Chapter 40

Synthesis of Metal Nano-Powder By Electrical Explosion of Wire

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Metal Nano-powder due to their antimicrobial properties, are very much useful in medical sciences. Its high specific area makes it more useful for societal benefit in the form of composite material, lubricants and sorbents etc. Production of metal Nano-powder in large amount is possible by exploding wire technique, which is high energy density process.

40.1. Introduction

The word 'Nano-science' and 'Nano-technology' is very fascinating in scientific community due to their potential to deliver something amazing which can't be achieved by bulk size material.

'Nano' is basically the dimension range of the order of a billionth of a meter. Although the word 'nanotechnology' is relatively modern but the existence of nanostructures on earth is as long as life itself [1].

In modern world, it became more prominent when physicist Richard Feynman delivered a talk, entitled "There is Plenty of Room at the Bottom" in a meeting of American Physical Society at the California Institute of technology on December 29, 1959. In his talk, he described the manipulation of individual atoms to make small structures having very different properties. Nanostructures may exist naturally and due to development of advance instruments; it can be fabricated easily of desired size in the laboratory also. Generally, the range 1 to 100 nm [1, 2] is described as nanoscale level. Below this, there is atomic scale (0.1 nm) followed by nuclear scale (10^{-15} m).

Physical properties depend on the size range of measurement because various microscopic details average out when measurement is in the range of bulk size. The properties of material associated with the critical or characteristic length, so when dimension of material is reduced to the comparable of characteristic length, fundamental of physics and chemistry changes. For example, the conduction of electrons in solid depends upon the scattering length (the average length travelled by electron before the next collision by impurities and vibrating lattice.), when size of material becomes comparable to it then change in conduction behaviour of material may be observed. This is why physical properties like; resistivity, magnetization and dielectric constant etc. in Nano materials may be different from its bulk material. Nano powder is an agglomeration of ultrafine Nano size particle [4] in the range of 1-100 nm. Metal Nano powder, due to its high specific area [2], is very much useful in production of composite, lubricants and sorbents etc. [9]. Nanotechnology will be boosting in future also due to its vast applications in modern world.

40.2. Methods of Synthesis

Several methods have been developed for the generation of nanoparticles due to its vast applications. Synthesis of metal Nano-powder material is broadly categorised into two methods, first one is top down approach and second one is bottom up approach [5]. In first method bulk material is utilised and particle size is reduced to Nano size by different physical, chemical and mechanical processes. In bottom up method, atoms or molecules are the starting material for generation of nanoparticles. Mechanical ball milling, RF plasma, Laser ablation, sputtering and electrical explosion of wire (EEW) are some examples of top down method whereas physical vapour deposition, chemical vapour deposition, sol gel method and chemical reduction are examples of bottom up method. These above mentioned methods have their own advantages and disadvantages. Here we shall discuss some methods to generate Nano materials.

40.2.1. Ball Milling

This is a physical method to generate Nano particles. In this method sample powder is feed in hollow rotating cylinder containing hard balls of stainless steel or ceramic. These balls move through the surface of cylinder containing sample powder and transfer high mechanical energy to produce Nano powder. This method is very simple to install and cost effective but there are chances of contamination of Nano powder due to balls.

40.2.2. RF Plasma

In this method, bulk metal is placed in an evacuated chamber. This evacuated chamber is wrapped with RF heating coils. Metal is heated above its evaporation point by using these high voltage RF coils. Now precursor gas generally. He or Ar is passed inside the chamber to produce high temperature plasma. The metal powder that nucleated, diffuses quickly to colder region and collected by electrostatic collector.

40.2.3. LASER Ablation

In this method, high intensity laser beam is irradiated on bulk metal rod and if its fluence is more than so called threshold fluence for ablation then bulk material fragmented in the form of Nano particles. Pulse duration of laser, its fluence and energy determines the amount of particle produced in this method.

40.2.4. Electrical Explosion of Wire (EEW)

Electrical explosion of wire is century old observed phenomenon. Though it was interesting but not considered worthy to pay enough attention until world war-II. After knowing its potential utilisation in detonation for low density explosion, missile and space technology [11], metallic Nano-powder generation, controlled shock wave generation, study the dynamics of dense plasma etc; it drew attentions of many scientists and technologists to explore it in depth. It has been reported that if a very high current (~kA) flows in a thin metallic wire for very short duration (~μs) then due to transient responses of resistivity of metal, a significant amount of energy can be pumped into it, which emancipated with the explosion of wire.

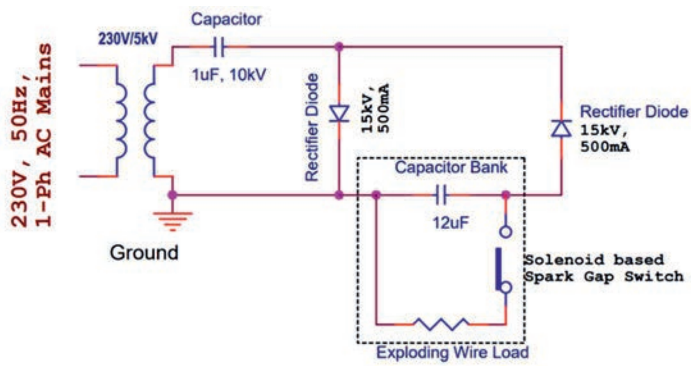

Figure 40.1. Schematic Circuit Diagram of Nano particle generator.

Energy storage capacitor is generally used for delivering such a high current [7]. Since rate effect is important aspect in EEW, hence current delivering circuit must have low inductance [10]. In EEW, resistivity of metal is a function of action integral [12] and can be expressed as following equation:

$$g = \int J^2 dt \tag{40.1}$$

Where, 'g' is action integral and 'J' is current density applied to wire. In our case as shown in Figure 40.1, 12 µF capacitor is used to store the energy. It can be charged up to 10 kV by using 5 kVA transformer and CW voltage multiplier. Wire is placed as a load separated by a solenoid actuated spark gap switch. When capacitor is fully charged (here we have charged it up to 8.6 kV voltage only to avoid unnecessary stress on capacitor) then solenoid is moved with 12 V DC power supply to trigger the spark gap switch, eventually high current passing through 0.5 mm diameter and 40 mm long Cu wire. Current evolution with time is shown in Figure 40.2.

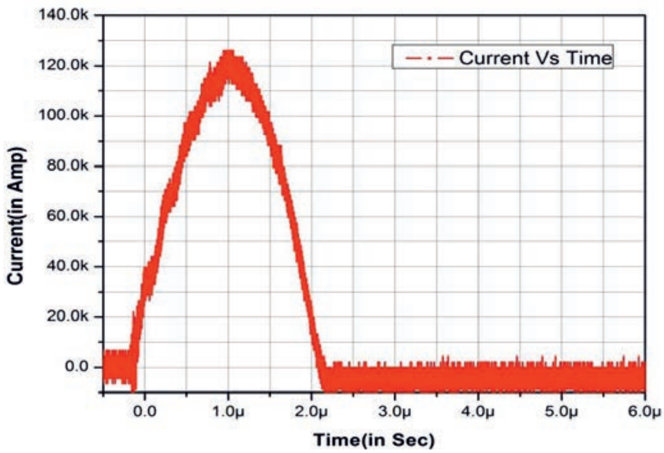

Figure 40.2. Variation of Current with time for Cu.

During the rise of current, metallic wire goes into various phases before explosion occurs. Initially, due to rise in current Ohmic heating starts in metal and if energy storage device has sufficient energy then it starts melting of wire, where metal exists in both solid and liquid phases before completely entering into the liquid phase. Current rising is considerably fast such that due to absence of nucleation site, it has been reported that liquid gets superheated and maintains its cylindrical shape due to inertia with few undulations on surfaces [10, 12]. The ever increasing current starts vaporising of superheated liquid. During vaporisation period, sudden increase of metal resistivity observed due to decrease in diameter of wire; this is known as 'burst'. Since energy density of wire is integral of action with resistivity, hence energy density of wire is very high during this action. During the vapour phase, abrupt rise in resistivity terminates the rise in current with very high EMF voltage. Initially due to high density of vapour and miniscule presence of electrons and ions, probability of avalanche occurrence is very low but in subsequent time due to expansion of metal vapour avalanche takes place and arc discharge happens. To synthesis pure metal Nano powder by EEW, explosion was performed in close chamber, pressurised with inert gas to avoid any contamination (in our case Ar gas at 2 atm pressure) [3]. When expansion of metal vapour occurs at very high speed then it gets scattered with surrounding gas molecules and rapidly condensed on substrate to form metal Nano powder. It has been reported [7] that in very beginning of expansion, condensation occurs at maximum rate and with decrease in ambient gas pressure expansion accelerates too. Hence particle size growth can be controlled with change in ambient gas pressure.

Now the question is how to determine the wire length and its diameter for given amount of capacitor energy to generate metal Nano powder!

Let us consider in our case; C = 12 µF, V = 8.6 kV, Hence stored energy of capacitor = $0.5 \times C \times V^2$ = 443.76 J. Now, diameter of wire D = 0.5 mm and length l= 40 mm. Hence volume of wire = $\pi \times D^2 \times l \times 0.25 = 7.85 \times 10^{-3}$ CC. Density of copper = 8.96 gm/CC. Hence mass of Copper to be exploded = 8.96 × 7.85 = 70.33 mg.

It has been mentioned [12], energy density e = 5909 J/gm and specific action g = 173000 A^2Sec/mm^4 to reach the Cu wire to burst phase. Specific action calculated from Figure 40.2 is 186943 A^2Sec/mm^4 and energy required to explode the wire = 5909 J/gm × 70.33 mg = 415.6 J.

From the above calculation we can conclude that our wire diameter and length are well optimized for the given capacitor energy.

40.3. Characterization

To know the crystal structure, grain size and average particle size of nanomaterials, their characterization is important. Generally, X-ray diffraction (XRD) and electron microscopy are important characterization tools for nanomaterials. XRD is very important tool to determine the crystallinity, crystal structures and lattice constants.

Since, X-ray wavelength is equivalent to interatomic distances hence X-ray beam scattered coherently to form a diffraction pattern. This X-ray diffraction is governed by Bragg's law,

$$n\lambda = 2d\sin\theta \tag{40.2}$$

Where 'd' is the spacing between planes.

Intensity of measured diffracted X-ray is function of diffracted angles and sample orientation. Width of Bragg's peak is combination of grain size, microcrystalline Strain and instrumental broadening effects. If instrumental broadening is corrected and strain components sort out then crystallite size can be determined by using Scherer's method. The main disadvantage of XRD is that it is not suitable for Low Z materials where neutron and electron diffraction is most suitable tool.

Electron microscopy is imaging technique where electron beams are used to scan sample surface to know its morphology and topography. Electromagnetic lenses are used as objective and magnifying lens to focus and magnify the electrons beam respectively. Since electron de Broglie wavelength is small hence it is capable to give better spatial resolution than optical microscopy. Scanning electron microscopy (SEM) utilises the secondary electrons and back scattered electrons signal to produce image. In Transmission electron microscope (TEM), electrons beam incident on ultra-thin sample and focused by objective lens. Transmitted electrons magnified by magnifying lens and focussed on imaging devices like florescent screen or CCD cameras to form images. Electron microscopes can achieve resolution better than 1 nanometre. In XRD, information acquired is an average over a large amount of material whereas in electron microscopy, few thousands or lakhs atoms are involved in information acquisition.

40.4. Summary

Materials having dimensions 1 to 100 nm are classified as Nano materials. Since this dimension is in order of characteristic or critical length of various physical properties of material hence fundamental of physics changes for Nano scale material. Metal Nano powder due to its various applications in medical sciences [6, 8], electronic industry, and lubricant and catalyst industry, is very much important to produce. There is top down and bottom up approach to produce metal Nano-powder. Electrical explosion of wire (EEW) is top bottom approach which can produce metal Nano-powder by supplying electrical energy to wire in presence of Argon (inert gas) media.

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Questionnaire

1. Why Nano Scale is so important and nanomaterial’s physical properties differ from their bulk?
2. What is quantum well, quantum wire and quantum dot?
3. What is superheating of a liquid?
4. What is top down and bottom up approach to synthesis Nano-powder? Explain them with examples.

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