SYNTHESIS AND CHARACTERIZATION OF MIXED OXIDE NANO COMPOSITES

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ABSTRACT

The emerging technology of nano structure has influenced the world of materials with its significantly enhanced physical, chemical and structural properties, leading to development of various combinations of metal matrix composites, having reinforcements in the order of nano scale. The present work reveals the influence of Nano oxide on mechanical properties of metals, most suitable synthesis technique which was found to be the co-precipitation method; which uses bottoms up approach for synthesising of mixed oxide (CuO-Al_2O_3) Nano particles. The precipitate is obtained by the reaction of copper nitrate, aluminium nitrate and sodium hydroxide, is centrifuged and subjected to calcination at 600˚C for 6 hours. The Nano oxide particles are characterized to confirm the surface morphology, particle size and the chemical composition of the oxide respectively. The obtained oxide is used as reinforcement with copper metal as the matrix material to form metal matrix composites using powder metallurgy with variable being the percentage of reinforcement. The MMC's are tested for parameters such as ultimate tensile strength using the Brinell hardness values and corrosion resistance. The test result suggests an increase in the mechanical properties and corrosion resistance with increase in the reinforcement are evident.

***Keywords*: Characterization, Metallic oxides, Scanning Electron Microscope (SEM).**

I. INTRODUCTION

Nanotechnology is the understanding and control of matter at dimensions of roughly 1-100 nanometers where unique phenomena enable novel applications. Nanotechnology involves imaging, measuring, modeling and manipulating matter at this length scale. Nanotechnology will indeed affect every industry through improvements to existing materials and products, as well as allowing the creation of entirely new materials' and 'produce important advances in areas such as electronics, energy and biomedicine.In the emerging field of nanotechnology, a goal is to make nanostructures or nano arrays with special properties with respect to those of bulk or single-particle species. Oxide Nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface sites. Particle size is expected to influence three important groups of properties in any material. The first one comprises the structural characteristics, namely, the lattice symmetry and cell parameters [1]. The field of nano composite materials has had the attention, imagination, and close scrutiny of scientists and engineers in recent years. This scrutiny results from the simple premise that using building blocks with dimensions in the Nano size range makes it possible to design and create new materials with unprecedented flexibility and improvements in their physical properties. This ability to tailor composites by using Nano size building blocks of heterogeneous chemical species has been demonstrated in several interdisciplinary fields.

Synthesis of nanomaterial by a simple, low cost and in high yield has been a great challenge since from the inception of Nano Science. Various bottom and top down approaches have been developed so far, for the commercial production of nanomaterials. [2]

1.1 Co – Precipitation Method

One conventional method to prepare Nanoparticles of metal oxide ceramics is the precipitation method. This process involves dissolving in a salt precursor, usually a chloride, oxy chloride, or nitrate. The corresponding metal oxides usually form and precipitate in water by adding a basic solution such as sodium hydroxide or ammonium hydroxide to the solution. The resulting chloride salts, are then washed away and the hydroxide is calcined after filtration and washing to obtain the final oxide powder. This method is useful for preparing composites of different oxides by co-precipitation of the corresponding hydroxides in the same solution. One disadvantage of this method is the difficulty to control the particle size and size distribution.

II. EXPERIMENTAL

2.1 Material

The materials used in preparation of mixed oxide Nano particles are 0.1M Copper Nitrate Tri Hydrate,0.1M Aluminium nitrate 0.2M flaked Sodium Hydroxide solution, CTAB ($(C_{16}H_{33})N(CH_3)_3Br$), Cetyltrimethylammonium bromide) as surfactant.

2.2 Nano Oxide Preparation

In the precipitation method employed by us, a solution of 0.1M copper nitrate trihydrate + Aluminium nitrate is titrated against 0.2 M flaked sodium hydroxide solution. The surfactant being CTAB ($(C_{16}H_{33})N(CH_3)_3Br$, Cetyltrimethylammonium bromide). The size of the salt solution droplets were controlled to ensure Nano size of mixed hydroxide precipitate, shown in Fig 1. The obtained precipitate is centrifuged, where the centripetal acceleration causes denser substances to separate along the radial direction (the bottom of the tube). By the same force lighter objects will tend to move to the top of the tube; in the rotating picture, which helps in separating the precipitate from the solution[3]. The centrifuge equipment is as shown in Fig 2

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Figure 1 Precipitation of Mixed Hydroxide

Figure 2 Centrifuge Process

The black precipitate obtained is dried at 60˚C to evaporate ethanol, used as cleaning agent and followed by calcination at 600˚C for 6 hours to convert the obtained hydroxide into Nano mixed oxide.

2.2 Specimen Preparation

The synthesized mixed oxide is mixed in the varying proportions of 0, 5%, 10%, 15%, and 20% with copper as base metal to carry out the ball milling process, as shown in Fig 3. [4].Pure copper metal powder is mixed and mixed oxide nanoparticle is filled into a die of required shape and length. It is placed in a slot of die compaction/ pressing machine, shown in Fig 4. A load of 22 kN, is applied for approximately 10 minutes, the load is then withdrawn and the newly formed billet is obtained and removed from the die. The obtained billets are subjected to sintering process of 640˚C for 2 hours. [5]

Figure 3 Ball Milling Equipment

Figure 4 Die Compaction Process

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III. TESTING

3.1 Hardness Testing

The specimen was test under Rockwell Hardness scale according to the ASTM – E18 standards, HRB scale was chosen for then testing of copper specimen according to the same E18 standards. A ball indent of 1/16th of an inch diameter made of unhardened steel is fit into the slot and the machine is set to a load of 100kN. The billet is placed on the pulpit and raised up until the ball indent just touches the billet. A rotation worth two and a half turns is provided until the meter points at the value "3". The load is then applied for 15 seconds; specimen was unloaded to obtain the hardness values. The procedure was repeated for 6 times on different areas of the specimen to eliminate error in the hardness values, referred from ASTM E18-12. Later ultimate tensile strength is approximated using the equation 1. [6]

Ultimate tensile strength (MPa) = 3.3 * BHN (Equation 1)

IV. RESULTS AND DISCUSSION

4.1 Characterization of Nano Copper Oxide

The obtained CuO – Al_2O_3 Nano powder was characterized using techniques such as SEM, EDAX to reveal the surface topography, chemical composition, to confirm the presence of synthesized oxides.

SEM micrograph of the calcined (600 ºC) mixed oxidenanoparticles are shown in Fig. 6. It shows that higher tendency of agglomerations. We collect the SEM images in different scales; the images show the cage like structures. The average particle size was around 8.3 nm.

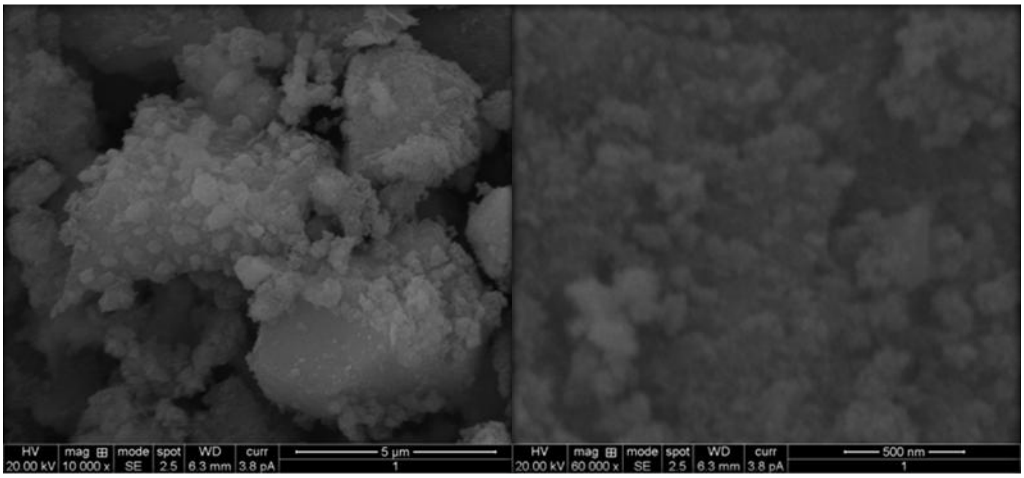

Figure 6 SEM images of CuO – Al_2O_3

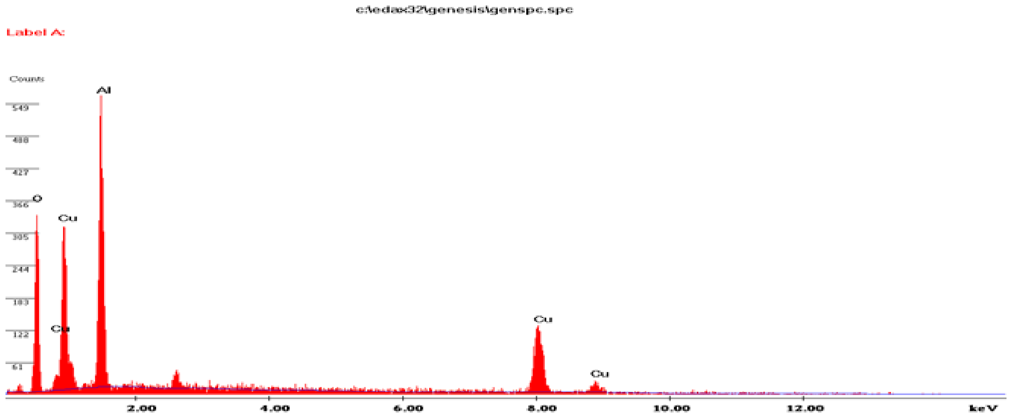

Figure 7. EDAX Analysis of CuO

Fig.5 gives the EDAX spectrum of the calcined CuO-Al_2O_3 Nanofibers at 600˚ C. It confirms the presence of the elements in CuO-Al_2O_3. From the EDAX measurements, it was obtained that Copper, Aluminum and Oxygen are present in the weight percentages of 63.99, 21.03 and 14.99 [9]

4.2 Hardness Test

The hardness test was conducted according to ASTM E-18 standards and the results obtained are tabulated in Table 2, there is a considerable change in the hardness values resulting from the addition of oxides to the base metal. The variation in the oxide content is studied with the help of the plot of Hardness v/s % of oxide content shown in Fig. 9.

Table 1 Variation of Hardness with Oxide Content

% OF OXIDE	ROCKWELL HARDNESS 'B'SCALE
0	50
5	75
10	78
15	85
20	88

As shown in the Fig 8. There is a considerable increase in the hardness value with addition of 5% oxide content to the base metal. Later with the increase in the percentage of oxide content there is change in the hardness value. Further increasing the oxide content will not have much effect as the curve reaches a flat contour.

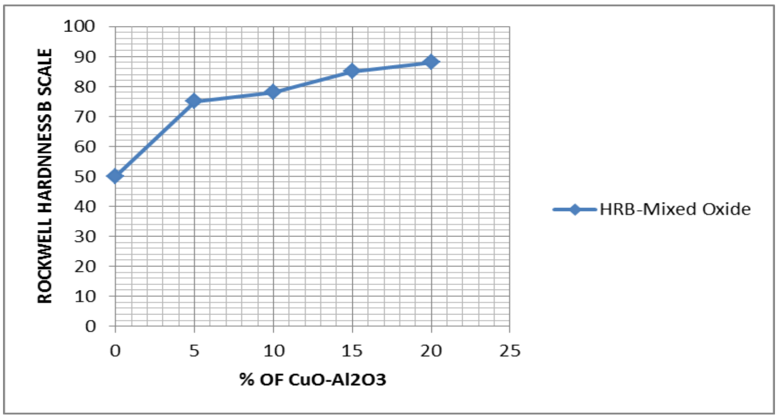

Figure 8 Variation of Hardness with Oxide content

4.3 Ultimate Tensile Strength

The ultimate tensile strength of the materials can be obtained by considering the values of hardness .i.e., by the Brinell values. The Rockwell hardness values were converted to Brinell hardness values by the conversion table E140 – 07 and the ultimate tensile strength was obtained using the equation (2). [10]

Table 2Variation of Ultimate tensile strength with oxide content

% OF OXIDE	ULTIMATE TENSILE STRENGTH (MPa)
0	295
5	458
10	474
15	534
20	582.5

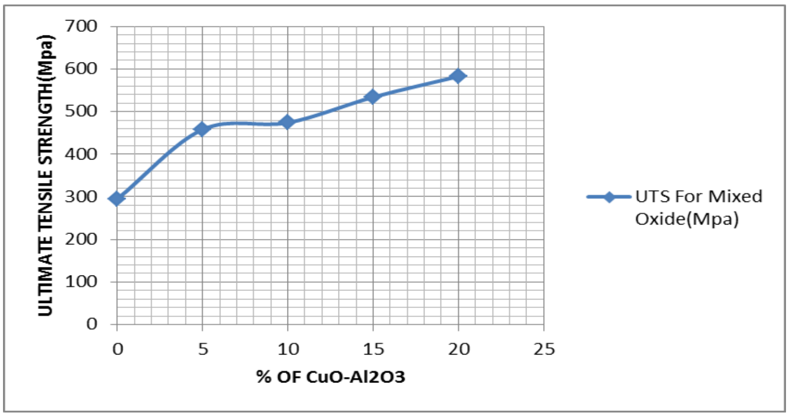

Figure 9 Variation of UTS with Oxide content

The ultimate tensile strength values tabulated in Table 2 shows that the addition of oxide content enhances the UTS values. It is seen that addition of 5% of oxide content increases the strength value by almost 35% and further increase in the oxide content increases the strength value. The graphical representation of the effect of oxide content is as shown in Fig 9

V. CONCLUSIONS

Amongst various methods available to produce nanomaterials, a much better control of the product nanostructures can be achieved by direct co-precipitation (CP) of the oxide components from a liquid solution with subsequent Calcinations.The advantage of using Precipitation method is that one can control the stoichiometry of the oxide nanostructures in a precise way. It was found that the value of density reduced with respect to parent metal (pure copper) due to the addition of less dense oxides, while the hardness increased with increasing percentage of the Nano Copper oxide powder intern Ultimate Tensile Strength.

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