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Synthesis of Aluminium Nanoparticles in A Water/Polyethylene Glycol Mixed Solvent using µ-EDM

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Abstract: Nanoparticles present a practical way of retaining the results of the property at the atomic or molecular level. Due to the recent use of nanoparticles in scientific, industrial and medical applications, synthesis of nanoparticles and their characterization have become considerably important. Currently, aluminium nanoparticles have attracted significant research attention because of their reasonable cost, unique properties and interdisciplinary emerging applications. The present paper reports the synthesis of aluminium nanoparticles in the mixture of Deionized water (DI water) and Polyethylene Glycol (PEG) using a developed micro-Electrical Discharge Machining (µ-EDM) method. PEG was used as a stabilizer to prevent nanoparticles from agglomeration produced during the µ-EDM process. The synthesized aluminium nanoparticles were examined by Transmission Electron Microscopy (TEM), Energy Dispersive Analysis by X-rays (EDAX) and Selected Area Electron Diffraction (SAED) pattern to determine their size, shape, chemical nature and crystal structure. The average size of the polyhedral aluminium nanoparticles is found to be 196 nm.

Keywords: Aluminium nanoparticle, µ-EDM, PEG, TEM.

1. INTRODUCTION

Nanotechnology is a broad and interdisciplinary area of research and development activity that has been growing worldwide in the past few years. It has the potential for revolutionizing the ways in which materials are created at nano level scale known as nanoparticles. Nanoparticles are cornerstones of nanotechnology which play a significant role in the present century, due to their enhanced size-dependant characteristics compared to extremely fine or larger particles of the same material. Their uniqueness arises from their large surface area to volume ratio, as these materials have size typically in the range of 1 nm to 100 nm. During the past few decades, metallic nanoparticles have received significant curiosity by the researchers because of their novel physical, thermal, electrical and catalytic properties, and interdisciplinary emerging applications [1,2]. In recent years, nanofluids have attracted great interest because they are proved to be much more superior when compared to the base fluids (conventional fluids).

Nanofluids are known as colloidal suspensions, i.e. suspension of nanoparticles in a base fluid. In most of the studies the common base fluids include either water or organic liquids. For various industrial applications, nanofluids are prepared by combining a large variety of nanoparticles and base fluids. Nanofluids have become significant in various industries due to their enhanced thermal conductivity compared to the base fluids [3,4].

Aluminium nanoparticles have recently attracted significant attention in the midst of a choice of metal particles because of their reasonable cost, unique aforesaid properties, and are widely used in various potential applications include automobiles, catalysis, solid rocket propellants, explosives, lubrication, etc [5,6,7]. A wide variety of liquid and vapor phase reaction methods have been developed for the synthesis of aluminium nanoparticles. These include combustion flame [8], laser ablation [9], wire explosion [10], wet chemical process [2], and aerosol synthesis [11]. Karasev et al. [8] have studied the formation of alumina nanoparticles by combustion of Al droplets in air. They found that the alumina nanoparticles are generated as aggregates composed of primary particles whose diameter lies in the range of 10-140 nm. The size of aggregates varies in the range from 0.1 to a few microns. Kuzmin et al. [12] generated Al nanoparticles by laser ablation of Al target in water and ethanol saturated with hydrogen. They found that the particles are almost spherical in shape with the size range of 30-50 nm. Sarathi et al. [10] used wire explosion technique to generate aluminum nanoparticles in different inert ambiences. The particles generated were found to have a mean diameter between 30-45 nm. Lee and Kim [13] prepared aluminium particles with geometric mean diameters of 139-614 nm and good monodispersity in dibutyl ether by a wet chemical process. They found that by using oleic acid as an organic surfactant to the precursor solution, the size of aluminium particles have reduced to approximately 35 nm. Hemalatha et al. [2] synthesized ethylene glycol based alumina nanofluids through chemical routes. The average particle size was found to be 43 nm. Kalpowitz et al. [14] have used using aerosol synthesis method to generate aluminium nanoparticles. The size of polyhedral aluminium particles lies in the range of 50 nm to 100 nm with an average particle size of approximately 87 nm.

However, from the above mentioned methods used by the researchers for synthesis of aluminium nanoparticles, some of them are normally hard to control the particle size and distribution, and their subsequent aggregation. Further, most of these methods exhibit low production rates and high cost. Therefore it is essential to find a simple, compact, versatile and cost-effective method for synthesizing aluminium nanoparticles at high yield rate. The present paper presents a micro-Electrical Discharge Machining (μ-EDM) method for synthesis of aluminium nanoparticles in the mixture of deionized (DI) water and stabilizer - Polyethylene Glycol (PEG) which has not yet addressed in detail. This method
does not require costly equipment in its setup, leading to remarkably lower investment costs compared with the other methods. μ-EDM method is a non-contact machining process which is basically, based on the thermo-electric energy created between the two electrodes- tool and the workpiece surrounded by dielectric fluid. In this process, the material is removed from both the electrodes in the form of debris through melting and evaporation by the initiation of repetitive spark discharges. The aluminium nanoparticles synthesized in the dielectric medium were examined by Transmission Electron Microscopy (TEM), Energy Dispersive Analysis by X-Rays (EDAX) and Selected Area Electron Diffraction (SAED) pattern to determine their size and distribution, chemical nature and crystal structure.

2. EXPERIMENTAL DETAILS

The schematic diagram of the indigenously developed µ-EDM setup is illustrated in Fig. 1. The µ-EDM cell consists of a tool electrode (cathode) and a workpiece electrode (anode) separated by a small gap known as spark gap, and submerged in a dielectric medium (DI water). In the setup, a piezoactuator is used for tool feed control during micro-machining to maintain a proper spark gap between the electrodes.

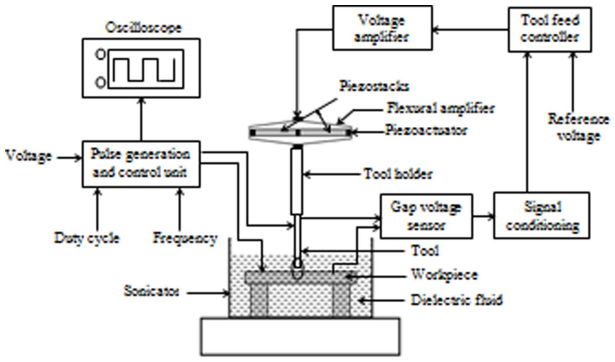

Fig. 1. Schematic diagram of the indigenously developed µ-EDM setup

The µ-EDM system was attached to a pulse-generation circuit with a transistor type discharge control. This enabled the system to accurately control the discharge energy for a single pulse. The transistor type can handle large current and thus, the material removal rate is much higher with this generator. The pulse control circuit consists of pulse width modulator, which produces rectangular pulses with different frequencies. The frequency and the duty cycle of the discharge can be monitored by changing the resistance values of the potentiometers. The pulse duration could be set by varying the frequency and the duty cycle. As soon as the short-circuit signal caused by direct contact of tool and the workpiece is detected, the tool is retracted. This prevents the damage of the tool and the workpiece. The feedback circuit senses the average gap voltage and this signal is compared with reference voltage to detect the gap status. Then, the information of discharge gap is transferred to a tool feed rate controller that it sends to the linear amplifier, which amplifies the voltage and makes it fall between safe operating range of the actuator. This amplified signal is directly sent to the actuator which feeds the tool and controls the spark gap. An ultrasonicator (frequency 55 kHz) was used for efficient removal of debris from the spark gap region and to avoid the accumulation of debris in the spark gap. This result in stable spark discharges and increased machining efficiency. During machining, the pulse discharge waveform were monitored and captured by Hewlett Packard oscilloscope.

The machining parameters of µ-EDM used for the synthesis of colloidal aluminium nanoparticles are listed in Table 1.

Table 1. Machining parameters of µ-EDM for colloidal aluminium nanoparticles synthesis

Parameters	Values
Dielectric fluid	200 ml
Workpiece thickness	460 µm
Tool diameter	900 µm
Input voltage	20 V
Peak current	2 A
Frequency	4 kHz
Duty cycle	30%
Pulse duration	75 µs
Environment	Room temp.

Before starting of the experiment, the aluminium electrodes were cleaned with acetone to remove the impurities, if any, present on the surface. This may ensures the generation of pure aluminium particles during the experiment. A stabilizer - PEG is added to the DI water (concentration- 1wt/vol % in DI water) to prevent the agglomeration and settling of aluminium nanoparticles generated during the experiment.

Based on the experimental study, the required input voltage as shown in Table 1 is set for the experiment. From the initial position the tool is moved towards the workpiece using a vertical slide till the gap between the tool and the workpiece reaches a value equivalent to the spark gap. When the required spark gap was reached, sparks are produced at the supplied pulse frequency across the plasma channel, generated by the breakdown of the dielectric fluid. Due to the thermal action of sparks the material is removed in the form of debris from the electrodes through melting and evaporation. As the spark gap is increased during machining the tool is fed automatically by the piezoactuator so that the machining at each point can continue without any interruption. The debris of aluminium is suspended in the mixed solvent and the obtained colloidal suspension was collected in the glass vials. Then, characterization of the colloidal suspension of particles was carried out using TEM, EDAX and SAED studies.

3. RESULTS AND DISCUSSION

The TEM image of aluminium nanoparticles synthesized in the pure DI water and PEG mixed solvent using µ-EDM method is shown in Fig. 2. The particles produced are polyhedral in shape and dispersed with a little broad size distribution (range: 45 nm–500 nm and mean size: 196 nm) as shown in Fig. 3. Due to the polyhedral shape of the particles, diagonal distances were used as the descriptive size measurement for each particle. The generation of polyhedral aluminium particles may be attributed to the annealing of spherical particles formed during nucleation and particle growth.

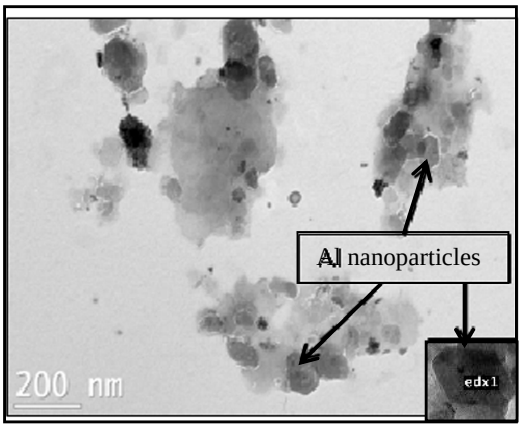

Fig. 2. TEM image of Al nanoparticles in DI water and PEG mixed solvent.

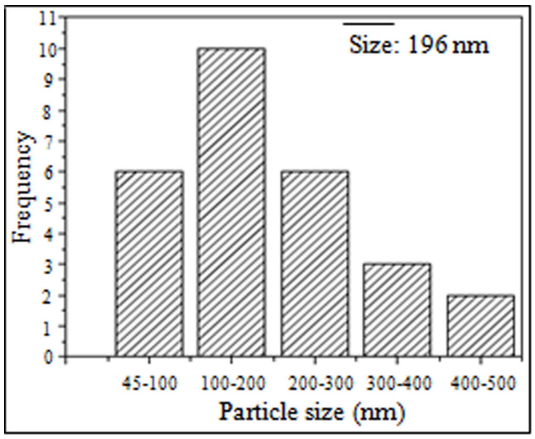

Fig. 3. Size distribution of Al nanoparticles in DI water and PEG mixed solvent.

The EDAX pattern of polyhedral particle as shown in Fig. 4 exhibits a clear peak of aluminium, carbon and oxygen intensity peak seems to have originated from the PEG stabilizer present in DI water. The possible presence of other trace level impurities cannot be excluded.

Fig. 5 shows the SAED pattern of aluminium nanoparticle in the mixed solvent. The image showed a bright ring pattern rings, with the first ring diameter from the center yielding lattice (d) spacing of 0.239 nm, that perfectly matched with the crystalline aluminium lattice distance of 2.33 Å for (111) plane. However, the diffraction pattern shows amorphous diffuse scattering rings rather than clear diffraction rings. This is attributed to the amorphous nature of stabilizer present in the peripheral regions of the particles. Thus from the EDAX and SAED outcomes it is confirmed that the particles generated are of crystalline aluminium with a thin shell of oxide coating.

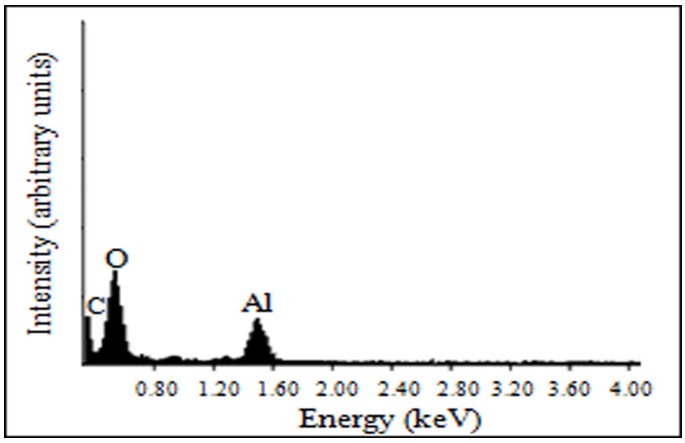

Fig. 4. EDAX pattern of Al particles in DI water and PEG mixed solvent.

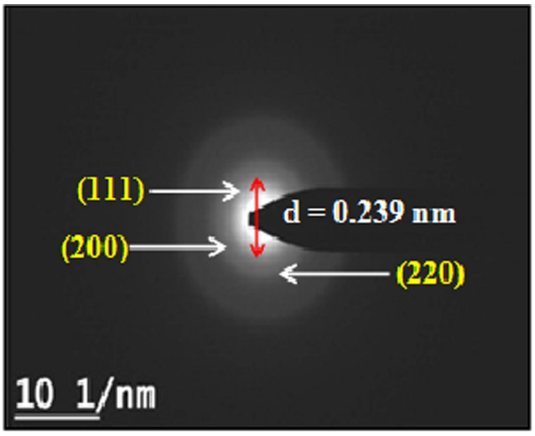

Fig. 5. SAED pattern of Al nanoparticle in DI water and PEG mixed solvent.

When PEG was added to the DI water, the mean size of the aluminium particles has observed to be larger in size rather than less than 100 nm. This is attributed to the clustering of particles. This indicates that the PEG polymer does not form a strong barrier to prevent close contact between aluminium particles. Also, by visual inspection sedimentation of nanoparticles in DI water and PEG mixed solvent was taking place after 24 hr of the collection of the colloidal suspension.

4. CONCLUSIONS

In this investigation, aluminium nanoparticles are synthesized in DI water/PEG mixed solvent using µ-EDM method. Characterization was carried out for the synthesized colloidal suspension of aluminium particles, and the conclusions are drawn as follows:.

1. The TEM study shows that the size of aluminium nanoparticles suspended in DI water and PEG mixed solvent lies in the range of 45-500 nm and mean size was found to be 196 nm.
2. EDAX and SAED analysis confirms the presence of crystalline aluminium particles with a thin shell of oxide coating.
3. The size distribution study of synthesized aluminium particles shows that the measurement follows normal distribution.

Further work is needed to reduce the size and sedimentation of the colloidal aluminium particles synthesized using µ-EDM method.

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