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Synthesis and Characterization of Nano Copper Particles

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Abstract: Nanoscience and Nanotechnology are the growing fields of scientific research and commercial development. Nanosize powders are solid particles with typical size in the range of 1 to 100 nm. Nanosize powders have very large specific surface area giving rise to many special physical and chemical properties. Exploding Wire Method (also known as EWM) is a high energy density process by which a rising current is applied to a thin electrically conductive wire. A fine wire of metal is exploded by applying by a high voltage. The high voltage is usually Impulse voltage. The heat vaporizes the wire, and an electric arc over that vapour creates a shock wave and explosion. Exploding Wire Method is the best method for production of metal nanoparticles. When Exploding Wire Method is performed in a standard atmosphere containing oxygen, metal oxides are formed. Pure metal nanoparticles can also be produced with EWM in an inert environment, usually argon gas or distilled water. Pure metal nanopowders must be kept in their inert environment because they ignite when exposed to oxygen in air.

***Keywords**: Nano Particles, Wire Explosion Method, Trigatron Switch, XRD, SEM, TEM.*

I. INTRODUCTION

Nanoscience and nanotechnology are the study and application of extremely small things and can be used across all the other science fields, such as chemistry, biology, physics, materials science, and engineering. The ideas and concepts behind nanoscience and nanotechnology started with a talk entitled ”There’s Plenty of Room at the Bottom” by physicist Richard Feynman at an American Physical Society meeting at the California Institute of Technology (CalTech) on December 29, 1959, long before the term nanotechnology was used. In his talk, Feynman described a process in which scientists would be able to manipulate and control individual atoms and molecules. Over a decade later, in his explorations of ultraprecision machining,

Professor Norio Taniguchi coined the term nanotechnology. It wasn’t until 1981, with the development of the scanning tunneling microscope that could ”see” individual atoms, that modern nanotechnology began.

Nanoscience and Nanotechnology are the growing fields of scientic research and commercial development. Nano size powders or Nano powders are solid particles with typical size in the range of 1 to 100 nm. Nano size powders have very large specific surface area giving rise to many special physical and chemical properties. The uniqueness of nanoparticle arise from their high ratio of surface area to volume. For this reason, nano size powders are of many interesting application in material processing for electronics, magnetics, and optics. They are also applicable as catalysts, pigments and even used as propellants.

The production of nanosized powder is one of the main application of wire explosion, and it is a simple method for producing various kinds of nanosized powders with particle diameters of less than 100 nm. In the present work, wire explosion technique is adopted, which is a top down approach to produce nanopowders. This is basically an evaporation technique, where the particles are produced by passing a high pulsed current over a thin metallic conductor. This deposited energy melts, evaporates and ionizes the wire material resulting in a plasma that expands into the liquid medium. This high temperature plasma gradually cools due to the interaction with liquid resulting in a vapour of the wire material that condense uniformly in liquid media to form nanoparticles.

II. WIRE EXPLOSION METHOD

Exploding Wire Method (also known as EWM) is a high energy density process by which a rising current is applied to a thin electrically conductive wire. The heat vaporizes the wire, and an electric arc over that vapor creates a shockwave and explosion. Exploding Wire Method is best known to be used as a detonator in nuclear munitions, high intensity light source, and production method for metal nanoparticles.

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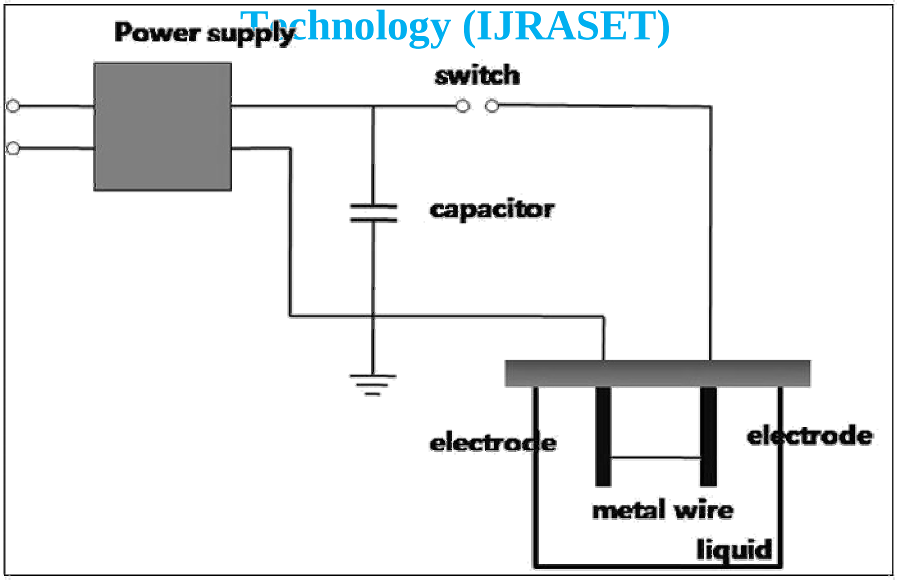

Fig. 1 Wire explosion method- Basic circuit

The basic components needed for the exploding wire method are a thin conductive wire and a capacitor. The wire is typically gold, aluminum, iron or platinum, and is usually less than 0.5mm in diameter. The capacitor has an energy consumption of about 25kWh/kg and discharges a pulse of charge density 104 - 106 A/mm^2, leading to temperatures up to 100,000K. The phenomena occurs over a time period of only 105-108 seconds. Wire explosion in liquid media is the method employed for nano material production. It is a top down approach that melts, evaporates , ionises the wire resulting in the production of plasma that expands in the liquid medium, later it condenses in the liquid to form nanoparticles. A non-oxide metal powder is produced which can be kept to the final stages of application. The process is as follows :

A. Rising Current, Supplied by the Capacitor, is Carried Across the Wire

1) The current heats up the wire through ohmic heating until the metal begins to melt. The metal melts to form a broken series of imperfect spheres called unduloids. The current rises so fast that the liquid metal has no time to move out of the way.
2) The unduloids vaporize. The metal vapor creates a lower resistance path, allowing an even faster current increase.
3) An electric arc is formed, which turns the vapor into plasma. A bright ash of light is also produced.
4) The plasma is allowed to expand freely, creating a shock wave.
5) Electromagnetic radiation is released in tandem with the shock wave.
6) The shock wave pushes liquid, gaseous and plasmatic metal outwards, breaking the circuit and ending the process.

B. Calculation of Energy to be Deposited for Wire Explosion

For a copper wire of 2.5 cm length and diameter of 0.08 mm ,

Total energy required for evaporation = Energy required to melt the wire + Latent heat of fusion + Latent heat of vaporization + Energy required to evaporate the wire

$E = mcs[tm-tr] + mlf + mcs[tb-tm] + mle$

Specic heat capacity of Copper (cs) = 385 J/Kg / degree Celsius at 25 degree Celsius.

Melting point of copper (tm) =1357.77 K

Boiling point of copper (tb) =2835 K

Room temperature (tr) =293K

Latent Heat of fusion of copper (lf) =207000 J/kg

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Latent Heat of vaporization of copper (le) =5069000J/kg
Density of copper = 8920 kg/m
Mass (m)=density*volume.
Volume $=1.256*10^{-10}$ m^3.
Mass (m) = $1.120*10^{-6}$ kg.
So Total energy (E) = 7.005 J.
Energy stored in capacitor = 0.5 CV ^{2}.
Available capacitor is of 50000pF.
0.5 CV 2= 7.005 J
So V= 16.739 kV.

For converting the wire into plasma state about 6 times the evaporation energy is passed. The amount of energy varies with the properties of the material used, the medium used and the atmospheric conditions. In this case the voltage was in about 70kV to 80kV range. This was done by trial and error method.

C. Setup for High Impulse Voltage Generation

An impulse generator is an electrical apparatus which produces very short high voltage or high-current surges. Such devices can be classied into two types: impulse voltage generators and impulse current generators. High impulse voltages are used to test the strength of electric power equipment against lightning and switching surges. Also, steep-front impulse voltages are sometimes used in nuclear physics experiments. High impulse currents are needed not only for tests on equipment such as lightning arresters and fuses but also for many other technical applications such as lasers, thermonuclear fusion, and plasma devices.

An example of impulse generator is the Marx generator. It consists of multiple capacitors that are first charged in parallel through charging resistors by a high-voltage, direct-current source and then connected in series and discharged through a test object by a simultaneous spark-over of the spark gaps. The impulse current generator comprises many capacitors that are also charged in parallel by a high-voltage, low-current, direct-current source, but it is discharged in parallel through resistances, inductances, and a test object by a spark gap.

The main components used for high voltage impulse generation are

1) Transformer
2) High voltage silicon diode rectifier
3) Capacitor
4) Resistor
5) Trigatron switch
6) Sphere gap

The experiment is carried out in a chamber made of PVC material having 200 mm diameter and 150 mm height. It has two electrodes fixed on an insulating wood and separated by a distance of 25 mm. A wire of diameter 0.04 mm is tied across the two electrodes. Once the wire is attached, the chamber is closed with the top cover such that the wire is dipped in distilled water. After each explosion, a new wire is tied between the electrodes. When an appreciable amount of energy is passed through the wire it turns into a plasma state.

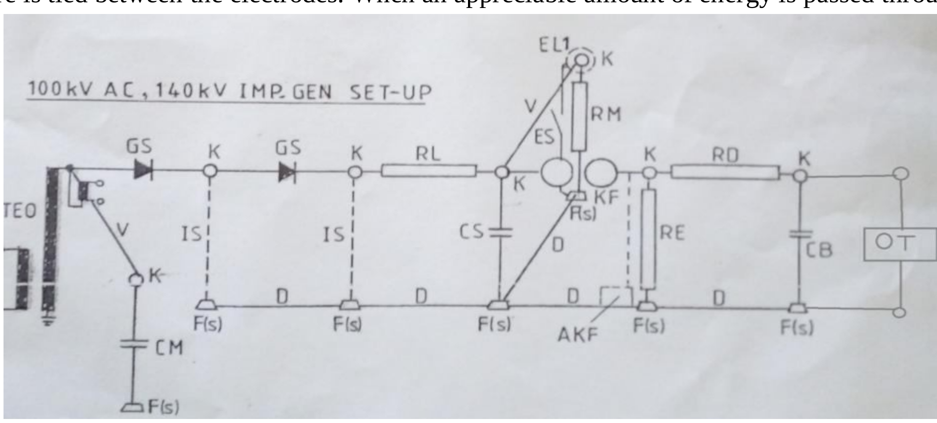

Fig. 2 Connection Diagram

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The ac supply is given to the primary of the transformer. The input voltage can be varied for changing the output voltage. The output is given to the rectifier and its output is used to charge the capacitor. When the capacitor voltage exceeds the sphere gap voltage or the break down voltage the high voltage in the range of kV reaches the wire connected between the wires and causing evaporation of the wire.

III. EXPERIMENTAL SETUP

The experimental setup for the electric wire explosion process designed for liquid media is shown in figure.The experiment is carried out in a cylinder of 200 mm diameter and 150 mm height and is made up of PVC material.The cylinder is partially filled with distilled water .The electrodes are fixed on an insulated wooden structure with a gap distance of 25 mm. The wire of diameter 0.04 mm diameter is connected across the fixed electrodes and can be rewired after every explosion.The top wooden support is used for additional support. Once the wire is connected the chamber is closed with the wooden structure such that the wire is dipped in distilled water.

Fig. 3 Experimental Setup

A capacitor of 50000 pF charged to 40 kV and stored electrical energy was transferred to the aluminium wire in the form of impulse. The wire is connected to the capacitor through a trigatron switch. When the triggering occurs ionization between the sphere gap happens and the stored energy is passed through the wire. It takes about 0.5s for triggering action normally. After several shots of the explosion(about 40 to 50 shots) , the colour of the distilled water changes to greyish black colour indicating the presence of nanoparticles.

Once the experiment is completed the distilled water is collected in a bottle and the sub-micron particles are allowed to settle down.The sedimentation process can be performed using a centrifugal seperator. Once seperated the particle are characterized using SEM,TEM and XRD analysis.

A. Characterization

Novel effects can occur in materials when structures are formed with sizes comparable to any one of many possible length scales, such as the de Broglie wavelength of electrons, or the optical wavelengths of high energy photons. In these cases quantum mechanical effects can dominate material properties. One example is quantum confinement where the electronic properties of solids are altered with great reductions in particle size. The optical properties of nanoparticles, e.g. fluorescence, also become a function of the particle diameter. This effect does not come into play by going from macroscopic to micrometre dimensions, but becomes pronounced when the nanometre scale is reached. The size and properties of the particles obtained are characterized by performing the SEM, TEM, and XRD analysis.

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, in wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperatures . The most common SEM mode is detection of secondary electrons emitted by atoms excited by the electron beam. The number of secondary electrons that can be detected depends, among other things, on the angle at which beam meets surface of specimen, i.e. on specimen topography. By scanning the sample and collecting the secondary electrons that are emitted using a special detector, an image displaying the topography of the surface is created. The types of signals produced by an SEM include secondary electrons (SE), reflected or back-scattered electrons (BSE), photons of characteristic X-rays and light (cathodoluminescence) (CL), absorbed current (specimen current) and transmitted electrons. Secondary electron detectors are standard equipment in all SEMs, but it is rare that a single machine would have detectors for all other possible signals

Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it passes through it. An image is formed from the interaction of the electrons transmitted through the specimen; the image is magnified and focused onto an imaging device, such as a fluorescent screen, on a layer of photographic film, or to be detected by a sensor such as a CCD camera. TEMs are capable of imaging at a significantly higher resolution than light microscopes, owing to the small de Broglie wavelength of electrons. This enables the instrument's user to examine fine detail even as small as a single column of atoms, which is thousands of times smaller than the smallest resolvable object in a light microscope. TEM forms a major analysis method in a range of scientific fields, in physical, chemical and biological sciences. TEMs find application in cancer research, virology, materials science as well as pollution, nanotechnology, and semiconductor research. TEM is a technique used for analyzing the morphology, crystallographic structure, and composition of a specimen. TEM provides a much higher spatial resolution than a scanning electron microscope (SEM), and can facilitate the analysis of features nearly at atomic scale (in the range of a few nano-meters) using electron beam energies in the range of 60-350 keV. In the present study, TEM model no. CM-12 (with EDAX attached) is used. The particle size measurements were performed through the TEM studies

X-ray crystallography is a useful method used for identifying the atomic and molecular structure of a crystal, in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. By measuring the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional picture of the density of electrons within the crystal. From this electron density, the mean positions of the atoms in the crystal can be determined, as well as their chemical bonds, their disorder and various other information.

IV. RESULTS

The wire installed between the electrodes was turned into particles in every discharge. However, we cannot say that all the wire was turned into nanosized particles. Figure 3 shows that a large number of submicron-sized particles exist in the produced Copper powders, even though discharge energy is 6 times larger than the vaporization energy of the wire (about 70kV to 80kV).

Fig. 4 Before and After

The existence of submicron sized particles has been attributed to the very high vapor pressure during the explosion. Under the high vapor pressure, the wire could not totally be vaporized even though the energy deposition exceeded the vaporization energy of the wires because additional energy would be required to vaporize the inner part of the wire. Thus, a little of the inner part of the wire still remained in a liquid state, finally the non-vaporized part was disintegrated into submicronsized liquid droplets, resulting in the formation of submicron-sized particles. The submicron-sized particles naturally settle down. The speed of sedimentation can be accelerated by using centrifugal separator.

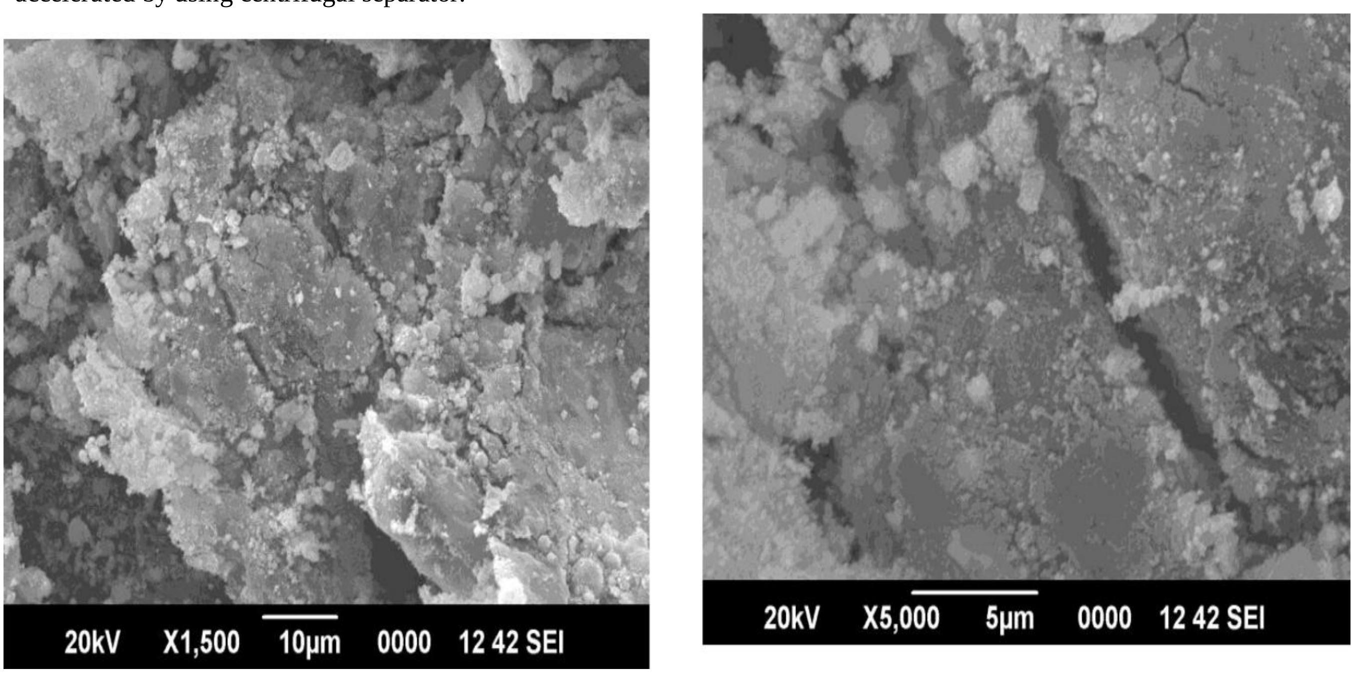

Fig. 5 SEM Result for different magnification of the sample

The change in colour of media after 40 to 50 shots. The change in colour indicates the presence of nanoparticles. The colour of the

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distilled water before the wire explosion is shown in first figure . After each explosion the colour of the medium started to change and after 60 explosions the colour become greyish black colour. This is an pure indication of the presence of nano particles.

Figure 5 shows SEM images of the particles collected at different magnification. This figure 6 shows the presence of nanomaterials. The obtained size of the particles is in the range of 100nm to 200nm. The particle distribution under different scales are also shown. The SEM analysis gives the particle size distribution at different magnications as shown in figure.

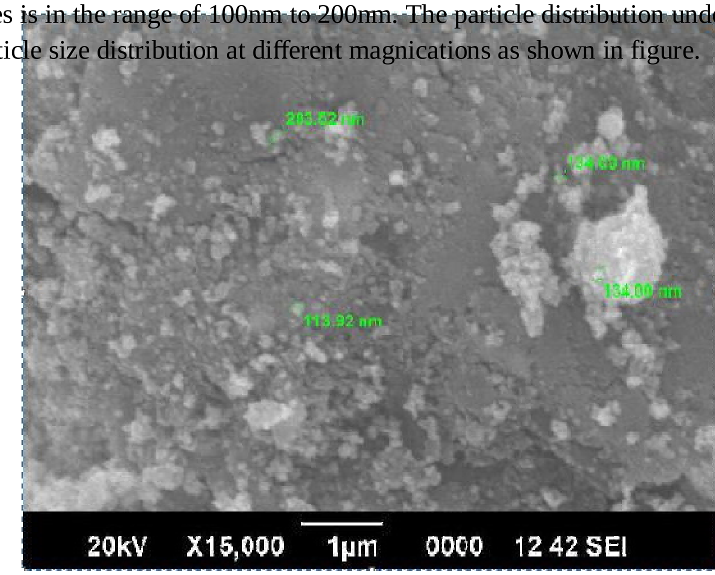

Fig. 6 SEM Result of the sample showing the presence of nanomaterials.

Figure 7 shows the XRD result of the exploded sample. It gives the details about the grain size and particle distribution of the material. It is a useful analysis in determining the particle size distribution of the material and also useful in identifying the material

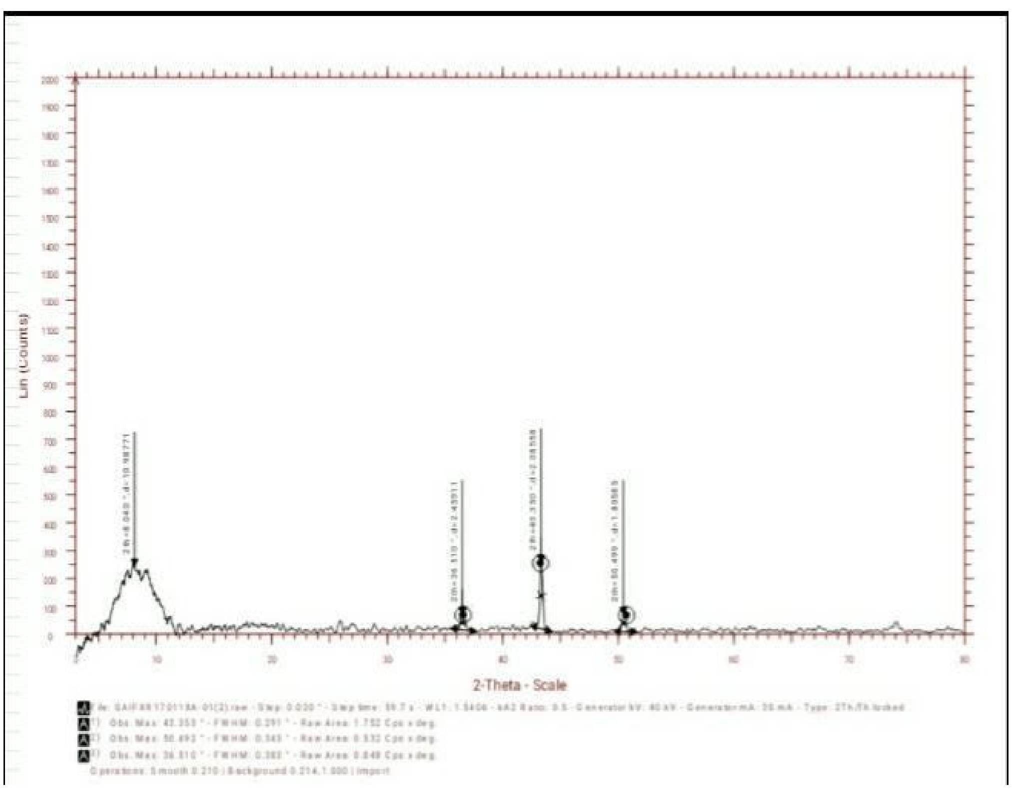

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Fig. 7 XRD result of the sample

From the XRD images obtained it is clear that the peaks in the graph is corresponding to copper. This confirms the prescence of copper in the given test sample

V. CONCLUSIONS

Successfully exploded copper wires of .08mm in diameter and 25mm in length in distilled water at a voltage in the range of 70kV to 80kV.,which is the voltage corresponding to 6 times the vapourization energy, to produce copper powders. The copper powder showed a wide size distribution. The existences of large numbers of submicron sized particles were attributed to unvaporized metal droplets. The powder was classied by diameter and was gathered by using a centrifugal separator. The size of nanopowder observed under SEM analysis was between 100 to 200 nm.

The advantage of the pulse power generation is that particle size could be controlled by varying the injected power and the method has high energy eciency and high product purity.

Main limitation is the high initial cost of pulse power experimental setup. If all the educational and research institutes in India, that have pulse power experimental setup starts their own production unit, then we can easily overcome this problem.

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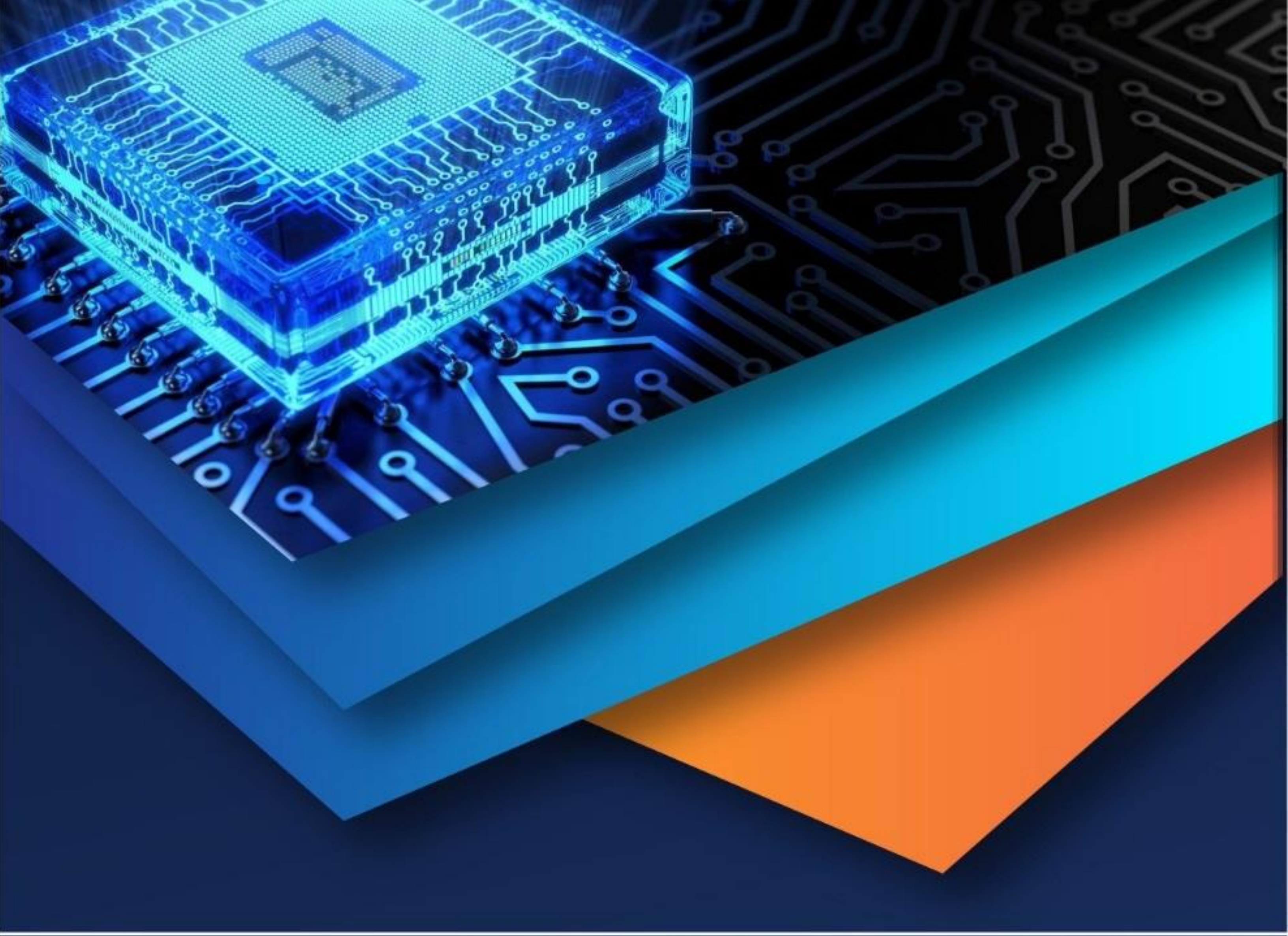

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