Preparation and Investigation of Cu-Co-Al2O3 Nanocrystalline Metal Matrix Composites

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Abstract-In this experimental work copper, cobalt and alumina (Al_2O_3) of size 74µm, 50µm and 44µm respectively are mixed at a proportion of 65%, 25% and 10% respectively .The mixture is reduced to nano scale by using planetary ball mill. The structure is studied with the help of scanning electron microscope. The morphology and composition are learnt with the help of x-ray diffractometry. This mixture of powder in nanoscale is converted into pellets of required dimension by using two routes of processing. The first route is powder metallurgical process and the second route is by casting. The hardness of the resultant pellet is tested with the help of Vicker's hardness testing machine and the magnetic properties of the resultant pellet is analyzed with the help of vibrating sample magnetometer.

Key Words: XRD, Vicker's hardness testing machine, powder metallurgy process and vibrating sample magnetometer

I. INTRODUCTION

The development of highly reinforced copper matrix composites instigated numerous research works. Ceramics particles like metal oxides and nitrides are commonly made as reinforcements. Cobalt with its high magnetic properties and alumina with its good hardness coupled with high temperature resistant properties form a suitable reinforcement in the copper base. Usually reinforcing materials are strong with low density while the matrix is ductile. The composite with the combination of strength from the reinforcement and the toughness of the matrix provide desirable properties, not normally available in any single conventional material. To fabricate metal matrix composites, among the various manufacturing technologies, powder metallurgy is one of the most advantageous techniques.

II. EXPERIMENTATION

A. MATERIALS SELECTION

Besides good thermal and electrical conductivity the other properties of copper include strength, hardness, and corrosion and wear resistances. Number of materials have been developed utilizing these properties to an optimum for a wide range of applications. Cobalt is known for its resistance to stress and corrosion at high temperatures. Cobalt has always been used in combination with other materials, but not in its pure form. Cobalt

plays an important role in the production of super alloys being used in aerospace applications. Alumina, the ceramic is used in wear protection and ballistics. When this unique combination is made in to a composite a unique property may be evolved and probably make a replacement to several alloy systems. So the combination of copper, cobalt and alumina was chosen for the investigation. Copper, cobalt and alumina of size 74μm, 50μm and 44μm respectively are mixed at a proportion of 65%, 25% and 10% respectively

B. PLANETARY BALL MILL

The reduction of the mixture from micro level to nano level was done with the help of PLANETARY BALL MILLING MACHINE. Here the milling is done for 15 hours, for every 1 hour the powder is stirred to avoid agglomeration. The milling is done at a speed of 250 rpm with 20 grinding balls. The grinding balls and grinding jar are made up of TUNGSTEN CARBIDE. Due to the centrifugal forces, collision of balls against each other and against the walls of the jar mechanical alloying and breaking takes place continuously at a faster rate resulting in the reduction of particle sizes to the nano regime.

C. DIE PRESSING

The dominant technology for the forming of products from powder materials, in terms of both tonnage quantities and numbers of parts produced is Die Pressing. Pellets of 20mm in diameter and 5mm in thickness are compacted at the pressure of 372 bar. The pellet of 10mm in diameter and 2mm in thickness is compacted at the pressure of 400 bar is compacted.

Fig 1 Compacted Pellets

D. SINTERING

By using the box furnace the 20mm diameter pellet is sintered at a temperature of 10˚c it took 2 hours and 20 minutes to reach the temperature of 10˚c and it is kept at this temperature for 1 hour and then cooled within the furnace for another 2 hours. Sintering helps to diffuse the particles and creates thermal bonding with each other. The 10mm diameter pellet is sintered at a temperature of 0˚c it took 3 hours to reach the temperature of 0˚c and it is experiment at this temperature for 2 hours and then cooled within the furnace for another 3 hours

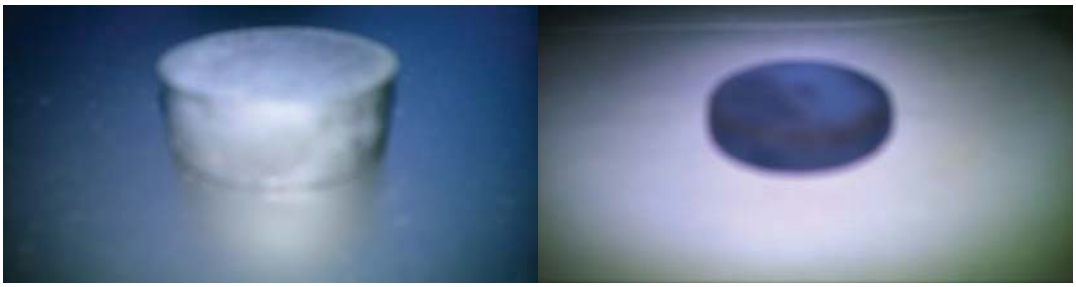


Fig 2 Sintered pellets of varying sizes

III. RESULT AND DICUSSIONS

A. NANO CHARACTERIZATION

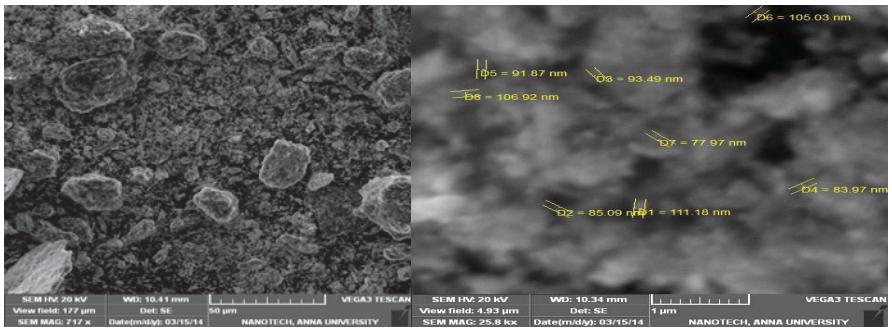


Fig 3 SEM image at (a) and (b) after milling

The scanning electron microscope images were taken after ball milling to find the size reduction. Initially the sizes of the particles were around 750nm. After 15 hours of milling the size, of the particles were on an average of 65nm. The powder got agglomerated when there was a pause in the milling process. So milling was carried our continuously. The Powders after continuous milling for 15 hours entered the nano regime. In order to ascertain the size reduction, the X-ray Diffraction analysis was made.

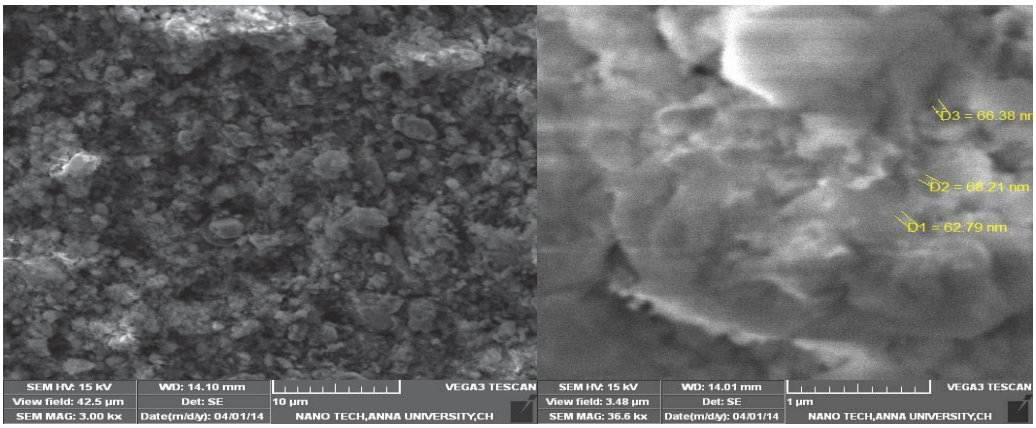


Fig 4 SEM image at the magnifications of (a) 5000 & (b) 36000 for pellet sintered at 950° C

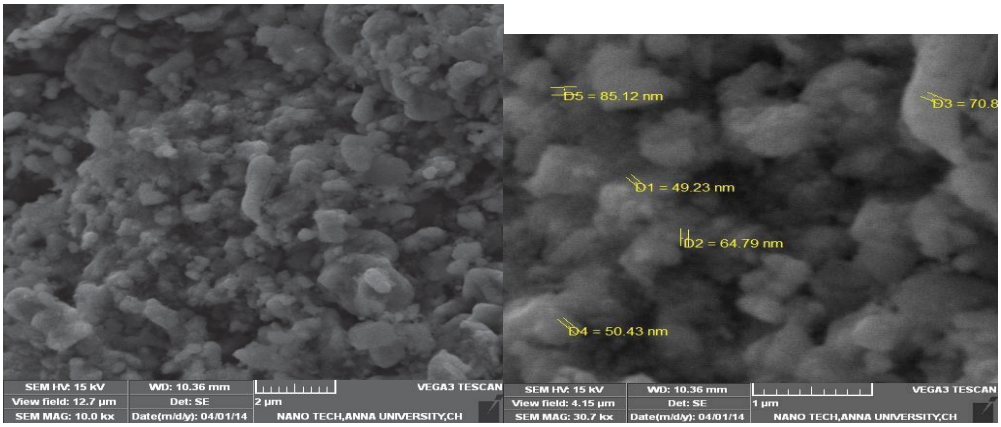


Fig 5 SEM image at the magnifications of (a)10000 & (b)36000 for pellet sintered at 710° C

The figures 4 and 5 show the magnified SEM images of the cast material. At elevated temperatures because of the diffusive nature of the copper particles having comparatively less melting points created an agglomeration. These agglomerations become a hindrance in finding out the nano particles during the SEM analysis. The particles could be viewed in the samples treated at lower sintering temperatures.

B. X-RAY CRYSTALLOGRAPHY

The Scherrer equation, relates the size of sub-micrometre particles, to the broadening of a peak in a diffraction pattern. XRD analysis was used to determine the size of particles of crystals in the form of powder .The crystallite size of the composite is based on X-ray diffraction line broadening and calculated by using Scherer equation.

$$d = k\lambda / B \cos\Theta \qquad (1)$$

where d is the average crystallite size of the phase under investigation, B is the Shape factor, λ is the wave length of X-ray beam used is the full-with half maximum (FWHM) of diffraction and θ is the Bragg's angle. With the 2Θ being 43.56° and λ=0.162nm ,B=0.59°or0.0103 and k=0.9(shape factor), crystallite size for the copper based composite is 15.24 nm. Figure 6 shown below shows the peak values for the composites. The peak values help to find out the FWHM. The peak values are also compared with the data base ICPDS comes along with origin pro software. The peak values obtained from XRD matches with the values available with the data base supported by international council for powder diffraction studies.

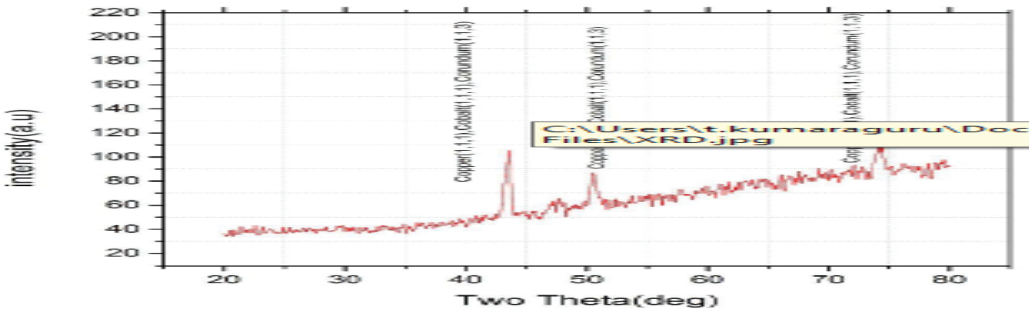


Fig 6 XRD analysis

C. HARDNESS TEST RESULTS

The Vickers indenter is four sided pyramid with square base and an apex angle between opposite sides of α = 136°. The hardness number is calculated by dividing the load (indentation force) by the surface of the imprint. In case of Vickers, the surfaces are the 4 pyramidal surfaces. The resulting formulae for the Vickers hardness is calculated as,

$$HV = F/A = 2F \sin(\alpha/2)/d^3 \qquad (2)$$

Where, HV = Vickers hardness number, F = Test load (in gram), A = Surface area of the imprint, d = Average diagonal length of an imprint α Face angle of the Vickers indenter. From the vicker's hardness values it has been observed that the hardness of copper increases with the reinforcements present in the nano scale. The nano particles cobalt and alumina increases the hardness values of copper from 36-40HV to 48-55HV. It is also inferred that the hardness values can go up with increase in the percentage composition of nano particles. If the nano conversion takes place using the chemical reaction techniques than the chosen mechanical alloying technique the agglomeration could have been prevented and nano stability cam be achieved. The nano stability plays an important role in elevating the properties of the composites. The Vicker's hardness calculated using the formula gives closer values to the one observed using the computerised micro hardness testing machine.

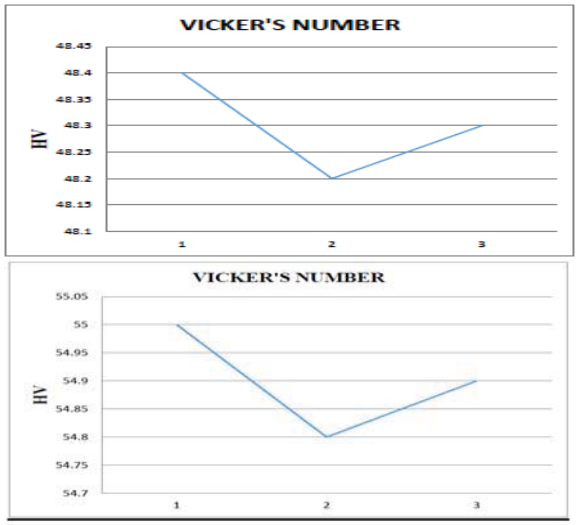
Table 1 Vicker's hardness value for materials sintered at (a) 700°C & (b) 950°C

(a)

Reading Number	Hardness Number
1	48.4
2	48.2
3	48.3

(b)

Reading Number	Hardness Number
1	48.4
2	48.2
3	48.3



(a) (b)

Fig 7. Vickers's hardness number at 50gf for pellet sintered (a) at 700° C & (b) 950°C

D. MAGNETIC PROPERTIES

A great deal of information can be learned about the magnetic properties of a material by studying its hysteresis loop. A hysteresis loop shows the relationship between the induced magnetic flux density (B) and the magnetizing force (H). It is often referred to as the B-H loop. From the results of the M-H hysteresis graph obtained from the VSM (Vibrating sample Magnetometer) it is clear that the resultant composite has a magnetic retentively MR of 83.721E-3 emu and saturation magnetization MS of 1.2559 emu and the magnetic coercively Hci is 240.87 G. The magnetic properties are higher than the properties of pure copper. The figure 8 shows the typical curve comprising the magnetic properties. The values are tabulated in Table 2.

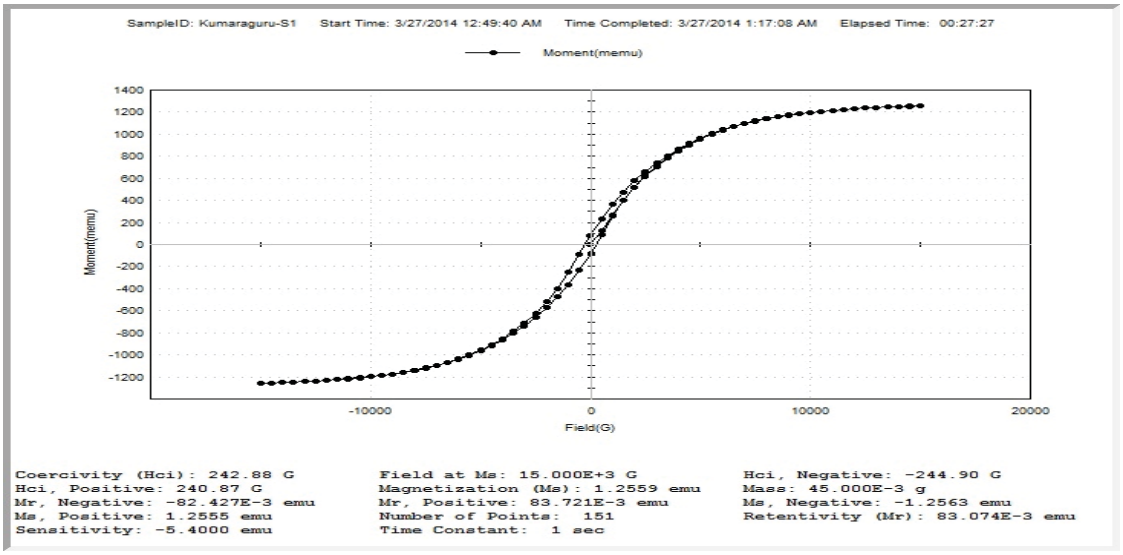


Fig 8 M-H hysteresis graph at room temperature (300K) for the composite.

Table 1.2 VSM Analysis Data

Coercivity (Hci)	242.88 G
Field at Ms	15.000E+3 G
Hci Negative	-244.90G
Hci Positive	240.87G
Magnetization (Ms)	1.2559 emu
Mass	45.000E-3 g
M_R ,Negative	-82.47 E-3 emu
M_R, Positive	83.721E-3 emu
Ms, Negative	-1.2563 emu
Ms, Positive	1.2555 emu
Number of points	151
Retentivity (M_R)	83.07E-3 emu
Sensitivity	-5.4000 emu
Time constant	1 sec

IV. CONCLUSION

- The copper based composite is reduced to nano scale with the help of ball milling, the microstructure is analyzed with the help of SEM analysis, the composition is confirmed with the help of XRD analysis, hardness of the pellets made of nano composites are measured with the help if computerised micro hardness tester and the magnetic properties is analyzed through VSM analysis.

- The composite powder is converted into pellet by powder metallurgy process and casting process .The resultant pellet is analyzed for hardness by vicker's hardness testing machine

- From vicker's hardness values it is seen that the hardness of copper is increased from 36-40HV to 48-55HV by reinforcing cobalt and alumina.

- It is observed from hardness values that if the sintering temperature is increased then hardness of the material also increases and the hardness obtained in casting process is greater than the hardness obtained in powder metallurgy process. Inadequate compacting pressure is the reason for the low hardness during compaction.

- From the results of the VSM analysis, it is clear that the resultant composite has higher magnetic properties than copper. This is due to the presence of cobalt. It is also seen that the nano composite has a Magnetic Retentivity, MR = 83.721E-3 emu, Saturation Magnetization MS =1.2559 emu and Magnetic Coercively Hci = 240.87G.

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