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Synthesis and Characterization of Copper Nanoparticles, Using Combination of Two Different Sizes of Balls in Wet Ball Milling

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Abstract

The applications of nanoparticles are in biological Nano sensors, optoelectronics, Nano devices, Nano electronics, information storage ,catalysis and Nano fluids which differ significantly from size to size of nanoparticles. But controlling the size and preparation in bulk of nanoparticles eco-friendly is interesting.

This paper reports on preparation of Cu nanoparticles by wet milling using a planetary ball mill, taking the combination of two different sizes of balls. The milled powder is characterized using X-ray diffraction (XRD) and particles size analyzer. Samples have taken at 0, 18, 24, 30 and 40 hours after milling the powder. The crystal sizes of milled powder for different milling times are characterized. By increasing the milling time crystal size decreases. After 40 hours average crystal size of milled powder is 21nm.

Keywords: *Copper Nanoparticles, Wet Ball Milling, Combination of Balls*

1. Introduction

Nano materials can be defined as those materials which have structured components less than 100nm at least in one dimension [1]. One of the most important application of copper nanoparticles is in Nano fluid due to its higher thermal conductivity in comparison to other materials. For synthesis of nanoparticlesthere are two approaches, namely, 'Top-down' and 'Bottom-up', available [1]. Top down approach involves breaking of bulk solid into smaller portions. It includes physical methods like attrition or milling, repeated quenching [1]. Bottom-up approach involves condensation of atoms or molecular entities to build up a nanomaterial [1]. It includes chemical methods like reduction of metal salts [1]. But Top-down process is eco-friendly and in Top-down process, Ball Milling is economically in research point of view. The ball-milling is generally used as a mechanical co-grinding of powders, initially different in nature, up to the preparation of a new powder, homogeneous in composition [2]. The milling is done in cylindrical containers called vials and containing balls. The nature of the milling tools can be as diverse as steel, agate, tungsten carbide etc. [2]. The vials are generally filled under an inert atmosphere to avoid side reactions, since the particles are fractured during themilling process and, therefore, new highly reactive surfaces can react with the surrounding gases [2].

Jorge E et al. [3] have prepared iron (Fe) nanoparticles, taking initial size of average 1-10 µm using vibratory mill (SPEX 8000 mixer), ball size 25.4mm, ball to powder ratio 10:1, ethanol added to 0.05 mL/g of iron powder to prevent the oxidations of particles, at 1800 rpm after 30 hours of milling, final sizes got in the range of 2-4 nm. **Mohammad Baghar Rahaei et al.** [4] have synthesized

nanoparticles, taking initial powders of Ti (5 µm) and C (50 µm) using Retsch-planetary ball mill PM 400, ball sizes 20 mm (4 ball) and 14 mm (3 ball), ball to powder ratio 10:1, Stearic acid 2wt% to prevent oxidations of particles, at 300 rpm after 8 hours of milling, average size was 103 nm and after 16 hours of milling average size 16.5nm.**O.K. Tan et al.** [5] have synthesized nanoparticles, taking initial powders of ZrO_2 (325 mesh) and Fe_2O_3 (<5 µm) using planetary ball mill (Fritsch Pulverisette-5), ball to powder ratio 20:1, at 200 rpm after 20 hours of milling, average size was <10 nm.**T.P. Yadav and O.N. Srivastava** [6] have prepared cerium (Ce) nanoparticles, taking initial size of average 5 µm using Attritor ball mill, ball to powder ratio 40:1, at 400 rpm after 30 hours of milling, average size was 10 nm.**Musa mutlu Can et al.** [7] have synthesized iron (Fe) nanoparticles, taking initial size in the range of 20-40 µm using S100, RETSCH planetary ball mill, Distilled water as a wet medium, ball to powder ratio 36:1, at 300 rpm after 24 hours of milling average size was 23.5 nm and after 48 hours of milling, average size was 33.2 nm.**G.R Khayati et al.** [8] have synthesized nanoparticles, taking initial mixture of Ag_2O (5-40 µm) and C (10-50 µm) powders, using planetary ball mill, ball to powder ratio 20:1 at 450 rpm after 22 hours of milling, average size was 28 nm. **Satya V. Ravikumar et al.** [9] have prepared copper(Cu) nanoparticles, taking initial size <450 µm, using Fritsch Pulverisette 5/4 planetary ball mill, ball size 10 mm ,ball to powder ratio 10:1, and toluene is used to prevent the oxidations of particles, at 300 rpm after 30 hours of milling, the average size of particles was 34 nm.

Thus literature survey shows that milling parameters are important factors to particles size and economical point of view. So here we have tried to take a best possible milling parameters to synthesize the nanoparticles as smaller size.

2 Experimental Procedure

2.1 Synthesis of copper nanoparticles

Table 1 The specifications for the milling systems.

Mill type	Retsch planetary ball mill PM 100
Milling time	40 hours
Wet milling medium	Toluene Rectified
Milling speed	250 rpm
Grinding media:	
Ball and jar material	Tungsten carbide
Ball size	5 mm and 3mm
Ball weight	540 g
Ball to powder ratio	8:1
Jar dimensions:	
Length	95 mm
Diameter	75 mm

Table 2 Specifications of Cu powder

Material	Cu
Synthesized by	Spectrochem PVT.LTD. Mumbai (india)
Purity	99.7%
M.W	63.55
Density	8900kg/m^3
Size	204 nm
Thermal conductivity	385 w/m-k
Other contents	0.3%

Mechanical milling of the micro sized metal powders was carried out in Retsch Planetary Ball Mill PM-100 with Tungsten carbide vials and balls to prepare ultrafine powders. The starting material (see Table 2) used for milling was elemental Copper particles for preparing ultrafine particles of Copper. Cu ultrafine powder was milled in a vial containing 68.75 gram of powder and 550 gram balls for 40 hours. But samples have taken at 0, 18, 24, 30, and 40 hours. The ball to powder weight ratio was 8:1. Milling was conducted at a speed of 250 rpm in wet medium. About 50 ml of toluene was used to prevent undue oxidation and agglomeration of powder. Tungsten carbide balls of 5 mm and 3 mm diameters in the ratio of 4:1by weight were used for milling. The specifications of the milling systems are given in Table 1.

2.2 Particles Characterization Techniques

2.2.1 X-Ray Diffraction (XRD) Analysis

Since a non-destructive testing technique, X-ray diffraction is a powerful tool for the analysis of crystalline structure [10]. X-ray has wavelengths comparable to the crystalline lattice constants, thus it can be used for the accurate measurement of lattice parameter, crystallite size, lattice strain etc. [10].In the present study crystallite size has been analysed using an X-ray diffractometer (see Table 3). Crystallite size is obtained from the X-ray diffractometer by using scherrer formula as given by the following formulae [10].

Crystallite size, D in A° = $\frac{k\lambda}{\beta \cos\left(\frac{2\theta}{2}\right)}$

Where, λ is the wavelength of X-rays used for this study, i.e., wavelength corresponding to Cu-Kα(1.5406 A°) , 2θ is the diffraction angle for the peak, k is a constant having value 0.9 and β indicates the full width at half maximum(FWHM) obtained by reducing instrument broadening from the FWHM of the sample as shown below.

FWHM,$\beta = \sqrt{(\beta_{sam}^2 - \beta_{sta}^2)}$

Where, β_{sam} is the FWHM value in radian for the particular peak of the sample obtained by fitting the peak using a lorentzian function and $\beta_{sta}$is the FWHM in radian for standard sample(LaB_6) corresponding to same peak position. But in the current project FWHM is directly taken from X'Pert High Score Plus analyzer.

Table 3 Measurement conditions

Diffractometer system	XPert-PRO
Measurement program	Panalytical
Anode material	Cu
K-Alpha wavelength	1.540598 A°
Generator voltage	45 kV
Tube current	30 mA
Scan range	10-80°(2 thetha)
Scan step size	0.016711
Scan type	Continuous
No. of points	4189

2.2.2 Nano Zetasizer

The particle size in nanometre range and dispersion stability of ultrafine particles in nanofluid was measured by Nano zetasizer (Model: Nano ZS, Malvern). The sample was prepared by dispersing small amount of ultrafine particles in base fluid with constant ultra-sonication for 30 minutes. Then the sample was kept in a sample holder with the help of syringe and analysed. The sample was analysed using Zetasizer software.

3. Result and Discussion

3.1 X-Ray Diffraction (XRD) Analysis:

Figure 1 shows the XRD patterns of Cu as a function of milling time. With increasing the milling time the peaks of element decrease in intensity and become broader, suggesting reduction in crystallite size. From initial to final crystal sizes are shown in Table 4.

Table 4 Crystallite size.

Sample	Crystallite Size (nm)
Cu initial powder	204
Cu after 18 hours	54
Cu after 24 hours	38
Cu after 30 hours	29
Cu after 40 hours	21

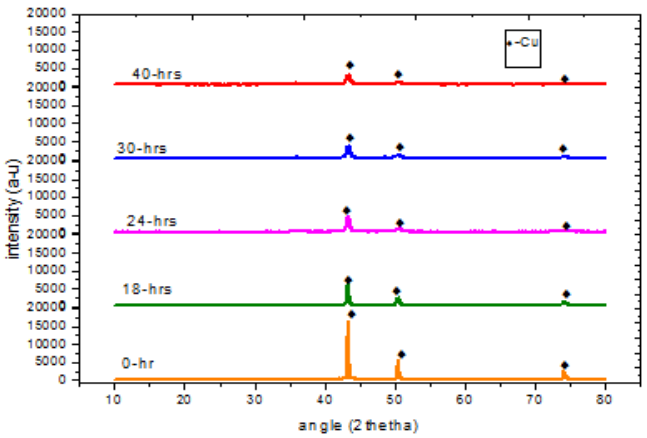

Figure 1XRD pattern of Cu powder as a function of milling time

The Table 4 and Figure 1 show that as the milling time increases up to 40 hrs. , the crystal size of powder decreases due to fracturing of crystal size.

3.2. SEM Analysis:

SEM images show agglomeration of particles. It is due to ultrafine particles agglomerate in the open atmosphere.

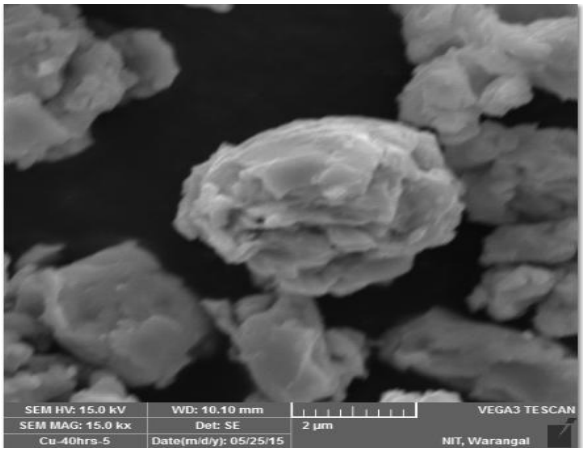

Figure 2 SEM image at 2µmscale

3.3 Nano Zetasizer Analysis

Figure 3 shows the distribution of copper nanoparticles in the fluid (peanut oil) in the average size of 20.40 nm.

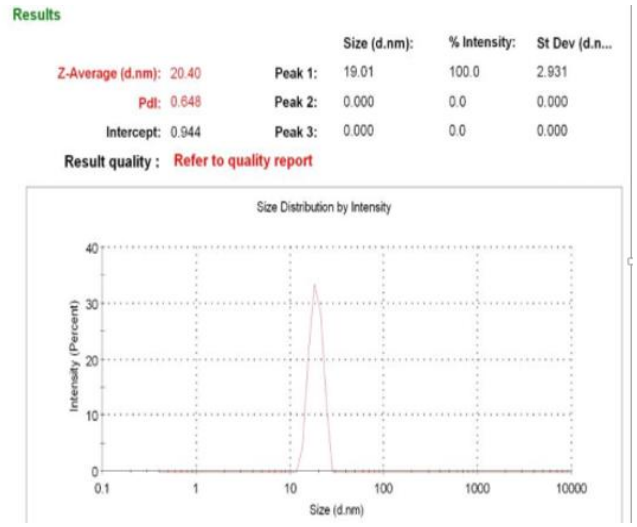

Figure 3 Distribution of copper nanoparticles in the fluid by intensity

Conclusions

Copper nanoparticles are prepared using wet ball milling. After the study of lot of research papers, different parameters like ball size, combination of ball, ball to powder ratio, rpm and medium of milling have optimized in point of view economically and eco-friendly. Some important points regarding the parameters of ball milling are as follows:

If we take size of balls larger, then impact force between the ball and powder will be more but contacting gaps between the balls will also more and thus size of particles after milling will be more. So ball size should be optimized.

If we take equal size of balls then contact gaps will be more but if we take two different sizes of balls then contacting gaps will be less and thus particles size of powder will get smaller. So it is suggested to take combination of balls.

At high ball to powder ratio, the mean free path of the grinding balls decreases and number of collisions per unit time increases. Consequently more energy is transferred to the powder particles resulting faster milling but it may be chance to cold welding of particles due to raise in temperature. So ball to powder ratio should be also optimized.

To reduce the cold welding, we have to use process control agent. This may solid, liquidor gaseous. If this is liquid then milling is called wet milling. The surface active agent adsorbed on particle surface interface with cold welding and lower the surface tension of the material. Thus a reduction in the surface energy results in the use of shorter time to obtain a particular particle size.

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