EXPERIMENT NO 4

Characterization of nano and milled powders

Objective:

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

Basic Theory:

- The method of powder production influences particle chemistry and structure, apart from the precise nature of particle size distribution.
- These properties also influence:
  a. the behaviour of the powder during compaction and sintering
  b. the composition, structure and properties of the sintered material.
- The larger depth of field in the scanning electron microscopy (SEM) is a distinct advantage, especially since it shows surface topography and can provide x-rays for compositional analysis.
- Important microstructure information can be gained by SEM analysis.
- Nucleation sites, contamination, cooling rate, grain size, and segregation can be assessed from SEM examinations.

X-ray diffraction (XRD) analysis

- X-ray diffraction (XRD) studies are carried out for the purpose of
  a. phase identification
  b. crystallite size and
  c. lattice strain of the powder samples

- These studies are made by using Panalytical X'Pert PRO diffractometer, operated with Cu-Kα radiation (having wavelength =1.54 Å) on X-ray diffractometer.
  a. The X-ray source is operated with Cu-Kα radiation at a voltage of 40 kV and current of 20 mA.
  b. The diffraction angle was varied in the range of 10-90° and the range of 2θ was selected such that all the major peaks of the expected phases to be present in the powder are covered.
  c. Subsequently, the XRD patterns were analyzed with the help of JCPDS (Joint Committee on the Powder Diffraction Standards) data file to identify the crystal structure of the constituent phases.

Advantages of electron microscope over optical microscope

1. They have a higher resolution and are therefore also able of a higher magnification (up to 2 million times). Light microscopes can show a useful magnification only up to 1000-2000 times.
   - This is a physical limit imposed by the wavelength of the light.
   - Electron microscopes therefore allow for the visualization of structures that would normally be not visible by optical microscopy.
2. Depending on the type of electron microscope, it is possible to view the three- dimensional external shape of an object (Scanning Electron Microscope, SEM).
   - In scanning electron microscopy (SEM), due to the nature of electrons, electron microscopes have a greater depth of field compared to light microscopes. The higher resolution may also give the human eye the subjective impression of a higher depth of field.

Disadvantages of electron microscope

1. They are extremely expensive.
2. Sample preparation is often much more elaborate. It is often necessary to coat the specimen with a very thin layer of metal (such as gold). The metal is able to reflect the electrons.
3. The sample must be completely dry.
4. It is not possible to observe color. Electrons do not possess a color. The image is only black/white. Sometimes the image is colored artificially to give a better visual impression.
5. They require more training and experience in identifying artifacts that may have been introduced during the sample preparation process.
6. The energy of the electron beam is very high. The sample is therefore exposed to high radiation, and therefore not able to live.
7. Maintenance costs are high.

Equipment/ Raw Materials:

- chemically synthesized Cu2O nano particles
- ball milled Al_2O_3 powders
- 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. Discuss the origin of signals and their use in the following modes of operation in SEM:

a. Secondary Electron Imaging

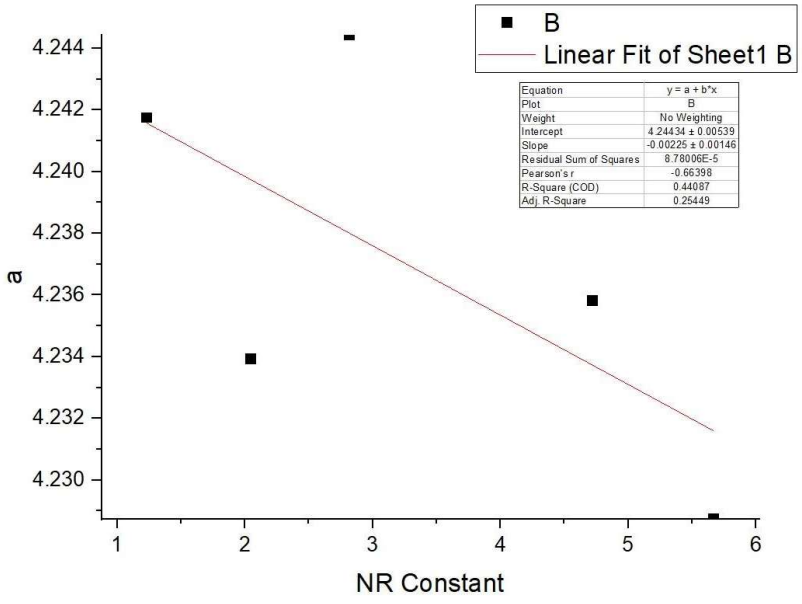
b. Backscattered Electron Imaging

c. Energy Dispersive X-ray analysis

Sol:

Mode of Operation	***Origin of Signal***	***Use***
Secondary Electron Imaging	Secondary electrons	High-resolution images of surface topography
Backscattered Electron Imaging	Backscattered electrons	Images of composition
Energy Dispersive X-ray Analysis	X-rays	Identify and quantify elements

Observations and calculations:



	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
1	theta in radian	theta in degrees	2 theta	h	k	l	sin theta	2 sin theta	d	(h^2+k^2+l^2	a	cos theta	cos^2 theta	cos^2 theta/ sin theta	cos^2 theta/ theta	NR
2	0.32046875	18.3125	36.625	1	1	1	0.315011	0.630023	2.4444	1.73	4.2287	0.949088	0.9007678	2.859476001	2.810781917	5.670258
3	0.37209375	21.2625	42.525	2	0	0	0.363567	0.7271334	2.1179	2	4.2358	0.931568	0.8678193	2.386960247	2.332259695	4.71922
4	0.53703125	30.6875	61.375	2	2	0	0.511587	1.0231748	1.5051	2.82	4.2444	0.859231	0.7382783	1.443112779	1.374739971	2.817853
5	0.64596875	36.9125	73.825	3	1	1	0.601972	1.2039446	1.2791	3.31	4.2339	0.798517	0.6376294	1.059233765	0.987090117	2.046324
6	0.81265625	46.4375	92.875	4	0	0	0.726116	1.4522322	1.0604	4	4.2417	0.687572	0.4727554	0.65107414	0.581740943	1.232815
7																

Conclusion:

Phase analysis

We got some tabular data of intensity and 2theta values. We made a plot of it in the Origin software and obtained some peaks. Then we have a data set where with values of intensity, 2theta value and also hkl planes of different samples. Then we compare both the datas and found out the hkl planes and the phases.

Crystal structure of Cu2O is simple cubic and that of Al2O3 is rhombohedral.

From the plot of a vs NR-constant, we got the equation,

y=-0.0022x+4.2443

From the above equation we can conclude that the value of the lattice parameter is 4.2443nm.

Standard lattice parameter value of Cu2O is 4.2696 A.

Error = 4.2696-4.2443 =0.2253A

Error%= 5.27%