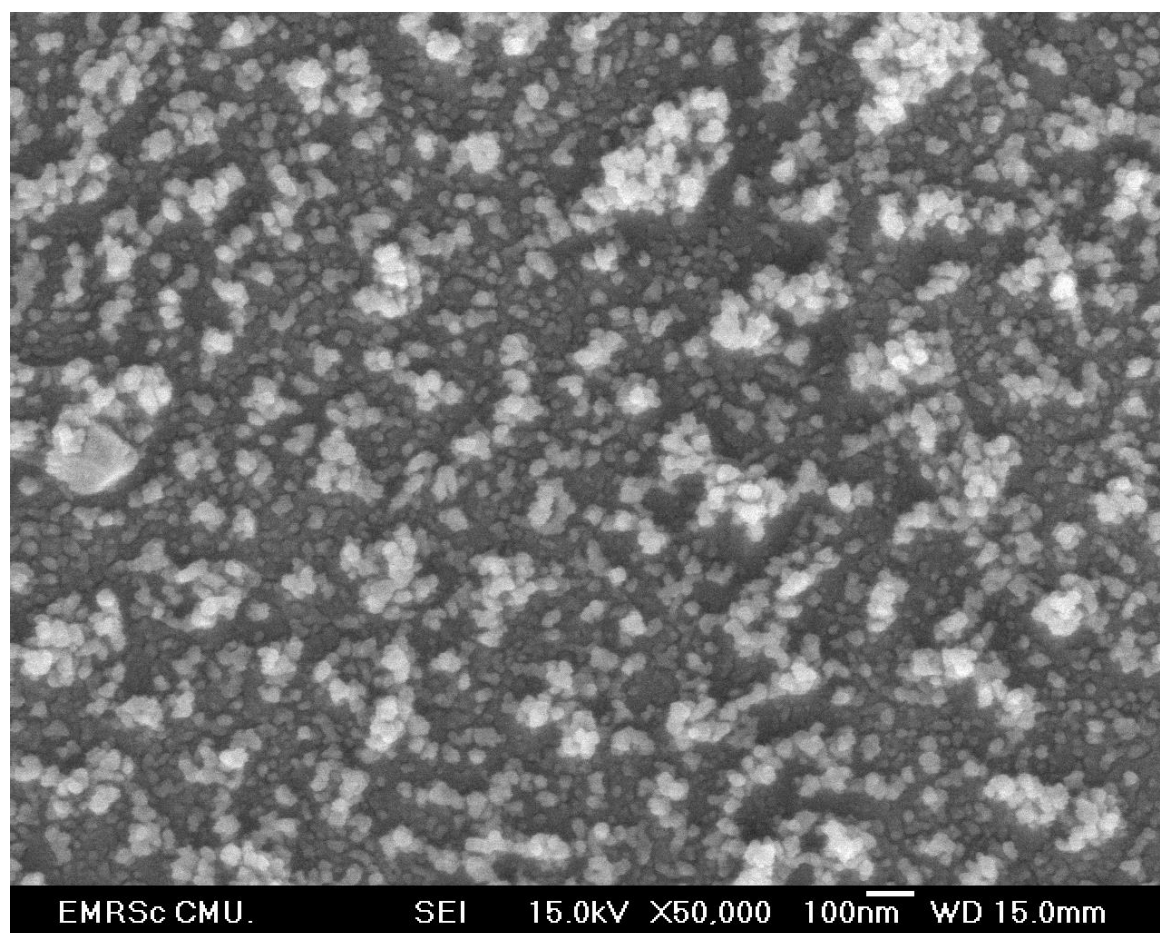


Figure 7. Scanning electron microscopy (SEM) of sparked titanium nanoparticles.

Unless wires are sparked inside of a magnetic field [21], in ambient conditions at atmospheric pressure, nanoparticles collected on the substrate during sparking discharge process will be amorphous. High energy will also break down gas molecules in the air between the sparking gap (even nitrogen) and will create different oxides, nitrides and carbonates [22]. Because of obtaining thermodynamically unstable products, annealing in controlled atmosphere will affect the crystallinity and phase of the sparked nanoparticles [23]. This is the reason why we see different phases in the Raman results of the sparked titanium.

To further explain the action of the sparking process in the air pressure atmosphere, the schematic illustrations of the nucleation of nanostructures were developed and are shown in Figure 9. When the voltage applied to the gaps is higher than the break down voltage, gas molecules in the sparking gap were ionized, and the electrons and ions produced from the neutral molecules migrated towards the anode and cathode, respectively. The bombardment of high energy electrons and ions melted and evaporated the metal tips. The expansion of the electron stream at the anode increased the bombardment area and therefore the sparked anode tips exhibited a low cone angle. For the cathode, positive ions have a larger mass and therefore transfer more energy to the cathode, resulting in a larger melting volume than the anodes. The nucleated molten nano-droplets were blown in the cone shape around the tip and deposited on the substrate which were placed under, next to, and over the gap. The reduced density and hemisphere area of the films under the tips of the anode are consequences of the reduced cone angle and lower molten volume. The growth of the nanoporous structures from the metal vapor on the sparking tips depends on the material and thermal properties and surface reactivity of the metal tips.

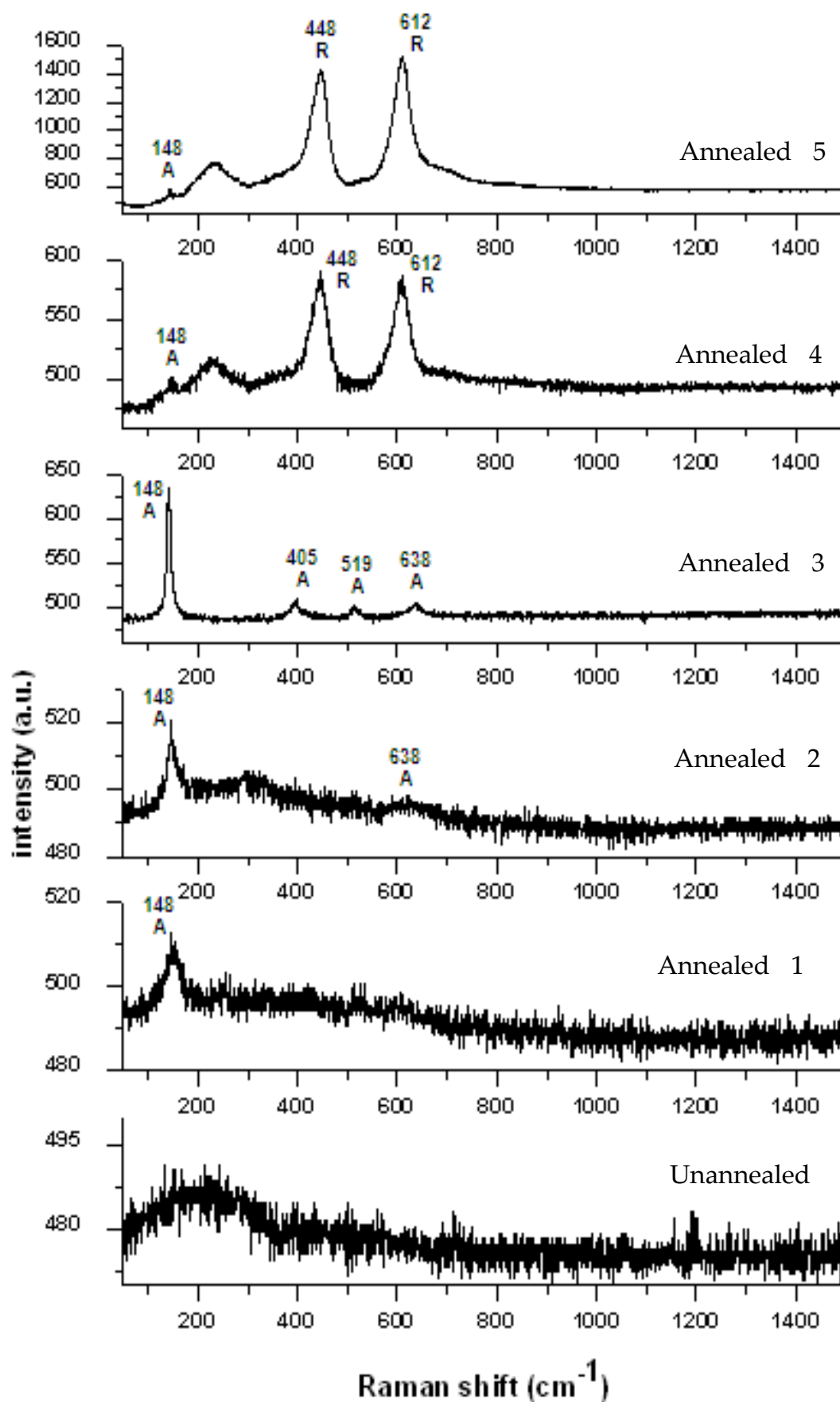


Figure 8. Raman spectra of the sparked titanium nanoparticles films annealed at different temperatures.

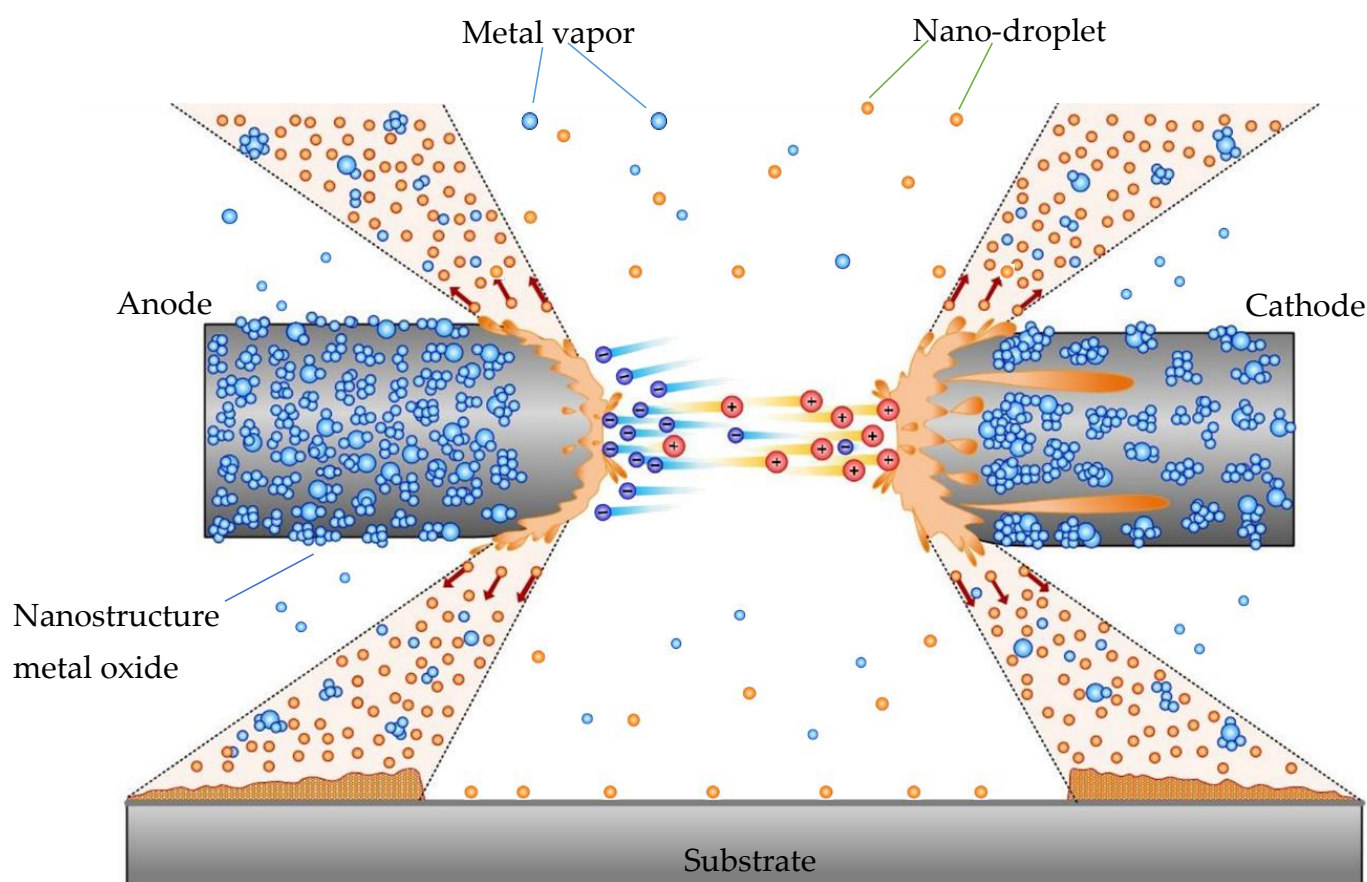


Figure 9. Schematic diagram of the nucleation mechanism of the nanostructure deposited by the sparking method.

There are a substantial number of applications involving the sparking discharge process in the field of aerosol science [24]. Even though we provide evidence in this research that most of the nanoparticles are made by the metal melt approach, the sparking discharge process can still be used for aerosol production and fabric filtration calibration applications, which are now more important than ever in light of the current COVID-19 crisis [25].

In Figure 10, the results with the filtration barrier and without the filtration barrier are both presented. The experiment was conducted as previously described, [26], using aluminum wires, with the modification of one output, in which a commercially available surgical mask as a filter is attached onto the aerosol outlet, leading to a differential mobility analyzer (DMA). The sparking discharge process suitability for the aerosol calibration was compared with the dispersing particle size standards™ (from Polysciences, Inc. Nanobead NIST Traceable Particle Size Standards). The results indicate that filtration capacity of the sparking discharge is 99.6%; obtained using the following equation.

$$E_f = 100 - \frac{\text{number of particles measured with DMA with filter}}{\text{number of particles measured with DMA without filter}} \quad (2)$$

This was compared by using a 50 nm particle size standard and the results obtained are not significantly different. A 50 nm particle size was used because this was the size that was generated from the sparking aluminum wires with the machine described in our experiment.

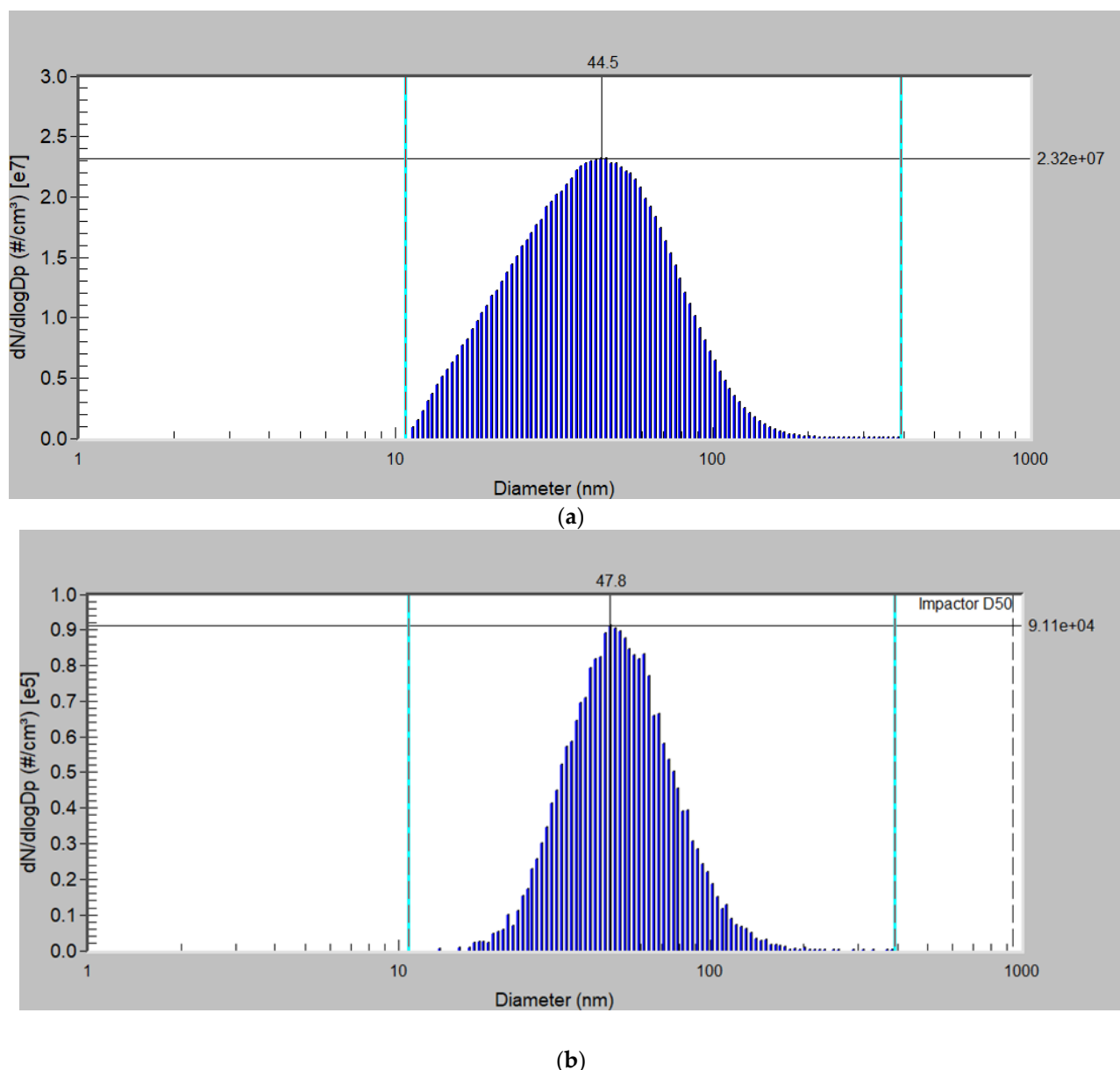


Figure 10. Filtration experiment measured by the differential mobility analyzer. (a) The aluminum sparked wire without a surgical mask as a filter. The concentration of nanoparticles produced by sparking was 2.32×10^7 particles at 1 cm³ at size of 44.5 nm (b) Results of differential mobility analyzer (DMA) from aluminum sparked wire with a filter from a commercially available surgical mask. Concentration of nanoparticle produced by sparking was 9.11×10^4 particles at 1 cm³ at size of 47.8 nm.

4. Conclusions

There are two approaches that are utilized in the generation of nanoparticles from a sparking discharge process. The first is an aerosol-based process [27], and the second is a metal melt process, that we used to modify the surface and create nanomaterials [28]. The sparking discharge studies by Kohut et al. [29] describe craters, undulated areas, and dendritic areas of nickel and copper electrodes (rode) in low numbers (one to three) as opposed to our study (100). Their results also support our approach to sparking discharge for nanoparticle generation through metal melt, with only a small portion of the electrode content being aerosolized for nanoparticle synthesis. This confirms the production of nanoparticles by the sparking discharge methodology and spark discharge apparatus as mainly coming from the metal melt process.

The findings by Domaschke et al. [30] are significant because they support our exclusive use of wires as the electrode material. In their work regarding the sparking discharge process, a high capacitance of the apparatus is also expected in order to improve the energy efficiency of the sparking discharge process. They also measured the effect of the gas flow during sparking discharge on particle size and the concentration of the produced particles, which we did not deal with because we did not use the gas flow during the synthesis of nanoparticles. Our sparking discharge process was done in an ambient atmosphere and under normal pressure conditions, while metal wires were discharged with no air flow.

In summary, we have demonstrated the preparation of nanoparticles using the sparking process of various metal tips at an atmospheric air pressure. It was observed that particle sizes are related to the surface tension of molten metals. The film coated under the sparking gap was produced by molten metal droplets, whereas the nanostructure covered on the sparking tips was formed by the deposition of metal vapors. The model was created to explain the coating pattern, and this model provides a practical tool that can be used to design a sparking machine for large scale application (see Supplementary Information for examples). This sparking discharge machine can be used for the calibration of fabric filtration barriers as a replacement for dispersing particle size standards.

Supplementary Materials: The following are available online at <https://www.mdpi.com/2073-4352/11/2/140/s1>.

Author Contributions: T.K. wrote, analyzed, and collected the data, S.R., wrote, collected and analyzed the data, and edited the manuscript E.K., collected the data, N.J., collected and analyzed the data, W.S. collected the data, W.T., collected the data. S.P. collected the data, A.P. collected the data, P.S. (Panupong Sanmuangmoon) collected the data, P.S. (Pisith Singjai) supervised the edit and acquire funding. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

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