EXPERIMENT NO 5

Particle size analysis

Objective:

To identify phases present and study the microstructural features of synthesized nano and milled ceramic powders.

Basic Theory:

- The success of any powder metallurgical process depends to a great extent on the complete characterization and control of the metal powders.
- The method of powder production influences particle chemistry and structure, apart from the precise nature of particle size distribution.
- These properties also influence the behavior of the powder during compaction and sintering, and the composition, structure and properties of the sintered material.
- The microstructure of the crystalline powder has a significant influence on the behavior of powder during compaction and sintering and on the properties of the final product.
- Fine grain size is always desirable, as it improves the mechanical properties apart from the sinter abilityt and the uniformity of dimensional changes.
- The grain size can be dependent on the powder particle size. The particular powder production method, e.g., rapidly cooled powder, would naturally give rise to small particles and also small grain sizes.
- It can be thus concluded that the particle size analysis is necessary for a complete characterization instead of an average value or even maximum or minimum values of size.
- There are a number of particle size measurement techniques available in powder metallurgy, each having their own limitations.
- This table classifies some of the common methods of particle size determination and their limits of applicability.

Mesh analysis

- Mesh analysis, also known as sieve analysis, is a common technique used in powder metallurgy to measure the particle size distribution of a powdered material.
- It involves passing the powder through a series of sieves with different mesh sizes , and then measuring the amount of powder that passes through each sieve.
- The following steps are involved in performing mesh analysis:
    1. Sample Preparation: A representative sample of the powder is prepared. The sample size should be large enough to be representative of the entire batch of powder, but not so large that it is difficult to handle.
    2. Sieve Stack: A stack of sieves is assembled, with the coarsest sieve at the top and the finest sieve at the bottom. The mesh size of each sieve decreases from top to bottom.

3. Sieving: The powder sample is placed on the top sieve, and the stack of sieves is shaken or agitated for a specified period of time. This allows the smaller particles of powder to pass through the sieves, while the larger particles are retained on the sieves.
4. Weighing: Once the sieving is complete, each sieve is weighed to determine the amount of powder that was retained on it. This information is then used to calculate the percentage of powder that falls within each particle size range.

X-ray diffraction (XRD) analysis

- The X-ray analysis is used for estimating crystallite size of nanomaterials and therefore it is extensively employed in the present work.
- Diffraction line broadening for crystalline materials has several causes, including strain and small crystal size.
- The destructive interference from the crystal is not complete at angles slightly off Bragg condition

$$\lambda = 2d \sin\theta$$

where λ is the x-ray wavelength, d is the interplanar spacing and θ is the diffraction angle.

- The width of the diffraction peak increases as the thickness of the diffracting crystal decreases.
- The most useful approach to particle size analysis using x-rays relies on the peak width at half of the maximum intensity as shown in this figure 1.
- Peak broadening at that intensity depends, in part, on the number of diffracting planes in the crystal.
- The Scherrer formula gives the crystal size *D* in terms of the broadening *B*, diffraction angle and x-ray wavelength as

$$D = \frac{0.9\lambda}{B\cos\theta}$$

The wider the diffraction peak, the smaller is the particle size.

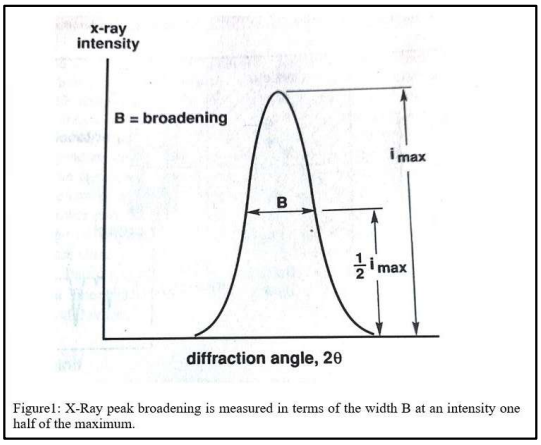


Figure1: X-Ray peak broadening is measured in terms of the width B at an intensity one half of the maximum.

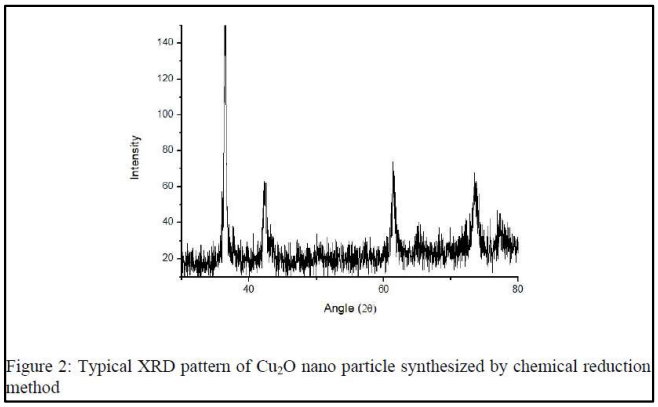


Figure 2: Typical XRD pattern of Cu_2O nano particle synthesized by chemical reduction method

Table1: The FWHM values of XRD pattern of Cu_2O nano particle

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
23.480850	14.143000	0.492000	3.78880	11.65
36.446790	121.376000	0.295200	2.46524	100.00
42.458020	39.839960	0.246000	2.12909	32.82
61.317050	43.526470	0.246000	1.51188	35.86
73.574810	30.597110	0.492000	1.28737	25.21
77.350730	10.451600	0.787200	1.23368	8.61

Electron microscopy analysis

- A widely applied technique for particle sizing uses the ability of the eye to rapidly size dispersed particles in a microscope.
- Microscopy is reasonably accurate; the tedium of sizing statistically significant quantities of particles has led to use of authentic image analyzers.
- The image for analysis is generated by optical, scanning and transmission electron microscopes.
- The instrument choice depends on the particle size; however, the larger depth of field in the SEM is a distinct advantage, especially since it shows surface topography and can provide x-rays for compositional analysis.
- The shape, size, morphology, distribution and microstructure of the powders are examined using a field emission scanning electron microscope (FESEM, Figure 3) coupled with energy dispersive X-ray (EDX) microanalyzer.
- The SEM is operated at an acceleration voltage of 20 kV. The micrograph images were taken in the secondary (SE) and back scattered (BSE) modes as per the requirements.

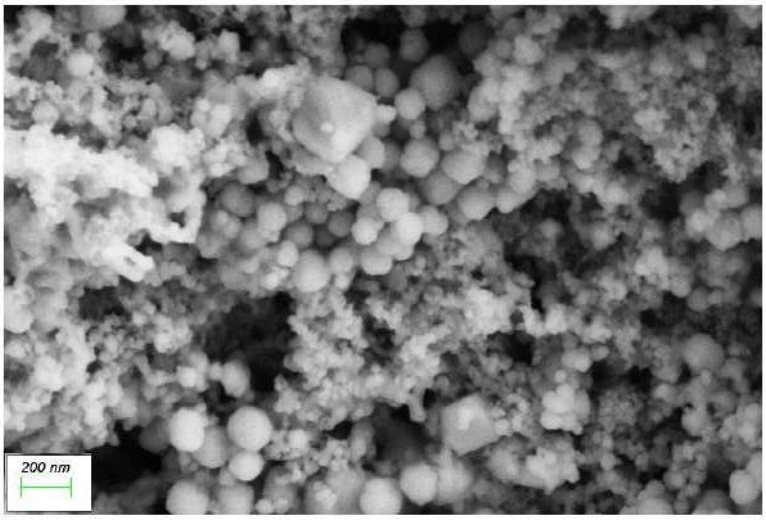


Figure 3: Typical FESEM image of the Cu_2O nano particles

Equipment/ Raw Materials:

- chemically synthesized Cu_2O nano particles:
- X-Ray Diffractometer (Panalytical X'Pert PRO)
- Field emission scanning electron microscope
- Pin Stubs
- Carbon tape

Procedure:

Powder sample preparation for scanning electron microscope:

1. Properly clean the pin stub sample holder using isopropyl alcohol or ethanol

2. Place a carbon tape on top of the pin stub

3. Spray very small amount powder gently on the carbon tape and ensure no agglomeration.

Lab deliverables

1. List and briefly write about different particle size analysis techniques.

Sol:

a) Sieve Analysis:
    - Principle: Sieve analysis involves passing a sample through a series of stacked sieves with progressively smaller openings. Particles are retained on the sieves based on their size.
    - Use: Commonly used for coarse particles, such as aggregates, sand, and granular materials.

b) Sedimentation:

- Principle: Sedimentation methods involve measuring the settling rate of particles in a liquid under the influence of gravity. Stokes' law is used to calculate particle sizes.
- Use: Particularly suited for fine particles and nanoparticles.

c) Electron Microscopy (TEM and SEM):
- Principle: Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) use electron beams to visualize and measure particle sizes and shapes at very high resolution.
- Use: Ideal for detailed analysis of nanoscale particles, including nanoparticles and nanomaterials.

d) X-ray Scattering (SAXS and WAXS):
- Principle: Small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS) techniques utilize X-ray beams to study the scattering pattern from particles, providing information on size and structure.
- Use: Valuable for nanoscale particle analysis and structural characterization.

2. What are the advantages and disadvantages of particle size analysis by microscopy?

Sol:

Advantages:

- Simplest way to organize particle size.
- Shape analysis is also possible.
- It is always to visualize particle.
- Particles can be counted.

Disadvantages:

- Optical microscopy requires long test cycle.
- Depth of particle not accessible.
- It is tedious and slow method.
- Unable to test ultrafine particles.

3. Briefly explain particle size analysis by DLS measurement.

Sol:

- Dynamic Light Scattering (DLS), also known as Photon Correlation Spectroscopy (PCS), is a technique used to measure the size of particles or molecules in a suspension or solution.
- DLS is particularly useful for analyzing particles in the nanometer and submicron size range.
- The Brownian motion of particles in suspension possess laser light to be scattered at different intensities fluctuation in the velocity of Brownian movement and hence particle size using stokes Einstein relationship.

Observations and calculations:

Total weight taken: 49 gm

Sieve Speed: 350 rpm

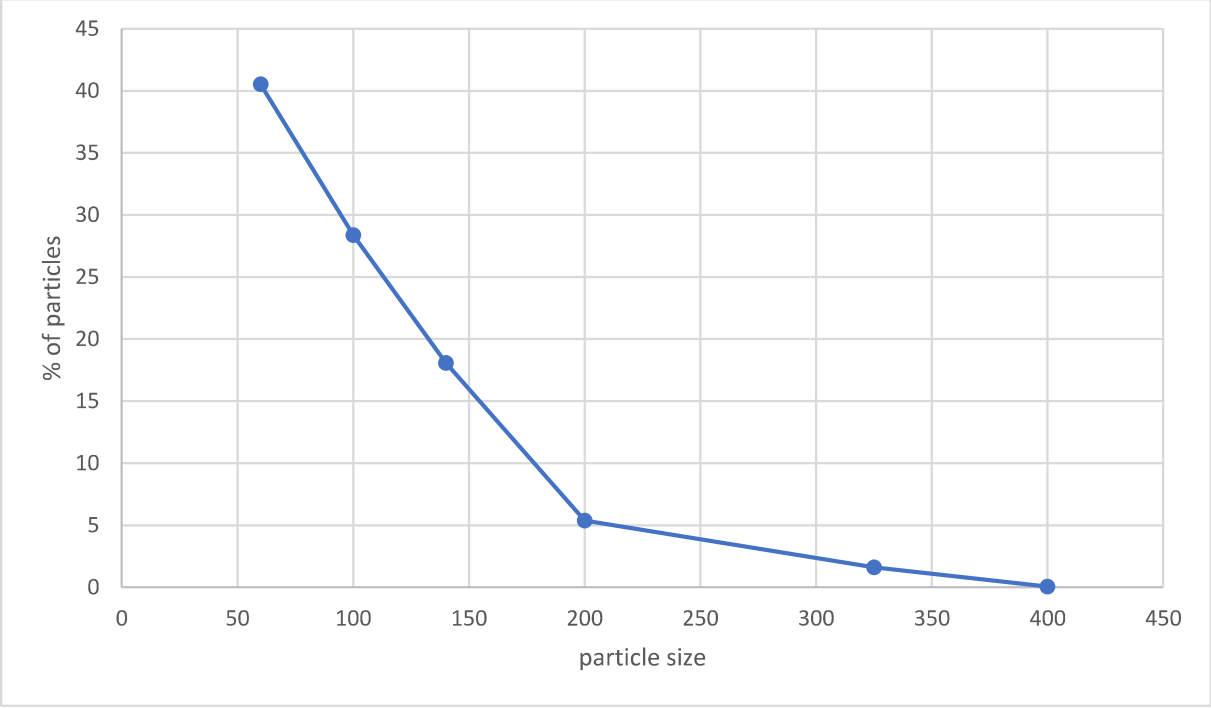
Sieve time: 5mins

Sieve Number	*Mesh Size*	*Mass retained*
1	60	20.26
2	100	14.19
3	140	9.04
4	200	2.69
5	325	0.8
6	400	0.03
		Total: 47.01

Total weight aggregate: 49 – 47.01 = 1.99 gm

The loss of weight might be because fines get stuck in mesh

% of particles vs particle size graph:



Conclusion:

The three techniques for particle size analysis are: mesh analysis, X-ray diffraction, and electron microscopy. Each technique has its own advantages and limitations. While doing particle size analysis using screening technique the mesh sizes used were 60, 100, 140, 200, 325, 400 (openings per inch).