Microstructure, Physical Properties and Hardness of Cbn/Ni-Cu Composites Fabricated by Powder Technology

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Powders of Ni and Cu were produced by water atomization technique to fabricate metal matrix composites containing various percentages of cubic boron nitride powders 1, 2, 3, 4, 5 wt.% cBN in a matrix containing 20 wt.%Ni and 80 wt.%Cu. Prepared mixtures were cold compacted under 400 MPa, and sintered for 2h at 1000oC in controlled atmosphere of 3:2 N2/H2 gas mixture. The microstructure and the chemical composition of the prepared powders as well as the consolidated composites were investigated by X-ray diffraction as well as field emission scanning electron microscope (FESEM) equipped with an energy dispersive spectrometer (EDS). The produced Cu and Ni powders have spheroid shape of size less than 100 micron, but the investigated cBN has a flake like shape with sharp edges and particle size less than 1 micron. It has been also observed that cBN and Ni particles were homogeneously distributed in the Cu matrix of the present cBN/Ni-Cu composites. The density, electrical resistivity, saturation magnetization and hardness of the composites were measured. It was observed that, by increasing cBN wt.%, the relative density was decreased, while the saturation magnetization, electrical resistivity and hardness were increased.

1. Introduction

Metal matrix composites (MMCs) are of high specific strength, specific modulus, damping capacity and good wear resistance, compared to unreinforced alloys. Ceramic reinforced MMCs have a great mechanical performance [1]. There has been an increasing interest in composites containing low density and low cost ceramic reinforcements. It offers a possibility to tailor the properties of a metal by adding an appropriate ceramic reinforcement and to meet demands in physical and mechanical management. Metals as Al, Mg, Fe, Ti, Ni, Cu, Ag, Co and Nb or alloys as Ni-Cu, Zn-Co, Co-Ni, Ni-Fe were mainly used as metallic matrices [2]. Among others nickel as a durable and tough metal has been widely used, due to it’s resistant to corrosion and abrasion [3]. There are several techniques for the fabrication of MMCs. Powder Metallurgy (P/M) is one of these methods. Metal matrix composites produced by P/M have unique properties. Because composites are sintered at lower temperatures in P/M method, undesirable compounds are less often formed at the interfaces. Accordingly, such composites have superior mechanical properties. In addition; P/M technique is a continually and rapidly evolving technology embracing most metallic and alloy materials and a wide variety of shapes, it has been frequently used to manufacture a verity of mechanical components owing to its low energy consumption, high materials utilization, and low production cost [4, 5].

There are several types of particles, whiskers or fibers ceramics that can be used as reinforcement in MMCs (as SiC, Al_2O_3, TiC, WC and diamond) according to the required application of the materials. Although diamond is the hardest known material in nature, it is rare and too expensive to be used as reinforcement. Cubic boron nitride "cBN" is synthesized in 1957 [6-8], and it is the second hardest known material and it possesses numerous excellent physical and chemical properties, high corrosion resistance and mechanical properties. Boron nitride has two crystal structures; hexagonal (hBN) similar to graphite and cubic (cBN) similar to diamond. However, cBN is much harder than hBN. Similar to the synthesis of diamond at high temperature and high pressure; cBN can also be formed at high temperature under high pressure (ultra hP/hT). A catalyst might lower the required temperature and pressure [9, 10].

Using cBN as a cutting tool at severe conditions of temperature and cutting speed [11] is referred to its high properties, such as high hardness, high thermal and chemical stability. It is used mainly for machining hard ferrous materials, especially when diamond reacts chemically with these materials at temperatures higher than 700°C. However, pure cBN compact is difficult to prepare even using (hP/hT) techniques [12]. Several research works succeeded to fabricate cBN using sintering additives. The sintering conditions depend on the used additives and/or binding materials. It requires a pressure of 6 GPa and a temperature of 1500°C. Powders of cBN used to fabricate tools of difference shapes are usually mixed with metal binders. Such sintering conditions are attained using ultrahigh pressure devices. In addition, when additives such as powders of Ni, Al and Cu, are added to cBN particles for solid phase sintering, their effect as sintering additives or binding materials is limited [13-16].

Copper base composites offer excellent strength properties for several applications. It is currently produced by P/M technique, in which cold compaction and/or hot pressing are performed at high pressure and temperature, because the expansion of compacts during sintering leads to the formation of pores and the weakening of the bond between the ceramic powder and the metal matrix [17, 18]. Alloying elements as Ni, Sn, Pb or Al can be added to the Cu base composites to improve its resistance, ductility and thermal stability without causing considerable damages of its form, electric and thermal conductivity and corrosion resistance [19, 20].

The primary objective of this research is to fabricate cubic Boron Nitride / Nickel- Copper Matrix Composites {cBN / (Ni-Cu)} to study the effect of cBN nano-particles content on the composite microstructure, physical properties and hardness. The objective may also be rewritten as the formation of cBN / (Ni-Cu) MMCs with high uniform distribution of cBN particles.

2. Experimental Methods and Procedures

2.1. Materials

Powders of Ni and Cu are prepared by the atomization technique at the Central Metallurgical Research and Development Institute, Cairo, Egypt using water atomizer model PSI; UK. Copper and Nickel powders are prepared separately by induction melting in silicon carbide crucibles in air. In case of Cu it is superheated to1300°C, and in case of Ni to 1700°C. After complete melting bottom pouring is carried out through a ceramic melt delivery nozzle of 6 mm diameter into a confined water atomizer operating at a pressure of 20 MPa. The high pressure water jets were directed against the molten stream. The melt flow rate, estimated from the operating time and weight of the atomized melt, is about 4 kg/min. The water flow rate, calculated from the water consumption rate, was about 200 l/min. Table (1) reports the atomization conditions applied for Cu and Ni powders fabrication. The size distribution of Cu and Ni powders is determined by conventional mechanical sieving vibrator model Control Lab FRANCE. Sieved powders with a specific size range of 100 µm are used for present investigation. Cubic boron nitride powder with an average particle size of 500-700 nm is purchased from (ILGIN Co. LTD- South Korea). Different mixtures of up to 5wt.% cBN and a mixture containing 20wt.% Ni and 80wt.% Cu are prepared by mixing with 0.5wt.% paraffin wax in acetone for 30 min. in a ceramic mortar. The obtained mixtures are heated at 60°C for 120 min. followed by cooling to room temperature.

Table (1): Applied conditions for the atomization of Cu and Ni powders

Parameter	Condition	Parameter	Conditio
Pouring temperature of Cu, ^{o}C	1300	Pouring temperature of Ni, ^{o}C	1700
Nozzle angle	35^{o}	Nozzle diameter, mm	6
Nozzle of water jets, mm	4	Molten stream flow rate, kg/min.	4
Water pressure, MPa	20	Water flow rate, l/min.	200
Water velocity, m/s	90		

2.2. Consolidation of cBN/20Ni-Cu Powders

The produced cBN/Ni-Cu mixtures are cold compacted under 400 MPa using a uniaxial hydraulic pressing machine model OMCN; ITALIA in a 9mm diameter cylindrical hardened steel mold. The obtained compacts are sintered for 2 h in a closed furnace in controlled atmosphere of 3:2 N_2/H_2 gas mixture at 1000^oC. Finally, the furnace is turned off and the sintered specimens are maintained in the furnace for cooling in N_2 atmosphere.

2.3. Methods of Assessment of Powders and cBN/20Ni-Cu Composites

An extensive microstructural and morphological study is preformed to investigate Cu, Ni, cBN powders and cBN/Ni-Cu mixtures, as well as the grinded and polished cBN/Ni-Cu sintered compacts by QUANTA FEG250-EDAX Genesis field emission scanning electron microscope equipped with an energy dispersive X-ray micro analyzer (EDS). Also, the chemical composition and phase analysis are investigated by X-ray diffraction (XRD) on a Brukur advanced X-ray diffractometer model D8 kristalloflex (Ni-filtered Cu Kα).

The expected theoretical densities of designed compositions are calculated by the rule of mixtures using theoretical density values of the elements Cu (8.96 g/cm^3), Ni (8.90 g/cm^3) and cBN (3.48 g/cm^3) [21, 22]. The theoretical densities of produced composites are calculated by using the rule of mixtures (equation 1) [23].

$$D_c = d_m \cdot V_m + d_f \cdot V_f \qquad (1)$$

where, d_c, d_m, d_f densities of the composite, matrix and dispersed phase respectively and V_m, V_f volume fraction of the matrix and dispersed phase, respectively.

The relative green density of the produced compacts is measured using a micrometer and a three digit balance. On the other hand, the densities of sintered materials are determined using water as a floating liquid, and sintered density (ρ) are calculated by Archimedes principal given by equation [24];

$$\rho = W_{air} / (W_{air} - W_{water}) \qquad (2)$$

where, W_{air} and W_{water} represent the specimen weight in air and water, respectively.

The electrical resistivity of the samples is measured using digital micro-ohmmeter (GDM- 8145) and the resistivity (ρ) is calculated in μΩ.cm according to the equation;

$$\rho = (R \times A) / L \qquad (3)$$

where, R is the resistance in μΩ, L is the length in cm and A is the cross sectional area in cm^2.

Magnetic hysteresis loops at room temperature (MH curves) of prepared cBN/20Ni-Cu mixtures, and sintered compacts using Lake-Shore model 7400, Japan. The saturated magnetizations of investigated specimens are measured as functions of sample weight.

Vickers hardness of cBN/20Ni-Cu sintered composites are measured using Vickers hardness tester model Shimadzu JAPAN by applying 50g load and 15 sec loading time at room temperature (25°C). The test is repeated for each specimen and the average of at least 5 readings along the specimen cross section is calculated.

3. Results and Discussion

3.1. Assessment of Powders and cBN/Ni-Cu Mixtures

Figure (1) shows sieve analysis of produced copper and nickel powders. Sieve analysis tester Model ControLab (made in France) is used. The amount of copper powder is 100 gm and that of nickel is 10 gm. Only fine particles are used here, and the largest particle size of copper is -53µm, while nickel powder shows different sizes between 125µm to -53µm. Because of the magnetic behavior of nickel, fine copper powder and large nickel particles are used.

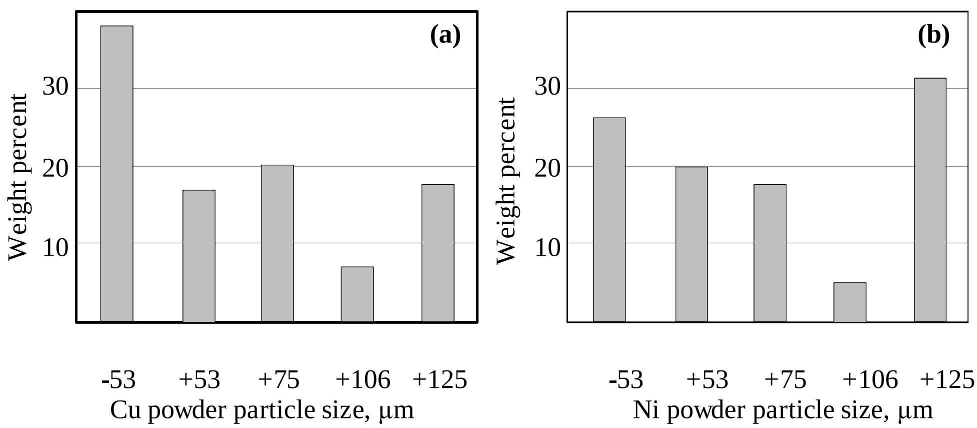

Fig. (1): Sieve analysis of, (a) copper powder, and (b) nickel powder.

Figure (2) shows the FESEM micrographs of as atomized Cu and Ni powders. It could be observed that, the particle shape of as-solidified powders is mostly spherical in case of Cu and cubic to irregular in case of Ni, and is of a rough surface morphology. Fig. (3) shows FESEM photos of cubic boron nitride and a mixture of cubic boron nitride, nickel and copper powders. The mixture contains 5% cubic boron nitride.

Figure (4) (a) shows XRD patterns for the investigated powders of Cu, Ni and cBN particles. Both Cu and Ni have fcc crystal structure and show (111), (200) and (220) reflection lines, but BN is of a cubic structure and shows (002), (111), (200) and (220) reflection lines. Moreover, sintered cBN/Ni-Cu composites consist of (fcc) Cu and (fcc) Ni as a major phase and the coexistence of cubic boron nitride. One of the important features here is the absence of any peak of improper or impurity phase. This implies that there is no reaction between the cBN particles and Ni or Cu metals at the applied sintering conditions (1000°C for 2 h). Only the intensification of the lines (111), (200) and (220), due to the incorporation of cBN nanoparticles in the metal matrix [25-27] may be observed.

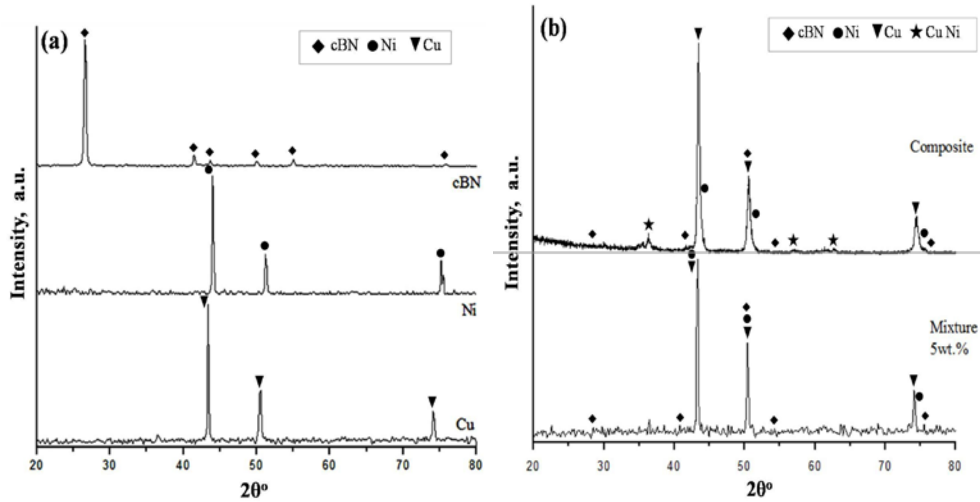

Fig.(4): XRD patterns of prepared: (a) Cu and Ni powders, as received cBN, (b) powder mixture and sintered 5 wt.% cBN/20Ni-Cu composite.

3.2. Microstructure of Consolidated cBN/Ni-Cu Powders

EDS analysis of mixed 5 wt.% cBN/20Ni-Cu using FESEM is presented in Fig.(5). Spot analysis of the marked position is performed and the results show the characteristic peaks for mixed 5 wt.% cBN/20Ni-Cu. Almost uniform distribution of the cBN nano-particles in Cu/Ni mixture is observed. Nanoscale cBN particles have large surface areas. Therefore, increasing nano-size cBN particles content up to 5wt% may increase the chance for the cBN grain growth, in addition the prolonged sintering time enhances grain coarsening [28].

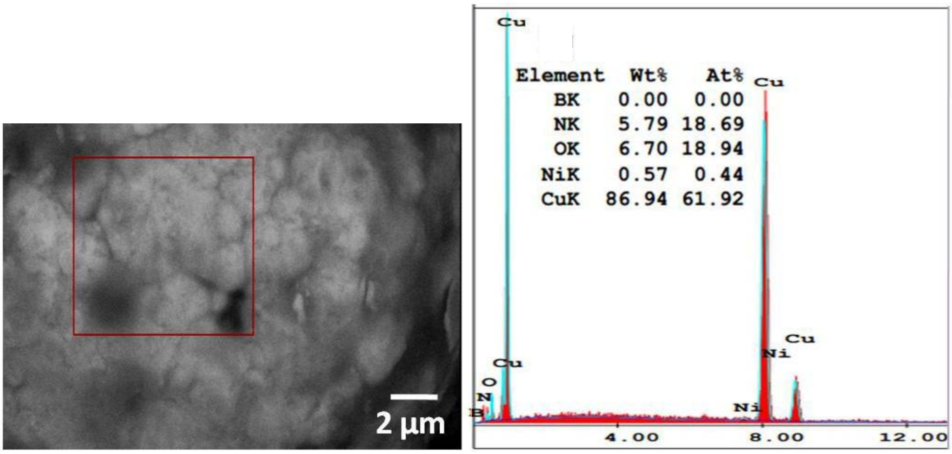

Fig. (5): EDS analysis of mixed 5% cBN/Ni-Cu particles.

In the Backscattered electron (BSE) images cBN particles are appeared to be gray, and this is confirmed with EDS analysis. In order to determine the distribution of elements in the structure, surface analysis is performed by Electron Dispersive Spectrum (EDS). Fig.6 shows the images of FESEM and EDS of sintered 5wt.% cBN/20Ni-Cu nanocomposite. The EDS analysis indicates the main spectrums of B, N, Ni, Cu and O elements at different locations, which correspond to the existing of cBN, Ni and Cu. Fig.(7) shows the distribution cBN in metal matrix leading to a homogenous microstructure of the sintered cBN/20Ni-Cu materials

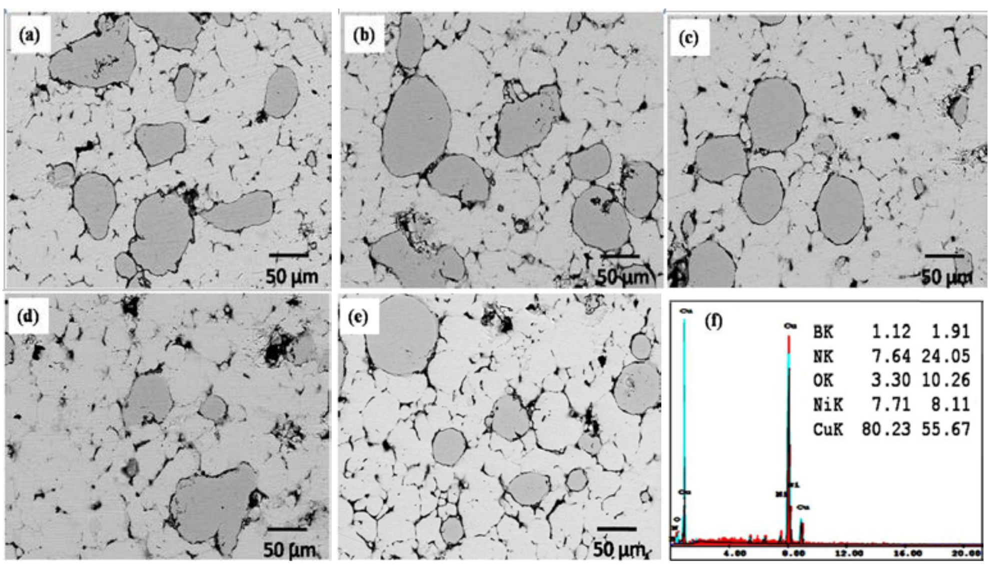

Fig.(6): FESEM micrographs of composites containing: (a) 1 wt.%, (b) 2 wt.%, (c) 3 wt.%, (d) 4 wt.%, (e) 5 wt.% cBN, and (f) EDS analysis for 5 wt.% cBN composite.

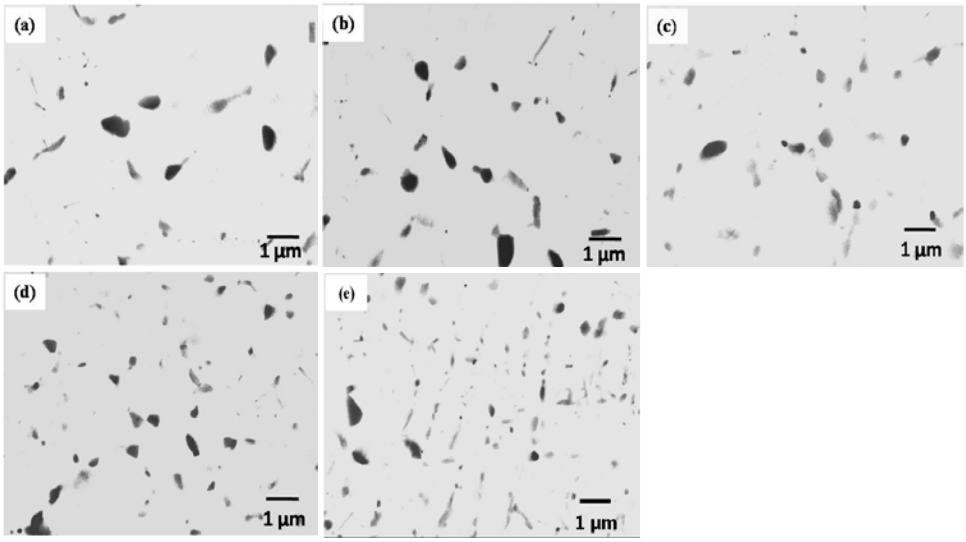

Fig. (7): FESEM micrographs of present sintered cBN/Ni-Cu with various cBN weight contents a) 1 %, b) 2 %, c) 3 %, d) 4 % and e) 5 % cBN.

3.3. Physical Properties of Powders and cBN/Ni-Cu Mixtures

The theoretical densities of the produced composites are calculated by using the rule of mixtures (equation 1), which is an approximate approach to estimate composite properties. The relative density (actual density by a theoretical one) of the investigated sintered composites is measured using a method based on Archimedes' principal.

Figure (8) shows the relative sintered density in comparison with the relative green density of cBN/Ni-Cu sintered materials with different weight contents of cBN. By increasing the cBN weight percent the green and sintered densities decrease. Due to low interaction between the cBN reinforcement particle and the metal matrix.

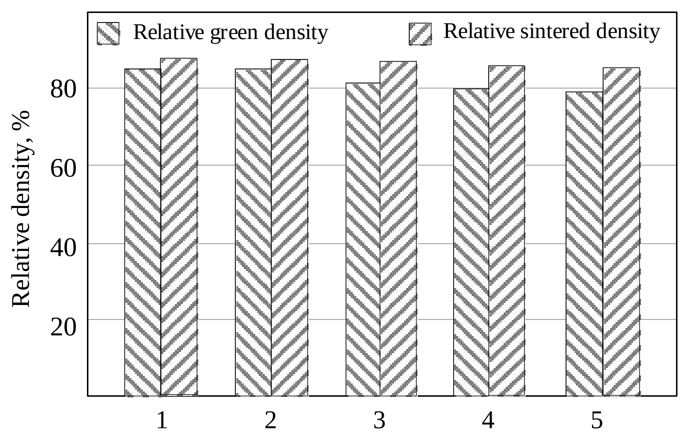

Fig.(8): The relative green and sintered densities of present cBN/20Ni-Cu composites.

Measured values of electrical resistivity of the sintered cBN/Ni-Cu composites are plotted in Fig.(9). The electrical resistivity of Cu-Ni composites is important, especially for electrical or electronic applications. The electrical resistivity increases with increasing the cBN content of the composite, due to the high resistivity of cBN reinforcement. Besides, increasing the cBN content, the sintered density decreases due to the increase of porosity, as could be observed in Fig.(8).

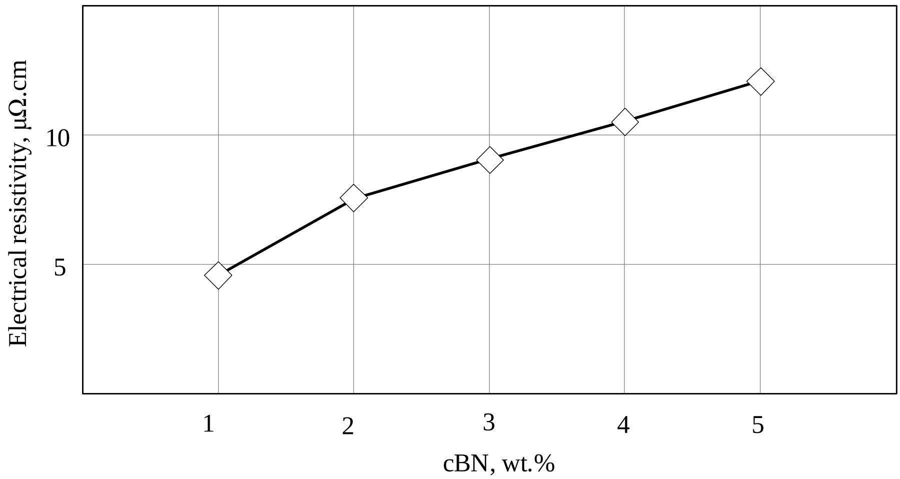

Fig.(9): Effect of cBN content on the electrical resistivity of cBN/20Ni-Cu composites.

Figure (10) shows the magnetic hysteresis loops of the investigated mixtures of cBN, Ni and Cu powders, and Fig.(11) shows the hysteresis loops of the investigated cBN/20Ni-Cu composites, under a magnetic field of 0.5 Tesla. In addition, Fig.(12) shows the effect of the cBN content on the saturation magnetization. It is obvious from Fig.(11) that the saturation magnetization (M_S) of the powder mixtures, decreases with increasing cBN content, due to the nonmagnetic properties of cBN powder [29]. However, in case of the cBN/Ni-Cu sintered composites the saturation magnetization increases with increasing of the cBN content. This could be attributed to the configuration of Ni-Cu compound during the sintering process [30]. The ratio of Ni-Cu decreases with increasing of cBN contents, which is in agreement with XRD results.

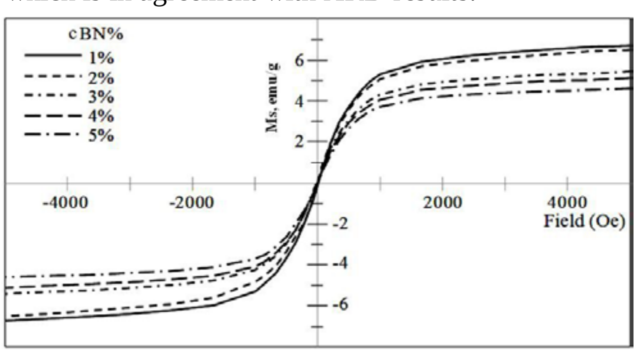

Fig. (10): Magnetic hysteresis loop of investigated mixed powders.

The saturation magnetization of sintered Ni is 51.94 emu/g is lower than the standard value (54.8 emu/g). This could be due to the presence of traces of contaminates as O_2 and P in the prepared Ni [31, 32].

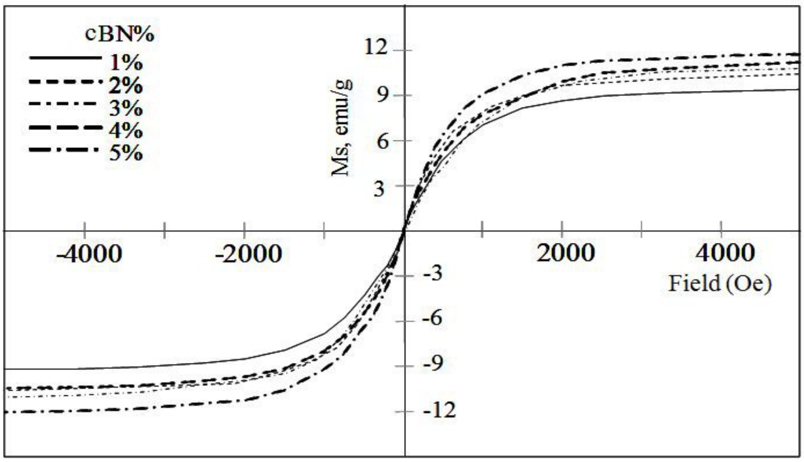

Fig.(11): The magnetic hysteresis loop of the investigated cBN/20Ni-Cu composites.

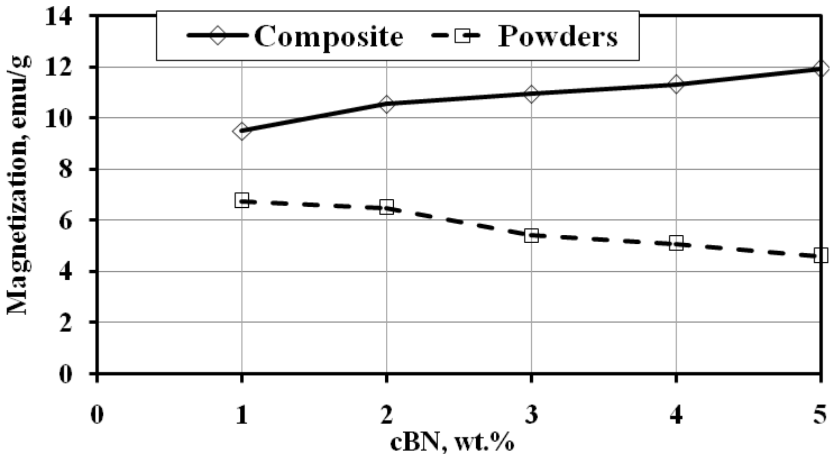

Fig. (12): Effect of cBN content on saturation magnetization of mixed powders and sintered cBN/20Ni-Cu composites.

3.4. Hardness of Sintered cBN/Ni-Cu Composites

The effect of cBN content on the hardness of present composites is shown in Fig.13. By increasing cBN (hard phase) content, the hardness of the composite increases. It is due to the contribution of cBN hard phase in the Ni-Cu matrix, and the tight adhesion between cBN particles and Ni-Cu matrix, as well as the higher densification and low porosity of the sintered cBN/20Ni-Cu composites. However, the increase of hardness is not pronounced, which may be due to the nickel and copper metallic binder effect. By increasing the soft metallic binder content (Ni-Cu) the densification increases leading to hardness increase.

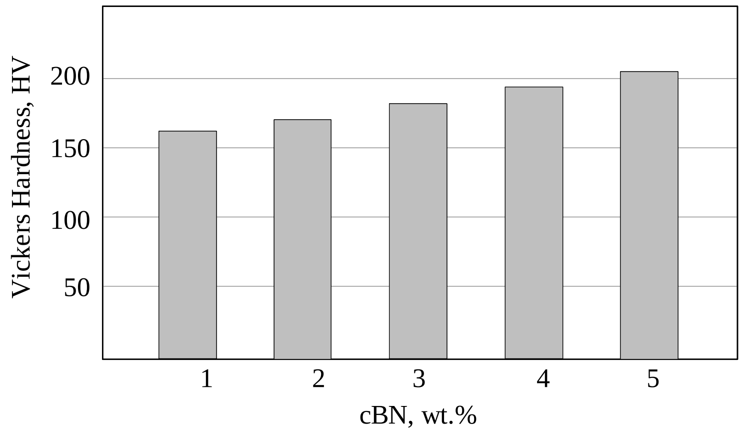

Fig.(13): Effect of cBN content on the hardness of cBN/Ni-Cu composites.

4. Conclusions

Powder metallurgy technique is applied to fabricate Ni-Cu metal matrix composites reinforced with different contents of cubic boron nitride. Based on present results it could be concluded that increasing cBN content leads to:

1- It decreases the density and increases the hardness.
2- The green density and sintered density of cBN/20Ni-Cu composites slightly decrease.
3- Both electrical resistivity and hardness of cBN/20Ni-Cu composites increase.
4- It decreases the saturation magnetization of the cBN/20Ni-Cu powders mixture, and increases the saturation magnetization of the cBN/20Ni-Cu composites.

Copper based composites with improved physical and mechanical properties are produced for several applications.

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