

Preparation of Aluminium Alloy Metal Nanoparticles by Liquid Pulsed-Laser Ablation

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Abstract

Background/Objectives: Nanotechnology has been stated in Malaysia New Economic Model and widely used in much application. The article focuses on nanoparticle formation using liquid pulsed-laser ablation technique. **Methods:** There are two methods have been used to produce the metal nanoparticles namely breakdown and build up method. In this article, the pulse laser ablation (build up method) has been used to generate the metal nanoparticles whereby the metal target is immersed in ultra-pure water. The Al alloy metal nanoparticles were studied using two laser parameters namely laser exposure time and laser power. **Findings:** The above mentioned method identified can generate the metal nanoparticles. The longer laser exposure time with higher power contribute higher weight loss of the Al metal. The new incoming black dots were detected on the surface of Al alloy metal samples that were shot by a laser beam. It was contributed to the formation of instabilities plasma on the metal surface towards Al nanostructures ejection. The average weight of a mass loss of Al alloy increases with the exposure time of laser shot was increases. It was shown that average of mass loss of Al metal alloy increased dramatically from the 30 second to 60 second at laser power of 1 watt. However, the higher ablation condition of 3 watt for 180s forms the bigger particle size of 900 nm and more. **Conclusion/Application:** At the condition of 3 watts for 30s, smaller particles size ranging of 76-1281 nm has been formed. The higher power and exposure time subsequently increase the size and homogeneity.

Keywords: Aluminium Alloy, Liquid Pulsed-Laser Ablation, Metal Nanoparticles, Particles Size, Surface Morphology

1. Introduction

Nanotechnology deals with matters that have a narrow range of size and smooth. The characterization is based on the low melting temperature, high surface area, optical characteristics, mechanical strength and its magnetism^{1,2}. In³ made experimental demonstration whereby the laser is focused on the surface of pure Fe metal immersed in water medium. This method is known as Liquid Phase Pulsed Laser Ablation. This method is simple and clean. It does not require specific purification process, low cost preparation test and the laser parameter can be controlled.

Nanoparticle is defined as particles that have a diameter of less than 100 nm⁴. Normally, metal nanoparticles have physical and chemical characteristics that are different from common metals which are low melting point,

high surface area, mechanical strength property which is similar to ordinary metals and magnetism characteristics¹. When the particle size becomes smaller lower than 100 nm, the number of atoms on the surface of the material is increased with the total number of atoms in a substance. Ratio of the surface to volume of the material increases.

Laser is focused on very small spot with high intensity of light than sunlight. It is to be monochromatic laser. According⁵, monochromatic light waves is defined as a light wave having a single frequency or a single wavelength. The laser mechanisms can be understood by Neils Bohr model of the atom⁶. Electrons move around the nucleus of an atom. Electrons can be on a ground state and excited state. When the electrons move from excited state to ground state, the electrons receive energy than other electrons. These electrons will emit powerful light called a photon. However, there is one more situation

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that explains the process of laser light. When electrons receive photon from another electron and move from ground state to excited state, the photon energy is double increased. This situation is called stimulated emission electron. Laser light is generated. Besides⁷ was reported the Ag-Au alloy nanoparticles can be synthesized by a high-voltage electrical explosion of twisted wires (of Ag and Au) in deionized water. The compositions of obtained nanoparticles were changed by adjusting the cross-section ratio of Ag and Au twisted wires.

When the laser is shot on the surface of the target metal sample, some of the laser energy is reflected. The laser energy reflection process depends on the type of metal and laser wave⁸. Laser energy absorbed by the metal sample is transferred from the optical photons into electrons and then into the lattice particles. The laser power is absorbed by the target metal sample⁸. The high acceptance of laser energy causes a photochemical process occurs in which atoms and molecules are isolated from the surface of the target metal sample. The surface of target metal sample becomes hot and causes vaporization process, generates plasma filled with atoms and ionized electrons. The resulting plasma absorbs some of the laser energy. This plasma expands and heated due to excessive laser energy. Finally, the plasma begins to inject from the surface samples exposed of laser light to the liquid phase. Metal nanoparticles are synthesized as a result of laser ablation⁷.

The objective of the study is to produce Al alloy metal nanoparticles using laser ablation in liquid medium. In addition, the effect of laser exposure time and laser power to the mass loss of metal sample was also studied. Nanoparticle size and particle size distribution of nanoparticle metal samples were also studied. The materials used are metal Al alloy, ultra-pure water, perspex block and sandpaper.

2. Experimental

2.1 Metal Alloy Cleaning and Characterization

Al alloy metal samples are cut to size dimension of 2 cm x 3 cm into 12 samples and stored in 12 containers petri plates. The surface of Al alloy metal samples was washed with little oil to prevent rusting. Al alloy metal samples then are cleaned with ethanol and water. Al alloy metal samples are polished using Manual Grinding Machine at 100 rpm and clockwise. Al alloy metal samples are polished using

three different grades of sandpaper. Al alloy sample polishing metal started using coarse grade sandpaper, grade 400. Then, Al alloy metal samples are cleaned with ethanol and water. Polishing of Al alloy metal samples are repeated using sandpaper grade 800 and grade 1200. Al alloy metal sample polishing are according to ASTM standards⁹ in which the sample polished is begins with coarse grade sandpaper to fine grade sandpaper. Metal surface structure of the Al alloy sample (after polished) is determined using Optical Microscope Machine. Magnification scale is set at 500 microns. Al alloy metal sample composition was determined using XRF machine. Initial mass of Al alloy metal was weighed and the reading was taken three times.

2.2 Laser Exposure Studies

The 500 mL beakers are cleaned with distilled water and dried. Al alloy metal samples and Perspex block is placed into a beaker of 500 mL. Position of Al alloy metal sample is at the top of the block. Ultra pure water is poured into a beaker which has provided the samples of Al alloy metal and perspex block. Filter paper used to filter impurities and suspended particles. The total volume of ultra pure water that is poured into a beaker is in the range of 71 ml to 75 ml to ensure the distance between the surface of ultra pure water and the surface of Al alloy metal samples in the range of 2 mm to 5 mm. Laser power is set at 1 Watt. Laser is shot in the middle of the Al alloy metal sample surface for 30 seconds (s). Then, ultra-pure water is stored in the universal bottle. Al alloy metal samples were laser shot 1 Watt was dried and stored into petri plates. Beaker and perspex block is cleaned with distilled water. The experiment was repeated with a sample of Al metal alloy with exposure time, 60 s, 120 s and 180 s. Laser power is set to 2 Watt. The experiment was repeated with a time of laser exposure on Al alloy metal samples at 30 s, 60 s, 120 s and 180 s. Then, the laser power is set at 3. Laser exposure time on the sample of Al alloy metal is set to 30 s, 60 s, 120 s and 180 s. Final mass Al alloy metal samples were weighed with electronic scales and weight readings are taken 3 times. Figure 1 shows the experimental preparation for pulsed laser ablation system in which Al alloy metal sample is immersed in ultra-pure liquid water.

3. Results and Discussion

Production and properties of nanoparticles of Al alloy can be determined based on the parameters that have been

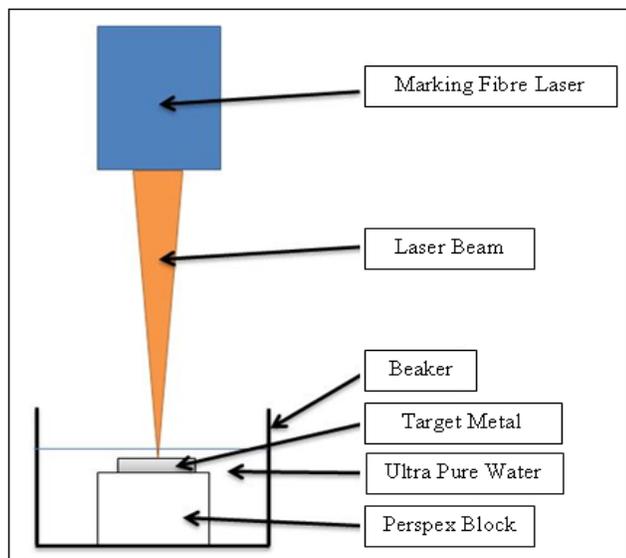


Figure 1. Pulsed laser ablation system.

studied which the average weight of a sample decomposition Al alloy, laser exposure time on the sample of Al alloy and laser power used. In addition, the morphology of the Al alloy samples also has been studied which is the morphology of the surface structure of Al alloy samples before and after laser exposure. Analyses of the sample composition Al alloy have also been studied. Table 1 shows the percentage composition of the components contained in Al alloy samples using XRF analysis. The sample used is metal Al metal alloy in which the Al metal component is a component of the highest percentage of 95.4 %.

The morphology of the surface structure of Al alloy samples have been studied using an optical microscope. The morphology of the Al alloy surface before and after laser beam treatment was shown in Figure 2 and Figure 3, respectively. Incoming black dots were detected on the surface of Al alloy metal samples that were shot by a laser beam. Al alloy metal plasma formed¹⁰ and the formation of nanostructures¹¹ that occurs on the surface of Al alloy metal sample were cannot be seen directly. Al alloy metal plasma rapidly expands as a result of the acceptance of high-power laser beam focused on the surface of Al alloy metal samples. Acceptance intensity of high power laser beam in the entire period of time led to the release of energy power of the laser beam is absorbed by the sample Al alloy metal. Nanoparticle Al alloy metal was released from the plasma¹². The area that is focused by the laser beam has suffered a loss of metallic Al alloy in which the metal nanoparticles Al alloy been released into the ultra

Table 1. Percentage composition of Al alloy

Component	Percentage Composition (%)
Al	95.4
Cl	0.58
Ti	0.009
Mn	0.11
Fe	0.697
Co	0.005
Cu	0.12
Zn	0.03
Ga	0.017
Pd	2.9
Cs	0.11



Figure 2. Surface morphology of Al alloy before laser shot (magnification of 500 μm).

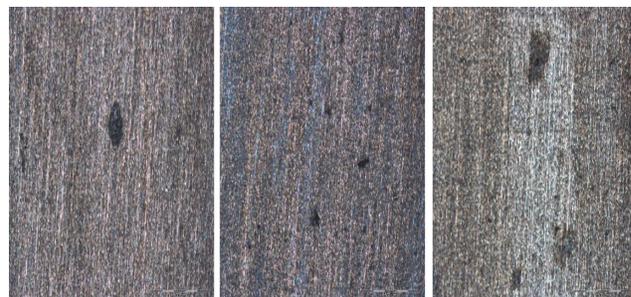


Figure 3. Surface morphology of Al alloy metal sample after laser shot (magnification of 500 μm).

pure water. The formation of nanostructures on the surface of Al alloy metal samples is formed.

Table 2 shows the average weight of mass loss of Al metal alloy in which Al alloy metal samples had been shot with the power of 1 Watt laser in ultra pure water. Time laser exposure on Al alloy metal samples was determined

from 30 seconds (s), 60 s, 120 s and 180 s. The average weight of a mass loss of Al alloy increases as the exposure time of laser shot increases. According to Figure 4, the graph of the average weight of mass loss of Al metal alloy increased dramatically from the 30 s to 60 s. Then, the average of mass loss of sample decomposes consistently.

Based on Table 3, the average weight of mass loss of Al alloy metals also increases when was shot with 2 Watt laser power in ultra pure water. In Figure 5, the average weight of mass loss of Al alloy metals rise. At the time from 30 s to 60 s, the average weight of a sample decomposition metal sample increase is not significant. At the time from 60 s to 180 s, the average weight of sample decomposition Al alloy metals rise sharply.

Table 4 shows the average weight of sample data decomposition Al alloy metal at laser power of 3 Watt. The

Table 2. Average mass loss of Al alloy metal sample at laser power 1 Watt

Time of laser exposure (s)	Average mass loss of Al alloy metal sample (g)	Average mass loss of Al alloy metal sample (g) $\times 10^{-3}$
30	0.000233	0.233
60	0.001133	1.133
120	0.001267	1.267
180	0.001400	1.400

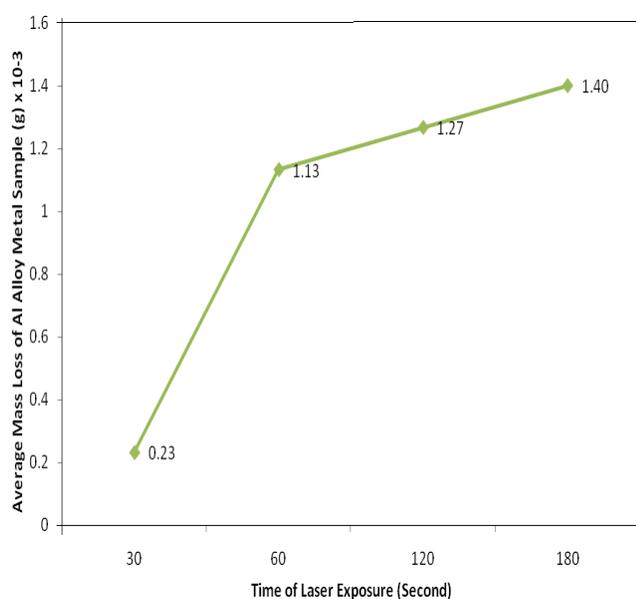


Figure 4. The average mass loss of Al alloy vs laser exposure time at laser power 1 Watt.

Table 3. Average mass loss of Al alloy metal sample at laser power 2 Watt

Time of laser exposure (s)	Average mass loss of Al alloy metal sample (g)	Average mass loss of Al alloy metal sample (g) $\times 10^{-3}$
30	0.001400	1.400
60	0.001667	1.667
120	0.002167	2.167
180	0.003100	3.100

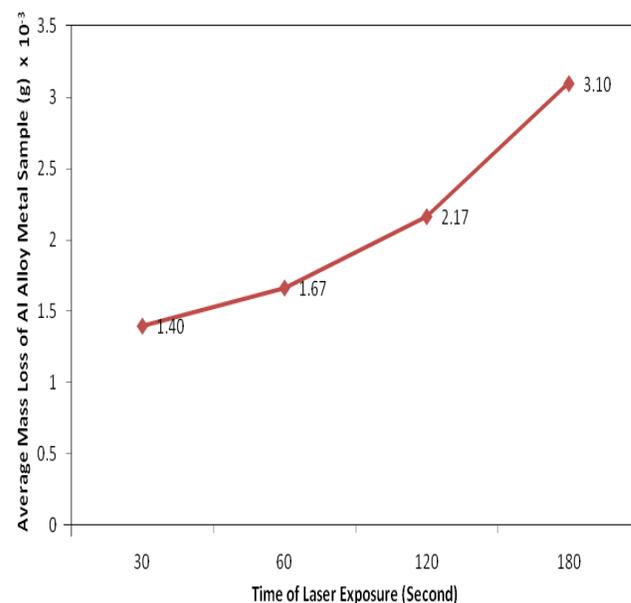


Figure 5. The average mass loss of Al alloy vs laser exposure time at laser power 2 Watt.

Table 4. Average mass loss of Al alloy metal sample at laser power 3 Watt

Time of laser exposure (s)	Average mass loss of Al alloy metal sample (g)	Average mass loss of Al alloy metal sample (g) $\times 10^{-3}$
30	0.003800	3.800
60	0.004033	4.033
120	0.004433	4.433
180	0.005667	5.667

results showed that the longer the exposure time of laser metal sample, the higher the average weight of sample decomposition Al-alloy metal. Based on Figure 6, there is an increase in the average decomposition of heavy metal samples dramatically from 120 s to 180 s. As a result, there are many differences between mass loss of sample before and after the shot fired by the laser.

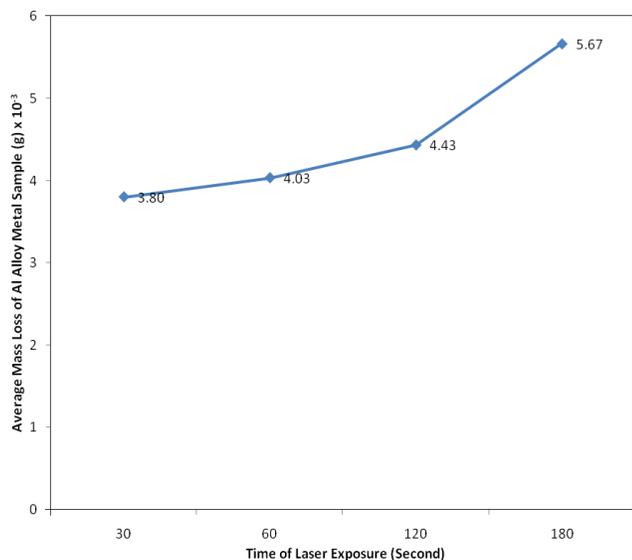


Figure 6. The average mass loss of Al alloy vs laser exposure time at laser power 3 Watt.

When the laser power is set at 2 Watt and 3 Watt, the mass loss of Al alloy samples are also increased when the laser exposure time is increased as shown in Table 3 and Table 4. This is due to the increasing in temperature and the heat received by the surface of the Al alloy metal samples during laser exposure and lead to increasing rapidly laser ablation rate. Al-alloy metal sample areas focused by the laser decompose rapidly and Al alloys heavy metal samples decreased¹³.

Other main parameter that play an important role in the production of metal nanoparticles Al alloy is laser ablation power. Figure 7 shows the average mass loss of Al alloy metal by different power laser ablation. Power laser ablation has been set for 1 Watt, 2 Watt and 3 Watt. These results showed that the average mass of a sample decomposition Al alloy metal increases as the laser power increased. Table 5 shows the average mass loss of Al alloy metal that has three types of laser power. It was shown that laser exposure time on a sample of Al alloy at 180 s and 3 Watt gives the highest average weight loss, represents to the decomposition Al alloy metals.

According¹⁴, when the laser power increased, the increase in heat will occur on the surface of Al alloy metal sample by a focused laser. Evaporation occurs too high in exposed areas focused by the laser also causes the formation of plasma. Plasma instabilities caused by a too high heat reception result of the high laser power in the release ejected of nanoparticles of metal and significantly reduced mass of Al alloy metal. The particle sizes distribution of Al

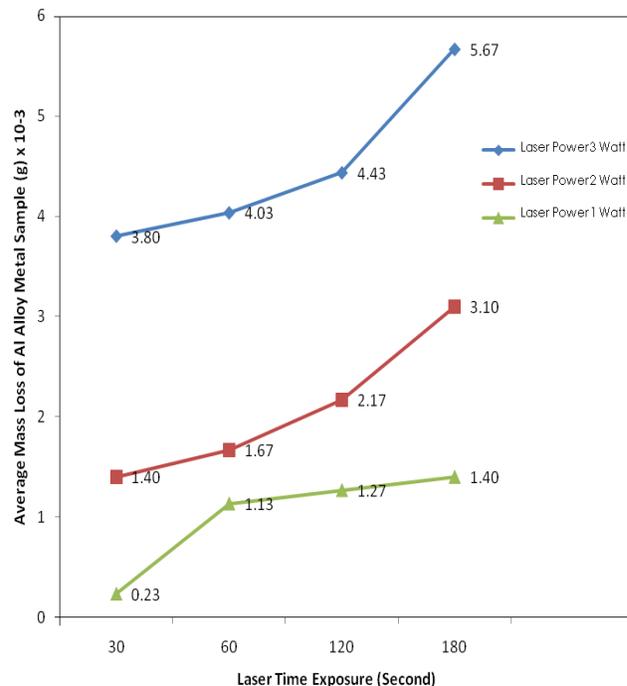
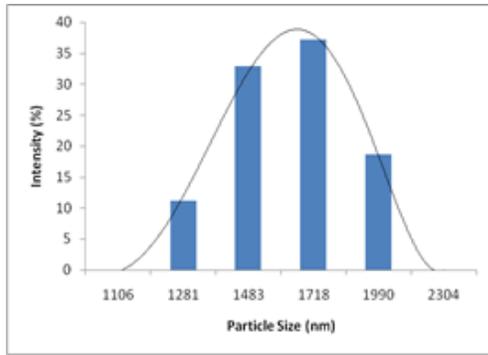


Figure 7. The average mass loss of Al alloy vs laser exposure time at different intensity of laser power.

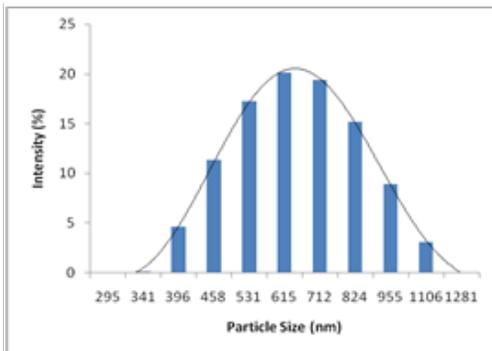
Table 5. Average mass loss of Al alloy metal sample varies laser power

Laser power (Watt)	Time of laser exposure (s)	Average mass loss of Al alloy metal sample (g)	Average mass loss of Al alloy metal sample (g) x 10 ⁻³
1	30	0.000233	0.233
1	60	0.001133	1.133
1	120	0.001267	1.267
1	180	0.001400	1.400
2	30	0.001400	1.400
2	60	0.001667	1.667
2	120	0.002167	2.167
2	180	0.003100	3.100
3	30	0.003800	3.800
3	60	0.004033	4.033
3	120	0.004433	4.433
3	180	0.005667	5.667

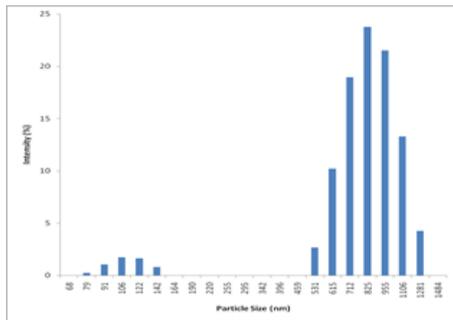
metal alloy at different laser power and ablation time were showed in Figures 8 to 11. It was clearly shown that Al alloy with smaller nanoparticles can be prepared at laser power and ablation time of 1 watt and 60 s, respectively.



(a)

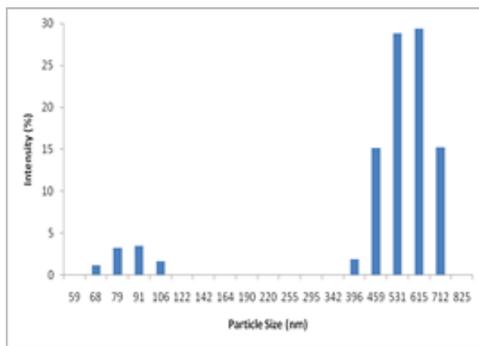


(b)

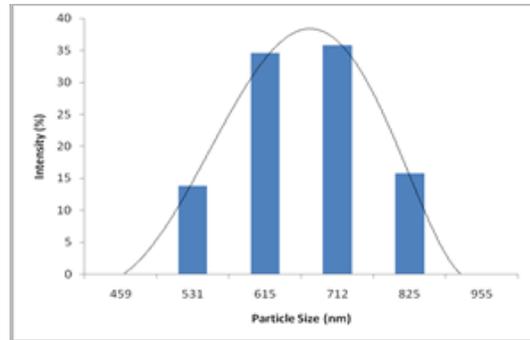


(c)

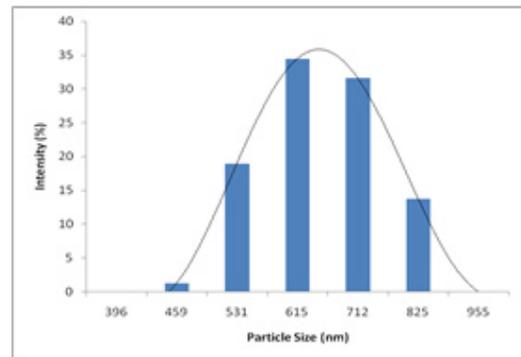
Figure 8. Particle size distributions Al alloy metal nanoparticle for 30 s exposure. (a) 1 Watt, (b) 2 Watt and (c) 3 Watt.



(a)

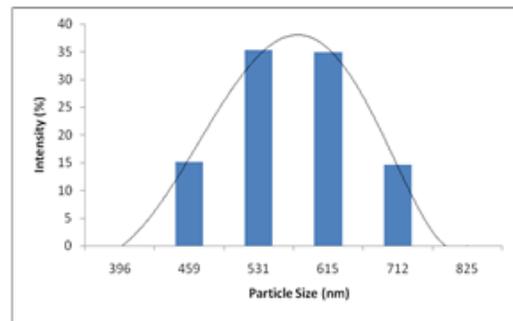


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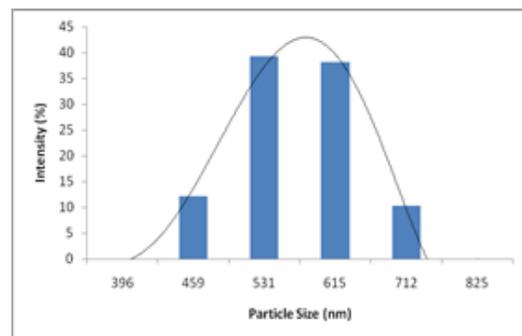


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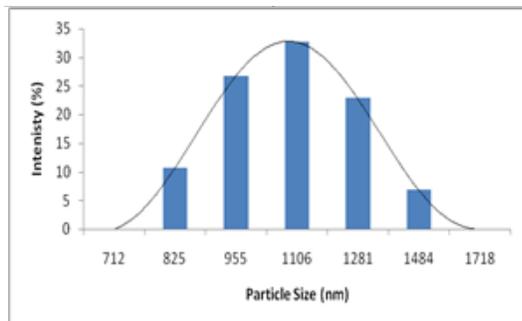
Figure 9. Particle size distributions Al alloy metal nanoparticle for 60 s exposure. (a) 1 Watt, (b) 2 Watt and (c) 3 Watt.



(a)

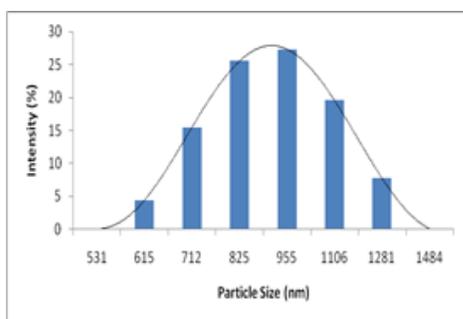


(b)

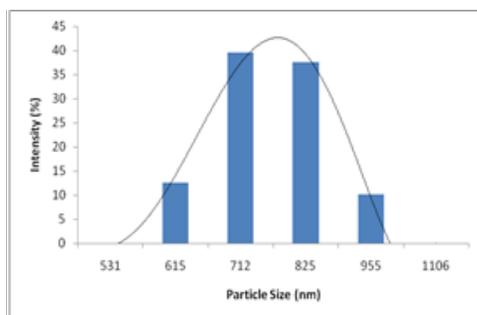


(c)

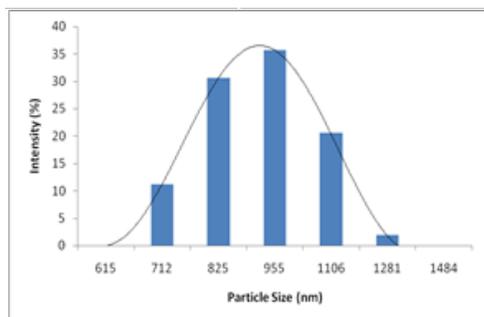
Figure 10. Particle size distributions Al alloy metal nanoparticle for 120 s exposure. (a) 1 Watt, (b) 2 Watt and (c) 3 Watt.



(a)



(b)



(c)

Figure 11. Particle size distributions Al alloy metal nanoparticle for 180 s exposure. (a) 1 Watt, (b) 2 Watt and (c) 3 Watt.

However, at higher ablation condition of 3 watt and 180 s, the Al alloy with bigger particle size more than 900 nm has been measured (Figure 11).

4. Conclusions

Based on this study, it was clearly shown the mass losses ranging of 0.233×10^{-3} to 5.667×10^{-3} gram are affected by the laser exposure time on the surface of Al alloy metal. This is maybe due to the increasing of heat and temperature around the surface of sample generated by the laser subsequently causes the evaporation and melting metal samples. Similarly to the laser parameters of the laser power, the higher power laser ablation, the higher mass loss of the target metal. This relates to the evaporation where formation of plasma on the samples is increased. Plasma is not stable as a result of high power laser. Metal nanoparticles are injected from the surface of the target metal. From the morphology of the sample, some black dots or crater are formed of the surface after being shot with a pulse laser ablation. Pulse laser ablation studies in the future should be extended other laser parameters such as different types of liquid medium, the distance between the liquid medium and target metal surfaces, and different metal. For the particle size distribution of metal sample, the result shows the particle size is not too fine and out scale of nano size. It is expected that the laser exposure time and laser power can increase the particle size distribution. At the condition of time exposure of 30 s at 3 watts, the smaller particles size ranging of 76–1281 nm has been formed. However, the higher power and exposure time subsequently increase the size and homogeneity.

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6. References

- Horikoshi S, Serpone N. Introduction to Nanoparticles. Microwaves in Nanoparticle Synthesis. Wiley-VCH Verlag GmbH and Co. KGaA; 2013 May. p. 1–24.

2. Patil P, Phase D, Kulkarni S, Ghaisas S, Kulkarni S, Kanetkar S, Ogale S, Bhide V. Pulsed-laser-induced reactive quenching at liquid-solid interface: Aqueous oxidation of iron. *Physical Review Letters*. 1987 Jan; 58(3):238–41.
3. Hong SM, Lee S, Jung HJ, Yu Y, Shin JH, Kwon KY, Choi MY. Simple preparation of anatase TiO₂ nanoparticles via pulsed laser ablation in liquids. *Bulletin of the Korean Chemical Society*. 2013 Jan; 34(1):279–82.
4. Abhilash M. Potential applications of nanoparticles. *International Journal of Pharma and Bioscience*. 2010 Jan; 1(1):1–12.
5. Singh SC, Zeng H, Guo C, Cai W. *Lasers: Fundamentals, Types, and Operations*. Nanomaterials. Wiley-VCH Verlag GmbH and Co. KGaA; 2012. p. 1–34.
6. Prasad MD. Niels Bohr and the atomic structure. *Resonance-Journal of Science Education*. 2013 Oct; 18(10):897–904.
7. Shoushtari MZ, Nezhad CR, Omidfar K. Fabrication and optical properties of Ag-Au alloy nanoparticles. *Indian Journal of Science and Technology*. 2016 Feb; 9(7):1–7.
8. Zhigilei LV, Lin Z, Ivanov DS. Atomistic modeling of short pulse laser ablation of metals: connections between melting, spallation, and phase explosion. *The Journal of Physical Chemistry*. 2009 Jun; 113(27):11892–906.
9. *Metallographic specimen preparation basics* by Pace Technologies. Available from: <http://www.metallographic.com/Technical/Basics.pdf>
10. Sasaki K, Takada N. Liquid Phase Laser Ablation. *Pure and Applied Chemistry*. 2010 May; 82(6):1317–1327.
11. Brown CL, Bushell G, Whitehouse MW, Agrawal DS, Tupe SG, Paknikar KM, Tiekink ERT. Nanogold Pharmaceuticals. *Gold Bulletin*. 2007 Sep; 40(3):245–50.
12. Harilal SS, Freeman JR, Diwakar PK, Hassanein A. Femtosecond laser ablation: Fundamentals and Applications. In: Musazzi S, Perini U, editors. *Laser-Induced Breakdown Spectroscopy*. Berlin Heidelberg: Springer; 2014. p. 143–66.
13. Mezzapesa FP, Sibillano T, Di Niso F, Ancona A, Lugarà PM, Dabbicco M, Scamarcio G. Real time ablation rate measurement during high aspect-ratio hole drilling with a 120-ps fiber laser. *Optics Express*. 2012 Jan; 20(1):663–71.
14. Jafarkhani P, Dadras S, Torkamany MJ, Sabbaghzadeh J. Synthesis of nanocrystalline titania in pure water by pulsed Nd: YAG Laser. *Applied Surface Science*. 2010 Apr; 256(12):3817–21.