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Synthesis of Copper Nanoparticles through Wire Explosion Route

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Abstract
In the present study, nanoparticles produced by wire explosion technique are characterized by Field Emission Scanning Electron Microscopy (FESEM) and X-Ray Diffraction (XRD) techniques. Wires having different diameters were fused at different energy levels. It immerged that energy deposited in the conductor and diameter of wire used have major impact on particle size produced by this technique. The parameters of the capacitor energy bank used in this study is much different from current trends of experimental parameters used in other studies.
*Keywords-* Copper nanopowder, FESEM, wire explosion technique, XRD.

I. Introduction

The nanotechnology and nanoscience are acquiring utmost importance in the field of scientific research and commercial developments. The nanoparticles are produced by using techniques like chemical methods, physical methods, mechanical methods and biological methods etc. One of them is wire explosion technique of producing nanoparticles, which has recently gained immense importance. The present study deals with nano copper particles production through wire explosion technique. The technique has various features that make it different & superior to other techniques. It consumes less time with high production rate. It is ecologically safe and is also pollution free process.

Nanotechnology has gained significant importance in the field of Science and Technology. Nanotechnology is the functional system of engineering which works at the molecular scale. In other words, nanotechnology is defined as a technology that can be engineered at the molecular level of material particles. It has got a lot of advantages in almost all industries [1]. It will lead to develop high quality, long lasting, clean, easy, and quick products for home, transport, communication, machines, agriculture and general industries [2]. Many Scientists are working on the nanotechnology for the generation of nanoparticles for better suitability in the new products that are made up of nanomaterials. Nanotechnology works at one dimension size of material particles, which ranges from 1 to 100 nanometers. It deals with atomic and molecular scale. The nanoparticles are used for manufacturing of cutting tools, metal farming tool, aerospace, automobile, home appliances etc. In nanotechnology, a particle is defined as a small object that behave as a whole unit with respect to its transport and properties.

Particles are further classified according to diameter. Coarse particle cover a range between 10,000 and 2,500 nanometers. Fine particles are sized between 2,500 and 100 nanometers. Ultra fine particles, or nanoparticles are sized between 1 and 100 nanometers [3].

In the electrical method, the nanopowder is synthesized by electrical or wire explosion. The method is also known as pulse electrical discharge technique. The process of explosion of wire is possible by passing pulse current through the wire. The present work is based on this electrical explosion method for production of copper nanopowder. Subsequent sections, discuss the wire explosion method adopted in present study. Exploding wire method is method for the production of metal and metal oxide nanoparticle that is capable of production of bulk amount of metal nanoparticles at low cost.

The basic circuit used in the wire explosion technique for production of nanoparticles is shown in Fig. 1.1. The circuit diagram consists of three units i.e. charging circuit, discharging circuit and capacitor bank. The capacitor bank is charged with high voltage charging system (DC source). The HV switch is used for the supply of current and to stop the current. The thin wire is mounted on the explosion die where L and R are the inductance and resistance of the circuit.
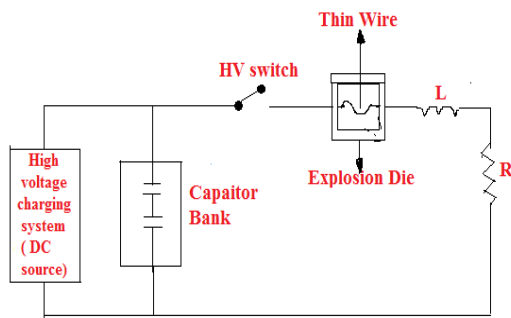
L- Equivalent Circuit Inductance;
R- Equivalent Circuit Resistance
Fig. 1.1 Circuit Diagram of Electrical Explosion
Method (set-up) [4]

In the present study experiments were conducted using different voltage/energy in copper wires of different diameter. Effect of operational parameters on the characteristics of nanoparticles has been studied.
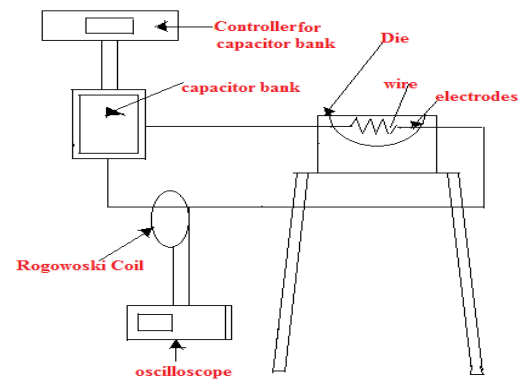
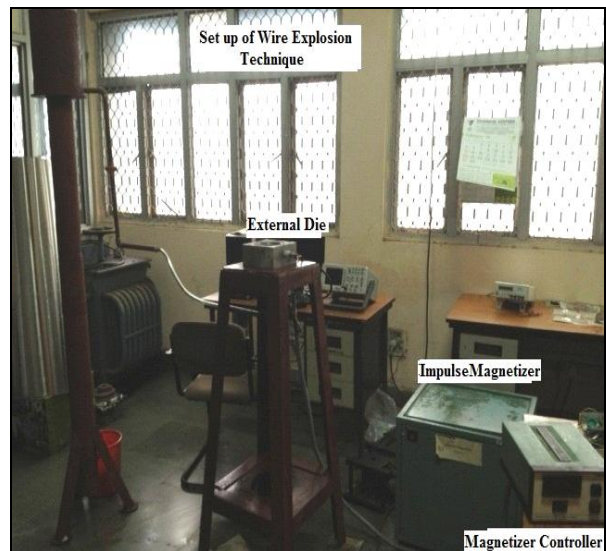## II.   Experimental
2.1 Experimental Set-Up
The sample of nanopowder was produced using wire explosion technique. Experimental set up was prepared by modifying an impulse magnetizer of walker, Scientific Inc. make. The highest storage capacity of the capacitor bank is about 5 KJ. It was modified by connecting the output lead, which was initially connected to pulse magnetizer die, to the electrode end of electro hydro forming die.

Figure 2.1(a) shows schematic of the experimental set up. All the components and instruments attached are represented using line diagram. Capacitor is shown in the figure basically represented as charging circuit, discharging circuit and capacitor bank itself. A controller is attached to the capacitor bank. It sets the charging limit voltage to which capacitor has to be charged to get desired energy storage. The two output leads of the capacitor bank are connected to two ends of the die electrodes. The fusing wire of desired cross section are mounted on the other end of these electrodes. It is also shown that Rogowski coil is mounted on one of the lead from capacitor bank. The coil is attached to oscilloscope to measure current waveform.
Figure 2.1(b) shows photo graph of wire explosion set up.

(a) Schematic representation of experimental setup

(b) Photograph of the setup
Fig. 2.1 Experimental setup

2.2 Description of External Die
Figure 2.2(a) shows schematic of die used in present study. It has hemispherical cavity inside which two electrodes are mounted. The die insulation between the electrodes and die is provided by nylon bushing between them. Sample is placed between lower die and upper blank holder plate. The plate is rigidly fixed using nut-bolts as shown in the Fig. 2.2(b).

Figure 2.2(a) shows the die without upper binding plate. A copper wire is clearly seen mounted on the two electrodes using scew.

Figure 2.2(b) shows the whole die and sample in mounted condition. The nut-bolts used to fastened the set up can also be seen.

(a) Die with wire mounted
(without blank holder)

(b) Die in assembles condition
Fig. 2.2 Schematic diagram of external die used in
wire explosion technique

2.3 Materials
Copper wire of varying diameter was used as material in the present study. The particle size of the nano powder depended on the diameter of the copper wire which was used in the experiment. The diameters of the wire used in the experiment were of 0.24mm, 0.54mm , 1.08 mm size.

2.4 Methodology
The effects of energy and wire diameter were studied using copper wire of 0.24 mm, 0.54 mm, and 0.54×2 mm diameters. Wire was mounted on the electrode and the cavity was filled with water. Aluminum sheet was used to cover the water cavity. The sheet was fixed using a blank holder plate on the top. The plate was tightened using four sets of nut-bolts and washers. The approximate estimation of blank holding force applied is 40 KN.

The desired energy was produced by setting the voltage limit to which the capacitor was to be charged. The applied voltage was set in the magnetizer controller. The value of applied voltages used in the experiment was 600 V to 950 V at some interval. First 0.24 mm diameter of copper wire was fused with the applied voltage of 600 V, 700 V, and 800 V. The wire was heated and vaporized by the energy deposited in the wire. The mounted wire fused instantly and the nano powder was produced.

Similarly, the process was repeated with the 0.54 mm and two number of 0.54 mm of copper wire at the applied voltage of 600 V, 700 V, 800 V and 900 V. The prepared samples of nanoparticles was present in the mixture of water and copper nanoparticle. The water was dried off from the samples and copper nano powder was separated from the water.

2.5 Experiments
Experiments were conducted at different parameters to see the effect of the same. The parameters are listed in table 2.1.

Table 2.1: Detail of prepared samples

Sample No.	Sheet Dimensions (l×b×t) cm×cm×mm	Wire Dia (mm)	Wire cross Section (mm²)	Voltage(V)/ Energy (KJ)
(1)	(13.2×13.2×0.3)	0.24	0.0452	600/1.89
(2)	(13.2×13.2×0.3)	0.24	0.0452	700/2.572
(3)	(13.2×13.2×0.3)	0.24	0.0452	800/3.36
(4)	(12.5×12.5×0.3)	0.54	0.2289	600/1.89
(5)	(12.5×12.5×0.3)	0.54	0.2289	600/1.89
(6)	(12×12×0.3)	0.54	0.2289	700/2.572
(7)	(12.5×12.5×0.3)	0.54	0.2289	800/3.36
(8)	(12.5×12.5×0.3)	0.54	0.2289	900/4.252
(9)	(13.2×13.2×0.3)	0.54	0.2289	900/4.252
(10)	(13.2×13.2×0.3)	0.54	0.2289	950/4.738
(11)	(13.2×13.2×0.3)	2×0.54	0.9156	600/1.89
(12)	(13.2×13.2×0.3)	2×0.54	0.9156	700/2.572
(13)	(13.2×13.2×0.3)	2×0.54	0.9156	800/3.36

III.   Results and discussion
Typical waveforms of the current obtained at 700 V using two wires of 0.54 mm diameter are shown in Fig. 3.1. It may be seen that the rise time is not more than 50 µs. The whole process took about 100-µ seconds. The peak is observed at about 50-µ seconds. The typical frequency of the waveform was estimated to be approximately 8 to 10 kHz and peak current was appear 10 kA.
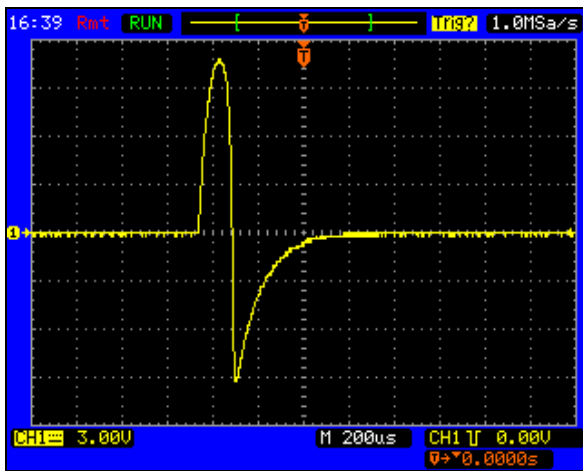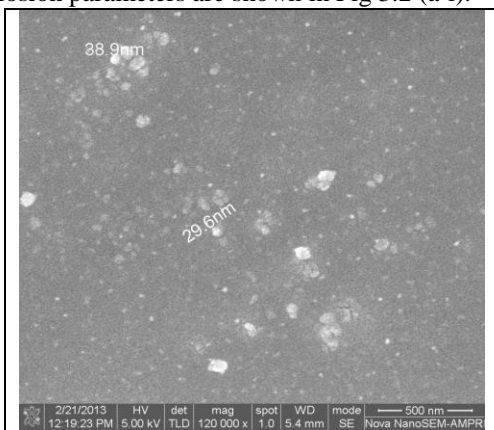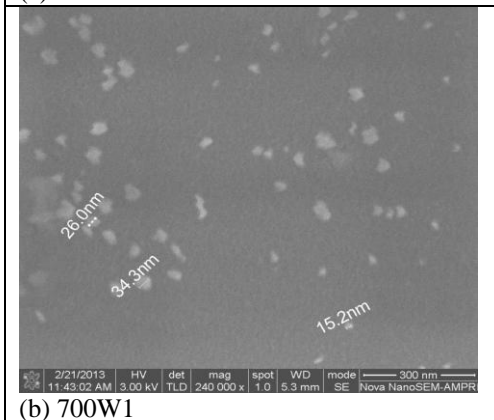
Fig. 3.1 Typical waveform of the current

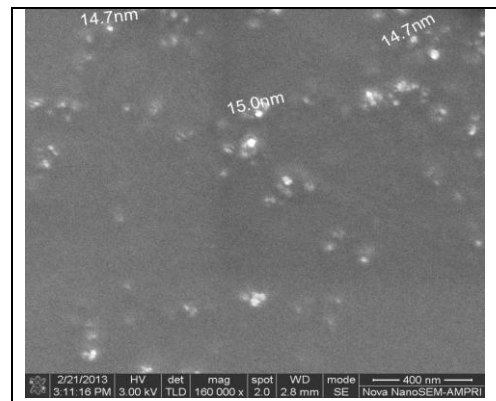## 3.1 FESEM Analysis of Nanoparticles

The particle size of the nanoparticles was measured from micrograph obtained by Field Emission Scanning Electron Microscopy (FESEM). The FESEM micrographs obtained at different wire explosion parameters are shown in Fig 3.2 (a-i).
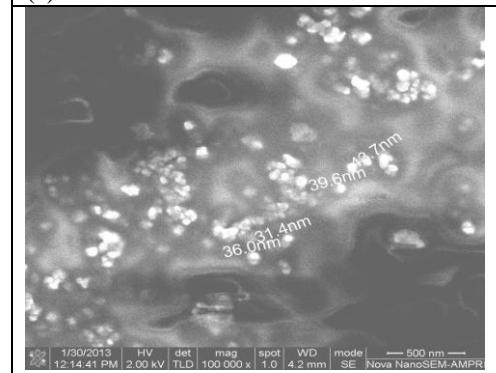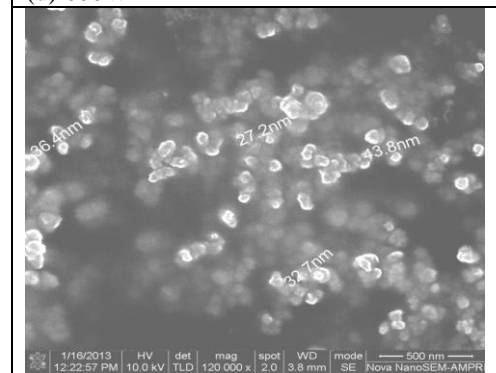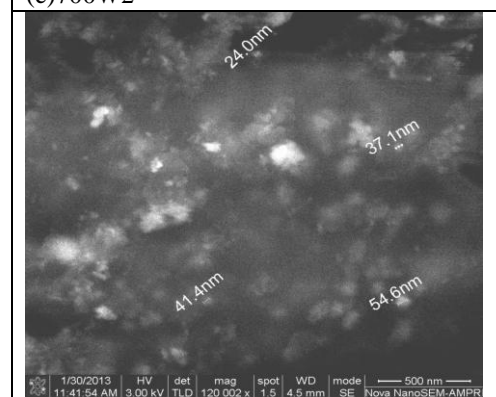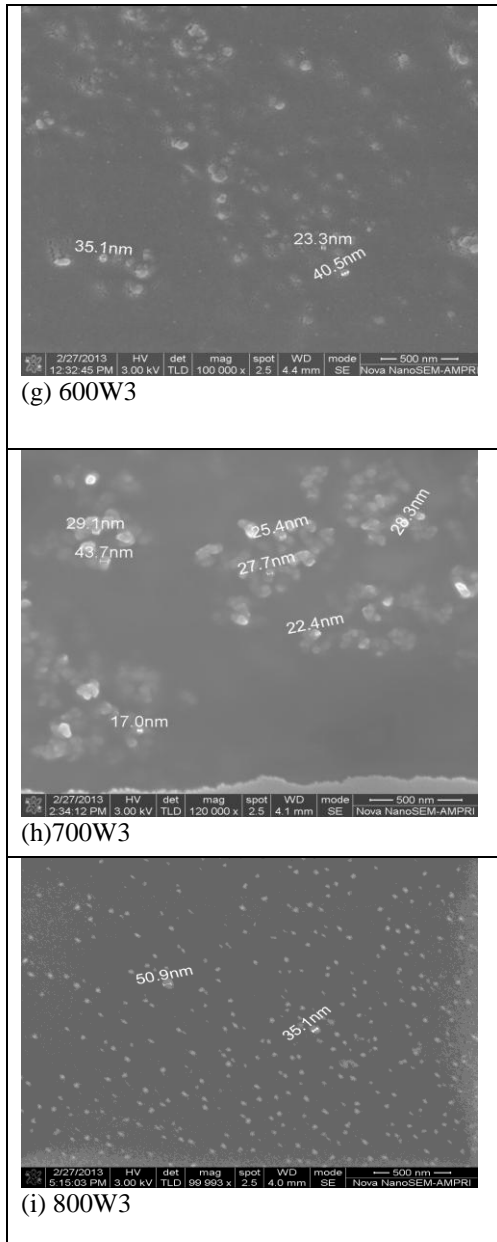

(a) 600W1

(b) 700W1

(c) 800W1

(d) 600W2

(e)700W2

(f) 800W2

(g) 600W3

(h)700W3

(i) 800W3

Note: W1: wire dia 0.24 mm; W2: wire dia 0.54 mm; W3: wire dia 0.54x2.
e.g. 700W2 means Micrographs of samples at 700V with 0.54 mm dia wire.

Fig. 3.2 Micrographs showing nano particles obtained at different energy and wire diameter.

Figure 3.2 shows the affect of energy and wire cross-section on nanoparticles size. The particle size of nanoparticles decreased as the energy increased. The particle size reduced from 30 nm to 17 nm for 0.24 mm diameter of copper wire as energy increased from 1.89 KJ to 3.36 KJ. Similar trends are observed for copper wire of other diameters (0.54 mm and 2×0.54 mm). Copper wire of higher diameter produced larger size particle at same energy level. Copper wire of 0.54 mm diameter produced 40- 35 nm particles as the energy increased from 1.89 KJ to 3.36 KJ. The particle size reduced from 35nm to 25 nm

when two parallel wires were used in place of one 0.54 mm wire ϕ and energy increases from 1.89 KJ to 3.36 KJ.

Table 3.1 Effect of diameter and energy on particle size

S. No.	Wire diameter (mm)	Voltage(V)/ Energy (KJ)	Average Particle Size (nm)	Particle size range (nm)
1.	0.24	600 / 1.89 700 / 2.572 800 / 3.36	30 <25 17	16.5- 66 17.0-38.25 6.66-46.66
2.	0.54	600 / 1.89 700 / 2.572 800 / 3.36	40 30 35	20-100 16.6-43.16 8.3-to 53.9
3.	2×0.54	600 / 1.89 700 / 2.572 800 / 3.36	35 29 25	10-80 16.6-49.8 16.6-45

3.2 X-Ray Diffraction Analysis

The X-ray diffraction analysis of nanoparticles is carried out by using C_u-K_α radiation of wavelength value (approximately λ) 1.5 Å for the prepared sample. Figure 3.3 shows the XRD spectra of nano copper particles using the wire explosion technique. The graph is plotted between the intensity (counts) and angle, where the x-axis indicates the values of 2θ and y-axis indicates the values of counts. A radial scan of diffraction angle (2θ) Vs intensity is shown where the angle is obtained from 40 to 90°. The four major peaks are recorded in the entire scan. It is observed that the first peak at 2θ= 43.070° is the characteristic peak of the copper particles indexed as [111] plane of copper phase at d= 2.09. The second peak at 2θ= 50.205° is the characteristic peak of the copper particles indexed as [200] plane of copper phase at d= 1.81. The third peak at 2θ= 73.973° is the characteristic peak of the copper particles indexed as [220] plane of copper phase at d= 1.28. The last major peak is recorded at 2θ= 89.784° is the characteristic peak of the copper particles indexed as [311] plane of copper particle at the values of d= 1.09 respectively. The all other peaks are at constantly forms may be considered as noise. No additional peaks were observed in the entire scan. Table 3.2 shows the data recorded in the X-ray diffraction analysis of copper nanoparticles.
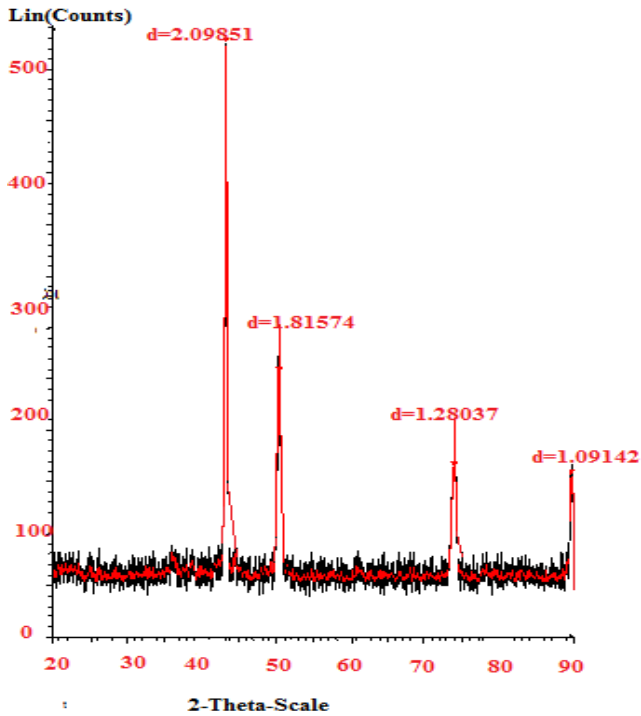
Fig. 3.3 X-Ray Diffraction of Copper
Nanoparticles

Table 3.2 Data of X-Ray Diffraction Analysis

S.No	Angle (2θ)	Value of 'd' (Å)	Counts	Intensity %	h k l
1.	43.070	2.09	576	100	1 1 1
2.	50.205	1.81	258	44.8	2 0 0
3.	73.973	1.28	165	22.8	2 2 0
4.	89.784	1.09	156	24.5	3 1 1

IV.   Conclusions
Following conclusions may be drown from this study:
1. Under present experimental set up increase in energy reduces the particle size and increase in wire diameter of copper reduces the particle size. No linear trend relation between these parameters exists.
2. The range of particles size is 10 nm to 100 nm obtained by wire explosion technique that is analyzed by field emission scanning electron microscopy.
3. Higher energy with the lower wire diameter should be preferred for finer size nanoparticles.

4. For the production of nanoparticles, the technique requires less time that is 100 μ-sec and lead to high production rate.
5. The wire explosion technique is ecologically safe, pollution free process, which is desirable for our environment protection.
6. Other material like zinc, alluminium etc may be taken up for the study and various combination of operating parameters and its affect on nanoparticles production may be studied.

V.   Acknowledgement

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