VOL. 14, NO. 1, JANUARY 2019 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

THE EFFECT OF COPPER ELECTRODE AND HSS TYPE ELECTRODE ON DC THERMAL PLASMA METHODE ON THE CHARACTERISTICS OF THE NANOPARTICLE Al_2O_3

Salahuddin Junus, Sumarji, Haidzar and Robertus Sidartawan
Department of Mechanical Engineering, Faculty of Engineering, Jember University, Kampus University, Jember, Kalimantan, Jawa Timur, Indonesia University, Indonesia
E-Mail: salahuddin.teknik@unej.ac.id

ABSTRACT

The manufacturing of Al_2O_3 nanoparticle using a DC plasma methode. The materials used are aluminum powder with dimensions of 88 µm. The electrode used are copper and HSS. The nano-powder Al_2O_3 obtained are tested using a Scanning Electron Microscopy (SEM), energy dispersive X-ray (EDX) and X-ray-diffraction (XRD). The results of the test of alumina nano-particles showed that the copper electrode has a spherical nano-particle and there is agglomeration with a size of 24.08 nm. With a purity of 96.68%. The HSS electrode produced nano-particles as 20.8nm in size, with a purity of 88.68%.

Keywords: Al_2O_3 Nanoparticle, DC thermal plasma, copper electrode, HSS electrode, SEM, EDX, XRD.

1. INTRODUCTION

Alumina or aluminum oxide (Al_2O_3) is a ceramic material that has many applications in engineering such as using its corrosion protection, hardness and high temperature tolerance properties [1]. Alumina has been often produced in nanoscale of 1-100nm. Nano-Alumina's application has been used in coating, catalyst, single crystal sapphire and composites [1, 2, 3, 4].

Nanoparticles could be made using a variety of methods including DC plasma. This method is used due to its high efficiency and could be controlled using filler particles [2]. This method has been used extensively and has produced many materials of metal, ceramic and polymer [3, 4].

Various electrode materials are used to minimalize production cost and for the durability of the electrode itself. An expensive electrode does not necessarily suggest that is effective [5]. The DC thermal plasma machine operates at very high temperatures to evaporate the micro particles to nanoparticles [6], where not only the working materials evaporates but also the electrode [5]. This indicates that the evaporating point and oxidation of the electrode material affects the purity of the produced nanoparticle.

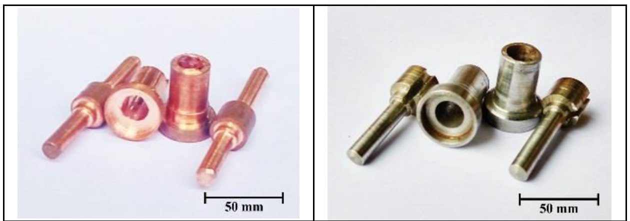
The purity of the nanoparticles that are produced by the DC method depends on the particle composition and the electrodes used [7]. Until now there are several materials that are used for electrodes i.e. graphite, silver, tungsten and copper [2].

Copper has a good electrical and thermal conductivity therefore it could create excellent sparks. Copper has a melting point of 1083^0 C and boiling point of 2600^0 C. Oxidation of the surface occurs at $187\text{-}377^0$ C by CuO (black) and changes at $T>797^0$ C CuO (brownish red) and at last melting point 1227^0 C, [8]. HSS meanwhile, has a higher melting point of 1200^0C- 1400^0 C and boiling point of 2750^0 C [9].

This research conducts production of nano-alumina using two types of different electrodes (copper and HSS). They are used to investigate the different characteristics of nano-alumina produced.
The characterization is used by SEM-EDX to understand the morphology and content of the produced material. XRD is used to understand the phases formed.

2. MATERIALS AND METHODS

Figure-1 shows the electrodes used in the DC Thermal Plasma device made from copper and HSS.



(a)	(b)

Figure-1. Types of electrodes used (a) Copper electrode; (b) HSS Electrode.

Figure-2 shows the scheme on the production of nanoparticles. Alumina particles are obtained from aluminum powder 88 µm in size. It is produced by MERCK (catalog number 1.01056.0250) with a purity of aluminum powder of 97.1%. DC plasma reactor is made from stainless steel with a water coolant system in a vertical position.

The electrode material used for anode and cathodes are the same i.e. copper and HSS. The current is 20 amperes with oxygen as the gas carrier 16.51 1/min. Aluminum powder passes the plasma in 6g/min. When the powder passes the plasma, it will break and evaporate to small cores and oxidizes into alumina (Al_2O_3).

X-Ray Diffraction (XRD) is used to understand the formed alumina phases. XRD is of PANalytical, X'Pert Pro with Cu radiation. To determine resulting size of the particles, the morphology and the elements, SEM

ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

(scanning electron microscopy) is used. SEM is of FEI, Inspect S50 with a voltage of 25kV. Energy dispersive X-ray (EDX) is used to determine the resulting element composition.

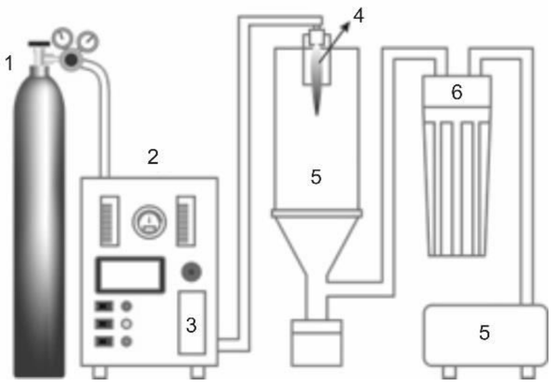


Figure-2. Schematic experimental set-up: (1) Oxygen; (2) Plasma power source; (3) Powder feeder; (4) Plasma torch; (5) Plasma reactor; (6) Powder Collector Chamber; (6) Vacuum.

3. RESULT AND DISCUSSIONS

Figure-3 (a, b, c) Shows the differences in color of the resulting alumina particles. From the differences it could be determined the whiter the color, the purer the alumina. The differences are due to impurities in the composition of alumina nanoparticles.

(a) (b) (c)

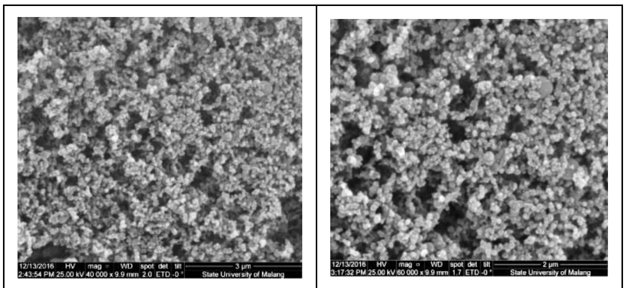
Figure-3. (a) Aluminum powder; (b) Alumina Nanoparticle with copper electrode; (c) Alumina nanoparticles with HSS electrode.

On the alumina with copper electrode, it has Al_2O_3 components as much as 96.68% with main impurities Cu) as much as 3.32% where it has a black primary color [8]. This causes the sample color to be white with a blackish color as shown in figure-3(b). The aluminaparticles with the HSS electrode has components of Al_2O_3 as much as 88.68% with impurities of Fe_2O_3 of 11.32%. Fe_2O_3 has a brown primary color, making the nanoparticle sample having a white brownish color as seen in Figure-3(c). Fe_2O_3 is formed by the oxidation on the HSS with the main component of Fe 87.66%.

3.1. Scanning Electron Microscopy (SEM) analysis

a) Alumina nanoparticle SEM results with copper electrode

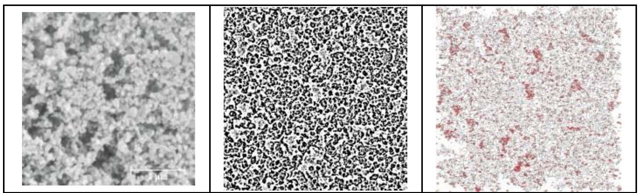
Particles show that it has a circular shape and it also has some agglomeration with diameters of 24.08 nm in average. The distribution of alumina particles under 100 nm reaches 93.5% while the sizes above 100nm is 6.5% as shown in Figure-4. The larger sized particles are formed due to passing the plasma and splits or evaporates imperfectly causing the core grain droplets are still large and starts cooling in that state. [10].



(a) (b)

Figure-4. SEM results of alumina particles with copper electrodes (a) magnification 40.000x; (b) magnification 60.000x

The results of the SEM on the alumina particles uses a magnitude of 60.000x as shown in Figure-4. The nanoparticles are characterized using *Image J* shown in Figure-5 below:



(a) (b) (c)

Figure-5. Processing the resulting images of the alumina particle SEM of copper electrodes using Image J (a) alumina nanoparticle; (b) Thresholding; (c) Outlines.

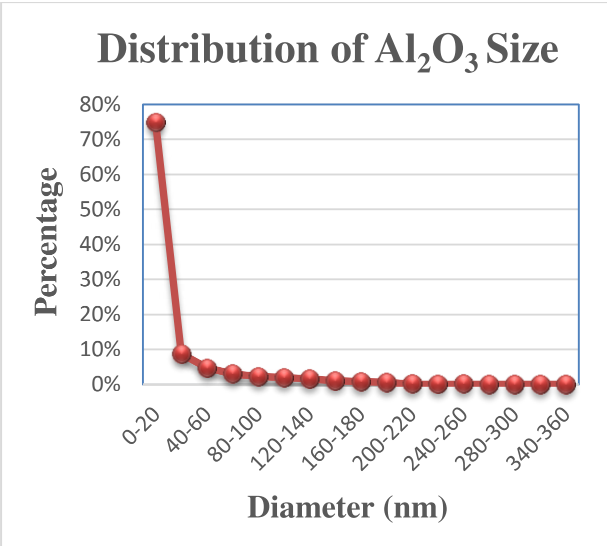


Figure-6. Distribution of alumina nanoparticle size on copper electrodes.

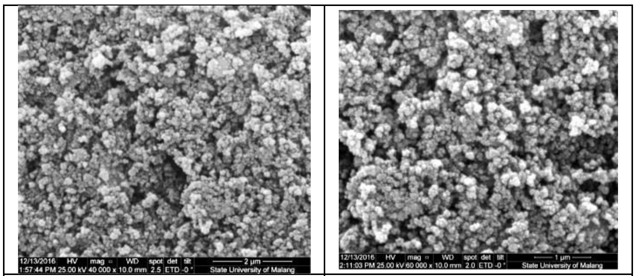
ARPN Journal of Engineering and Applied Sciences

From the results of thresholding and outlines (Figure-5) it is observed an area average of 1699, 1 nm^2. The data is then inputted in the equation (1) where we obtain an average sample 2 nanoparticle diameter of 24, 08 nm.

Figure-6 shows the distribution of nanoparticle size with copper electrodes. Alumina particle size under100nm reaches 93.5% while the remaining 6.5% are a above 100nm which are not considered nanoparticles.

b) SEM results on alumina nanoparticles on HSS electrodes

The following shows the results of SEM on alumina nanoparticles fabricated using DC thermal plasma method.



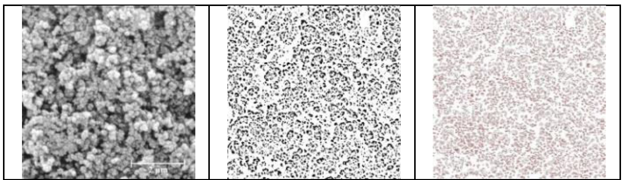
(a)	(b)

Figure-7. SEM results of alumina particles with HSS electrodes (a) magnification 40.000x; (b) magnification 60.000x.

The result of the SEM on the alumina particles uses a magnitude of 60.000x as shown in Figure-7.

From the process of thresholding in Figure-8 we could determine area average by using the analyze particle feature in the Image-J software where it resulted in a particle area average of 545.49nm^2. This data is then used to find the average diameter of the nanoparticles resulting in alumina particle size of 20.9nm.

The nanoparticles are characterized using *Image J* shown in Figure-8 below:



a	b	c

Figure-8. Processing the resulting images of the alumina particle SEM of HSS electrodes using Image J (a) alumina nanoparticle; (b) Thresholding; (c) Outlines.

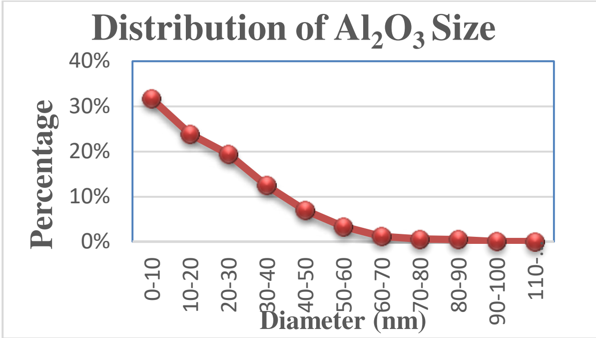


Figure-9. Distribution of alumina nanoparticle size on HSS electrodes.

The Figure-9 is a size distribution graph of the alumina particles on HSS electrodes. It has 99.97% of the particles sizing less than 100nm. It shows that nearly all nanoparticles passing the plasma breaks down to nanoparticle sizes. The remaining sizes above 100 nmis only 0.03%.

3.2 Energy Dispersive X-ray (EDX) analysis

The indication of the elements of Al, O and Cu as an impurity is shown in Figure-10. O, in the spectrum shows the oxidized particles. The oxidized aluminum forming the alumina is confirmed in the XRD analysis. Impurities of Cu in the spectrum could be due to the erosion and oxidization of the Cu electrodes during the manufacturing of the alumina. On the copper electrodes, 96.68% are Al_2O_3 while the rest are main impurities of CuO, colored mainly black resulting in white with blackish color in the sample.

The CuO forms due to the oxidation reaction on the 97.06% copper contents in the temperature of 377ºC - 1097ºC [11]. The plasma electrode forms plasma with oxygen as a carrier gas causing the electrode to oxidize.

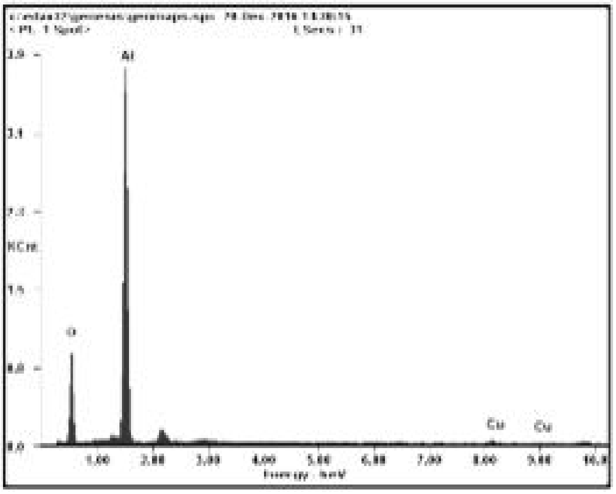


Figure-10. Results of energy dispersive X-ray (EDX) on alumina nanoparticles.

3.3. X-Ray Diffraction (XRD) analysis

The results of XRD alumina data processing resulted in findings dominantly in the *space group* p-4m2 which has a δ phase [11]. The alumina of this phase is formed in temperatures 800º-900ºC [12].

A. Alumina on HSS electrode

The processing of the XRD HSS electrode nanoparticles uses the software MDI Jade to understand the alumina phases formed.

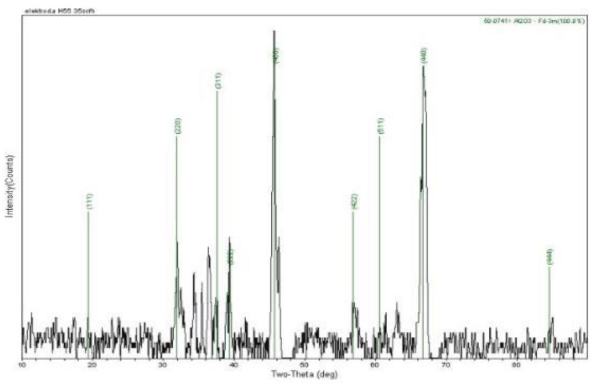


Figure-11. Results using XRD on HSS electrode nanoparticle.

The data was processed using the software MDI Jade6. From Figure-11, the alumina which are dominant are categorized into space group Fd-3m which are alumina in the phase of γ, [13]. According to [12], alumina phase γ forms in temperatures of 650-750^{o} C.

Figure-12 shows the relationship of XRD on Al_2O_3 on copper electrodes, copper and HSS where the differences on peaks of XRD diffraction shows the difference in intensity of crystal phase [13].

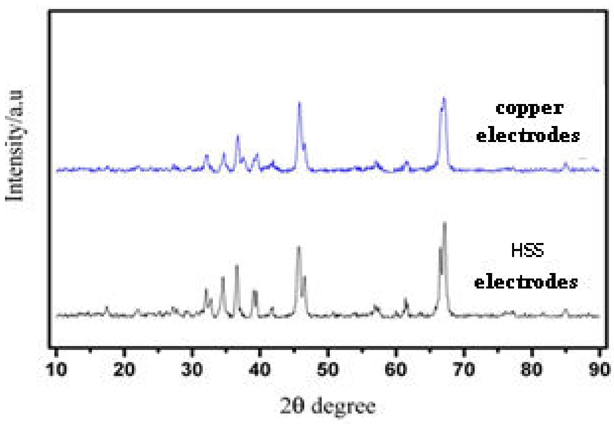


Figure-12. Relationship between XRD spectrums Al_2O_3, copper electrodes and HSS electrodes.

4. CONCLUSIONS

From this research it could be concluded:

a) From SEM results it is shown that the alumina average particle size varies in size. 25.54 nm for graphite electrodes, 24.08 nm for copper electrodes and 20.9 nm on HSS electrodes.
b) EDX purity testing shows a variation on alumina nanoparticles where copper electrode of 96.68% and HSS electrode of 88.68% %. The best purity shown by copper electrode and the least on HSS for this case.
c) From the XRD test on Alumina particles, it is shown that the Copper electrode shows that it has a variation of δ phase and the variation of HSS has a γ phase.

ACKNOWLEDGEMENTS

This work is funded by the Directorate for Research and Community Service-Directorate General of Research and Development Strengthening-Ministry of Research, Technology and Higher Education of the Republic of Indonesia. This publication is found by University of Jember.

REFERENCES

[1] Chang, H. dan Chang, Y. C. 2007. Fabrication of Al_2O_3 Nanofluid by a Plasma Arc Nanoparticles Synthesis System. Taipe: Department of Mechanical Engineering, National Taipei University of Technology.

[2] Watanabe T., dan Tanaka M. 2009. Thermal Plasma Processing for Functional Nanoparticle Synthesis. Yokohama: Tokyo Institute of Technology, Japan.

[3] Shinde et al. 2008. Uncapped silver nanoparticles synthesized by DC arc thermal plasma technique for conductor paste formulation. Panchwati, India: Centre for Materials for Electronics Technology (C-MET).

[4] Mohsenian, Esmaili, Shokri, dan Ghorbanalilu. 2009. Physical characteristics of twin DC thermal plasma torch applied to polymer waste treatment. Tehran: Shahid Beheshti University, Iran.

[5] Khan A., A. 2007. Electrode wear and material removal rate during EDM of aluminum and mild steel using copper and brass electrodes. Department of Manufacturing and Materials Engineering, International Islamic University Malaysia, Kuala Lumpur, Malaysia.

[6] P. Kong dan A. Kawczak. Plasma Synthesis of Nanoparticles for Nanocomposite Energy Applications. 2008. Idaho National Laboratory, US. Department of Energy.

[7] Prabandono, Kurniawan, Suyitno, dan Ubaidillah. 2012. Mesin DC Thermal Plasma Sederhana Menghasilkan Material ZnOdengan Kemurnian yang

Tinggi. Jurnal Politeknosains Universitas Sebelas Maret. 11(2).

[8] Zhukov, M.F. dan Zasypkin, I.M. 2007. Thermal Plasma Torches: Design, Characteristics, Application. Cambridge: Cambridge International Science Publishing Ltd. halaman 463.

[9] OSG, Corp. 2010. Material Safety Data Sheet (MSDS). Toyokawa, Japan.

[10] B. Shokri, S. Kh. Alavi, M. Mirzaie and H. Ghomi. 2009. The size control of Al nanoparticles synthesized by DC thermal plasma. Physics Department and Laser-Plasma Research Institute of Shahid Beheshti University, Evin, Tehran, Iran.

[11] M. F. Zukov, I. M. Zasypkin, A. N. Timoshevskii, B. I. Mikhailovdan G. A. Desyatkov. 2007. Thermal Plasma Torches. Cambridge, UK: Cambridge International Science Publishing Ltd. halaman 463.

[12] J. Metson. Production of alumina. 2011. The University of Auckland, New Zealand: Woodhead Publishing Limited.

[13] Gangwar, Gupta, Tripathi, dan Srivastava. 2012. Nanoscale: Phase dependent thermal and spectroscopic responses of different morphogenesis of Al_2O_3 nanostructures. The Royal Society of Chemistry.