The Effect of Changing the Volumetric Ratios of Zirconia on Some Physical Properties of (Cu-SiC) System by Powder Method

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Ahmed SH. Karim [1]
Tikrit University, College of Education- Physics Department, Iraq.

Zuheer Naji Majeed [2]
Tikrit University, College of Education- Physics Department, Iraq.

Salih Y. Darweesh [3]
Tikrit University, College of Education Tuz Khurmatu - Physics Department/, Iraq.

ahmed.shadhaan1993@st.tu.edu.iq   Zuheer1976@tu.edu.iq   Salih.younis@tu.edu.iq

Abstract :

The current research deals with the powder method to study a system with a mineral base of copper and the first support material of silicon carbide at a constant rate of 5% while the second support material was a variable from Nano crystalline and with a cementation ratio of (0,5,10,15,20) %. The powders were mixed by the volumetric method. Due to the difference in densities between the three powders, the grinding process was carried out for a two-hour period with a mill of local manufacture and with steel balls. Then, the mixed powders were pressed with a Turkish hydraulic press at a pressure of (5 tons) and for one minute. The prepared samples were thermally sintered in an English furnace at 900 °C for only two hours. Moreover, various tests were performed on the models including (hardness, density, porosity, X-ray diffraction) before and after sintering. The results showed that the best mixing ratio was 15% zirconia, which included micro-hardness (90.81 Hv) and less porosity (14.6%). Also, the density at 15% was (7.897 g/cm^3). X-ray diffraction showed that the materials used were the base material (copper) with a cubic phase (crystalline) at Miller's coefficients (111), (200) and (220), as well as silicon carbide (SiC) with the fixed support material and a cubic structure. The emergence of the variable reinforced zirconia Nano composite (ZrO$_2$), Miller's coefficients (111), (200), (220), (311) and (222), and the monoclinic composition of Miller's coefficients (011), (110), (1-11), (111), (002), (102-), (112), (013).

Keywords: Vickers Hardness, Monoclinic, hydraulic Press, Heat Treatment.

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Introduction:

The powder metallurgy technology is summarized in the preparation of mineral, ceramic or polymeric powders, as these powders are pressed to obtain products in the required shapes, then heated by the sintering process in order to improve the bonds between the particles and to obtain a product with a coherent mass (Rigid Mass). Also, molds are designed for this purpose in order to obtain products pressed with certain pressure levels, while the sintering process is carried out at temperatures lower than the melting temperature of the base metal [1]. As a result of the tremendous industrial progress, researchers have manufactured materials that have such distinct qualities as shock resistance, corrosion resistance, and low cost. There are the continuous phase, the Reinforcement Material or the Distributed Phase, and the phase surrounding the base material is called the "Interphase." The base materials are either metallic, ceramic, or polymeric, and the reinforcing materials are either minutes, fibers, sheets, or filaments [2]. Knowing the properties and specifications of the base materials as well as the support materials helps us determine the type of material that can be produced and the place in which this material is used. For example, in the aerospace industries, composite materials that work at high temperatures and low densities have been manufactured, but in the medical industries, for instance, composite materials have been developed with high resistance to corrosion and fracture [3]. Zirconium-iron additive materials is called cermet. In recent years, scientists have made a great effort to use these composite materials on a large scale in many electrical and thermal applications that require good heat-insulating properties at high temperature ranges. These materials have been in use due to their strong resistance to thermal shocks without any deformation under the thermal conditions[4]. The composite materials which form oxides ceramic materials with high melting points are composed of such elements as magnesium, aluminum and zirconium with ferrous additives called cermet. Metal-based composite materials have been dealt with in several studies starting in the seventies, and they are now used in sporting goods, electronics packaging, plates, and the manufacture of moving parts, among others, Metal-based composite materials can be defined as a metal matrix containing three-dimensional inclusions. [5]. The current research aims to study the physical properties of a system based on copper metal reinforced with nan oxide in order to improve the properties of copper in terms of durability and hardness, which has multiple industrial applications.

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The practical part

1.The materials

The base material used was copper (Cu) with a grain size (325 mish) of Indian origin from the company (CDH) with a purity of 99.5%, and also a fixed percentage of SiC with a grain size (500 mish) of German origin was used from Fluka with a purity of 99.5%. The support material was nano-zirconia (ZrO_2, granular size 30 ± 5 nm), originating in Germany from (Changasha Santech Co.), with a purity of 99.94%.

2. Sample Preparation Method

The powders were dried at a temperature of (100 °C) for an hour to get rid of moisture and other volatile materials. After that, the mixture weight of each component was prepared by following the weight ratios so that the silicon carbide ratio became fixed at (5%) for all mixtures, while the zirconia rates were (0,5,10,15,20) %. Weighing was done using a Japanese-origin Sartorius type sensor with accuracy of (0.0001) grams and calibrated by the Central Agency for Standardization and Quality Control. After the completion of the grinding process in two hours and mixing and obtaining a homogeneous powder, samples were formed by means of (Uniaxial) pressing technology in hardened steel mold of (60HRC) hardness. The ground and mixture were carefully placed inside the press mold to prevent any movement of the mold parts. Then, in order to avoid the possibility of flexible return, 5Ton pressure was applied for one minute [6,7]. For this purpose, a hydraulic press of (HALIM USTA) Turkish origin with a press capacity of (20Ton) was used to obtain samples from cylindrical samples with a diameter of (10) mm and a height of (6) mm. After the pressing process, the samples were not ready for testing and had weak resistance, the green resistance, which required care when transporting and handling until the sintering process took place. The sintering process was carried out using a German-origin (CARBOLITE) furnace at a temperature of (900°C) for two hours.

Tests and Measurements

1.Vickers Hardness Test

Micro-hardness is one of the most important mechanical properties, defined as the surface indentation obtained by the material [8]. The hardness was checked using the Vickers method by inserting a square-footed diamond pyramid onto the prepared patterns. By shedding the tool with a mass (500 gm) and a time (10 Sec), and when calculating the diameters of the resulting trace on the surface, the hardness values of the

models can be known by applying the equation according to the following relationship (1):

$$Hv = 1.8544\, P/D_v^2 \ Kg/mm^2 - - - (1)$$

If: P = Immediate load (Kg)

D_v = the average diameter of the pyramidal effect resulting from the load being shed onto the surface.

2. The True Density Test

The most important metallurgical test for composites produced by powder metallurgy technology is the density test due to its importance and effect on densification. Green density is also one of the important properties of powders and its improvement helps in obtaining smooth and regular compositions that can be determined by the weight and dimensions of the compress based on the relationship below [9,10,11]:

$\rho = M / V$ ....(2)

)   Where: ρ represents green density (g / cm^3

M: represents the mass of compresses in grams (gm), V: the volume of compresses (cm^3)

3.The True Porosity Test

The true porosity test was performed based on Archimedes' principle following the international standard (ASTM C373 -88), and a sensitive electrical balance was used with an accuracy of (0.0001g). Where [8,12]:

Theoretical density is:

$$\rho_{th} = \sum_0^n (\rho i * Xi) - - - - - (3)$$

Since: - ρ_{th} = the theoretical density (g / cm^3), ρ_i = the theoretical density of the elements composing a single compressed (g / cm^3), Xi = the volume ratio of each component in the compressor

Green Density and Bulk Density are:

$$\rho = \frac{W_d}{W_s - W_i} * \rho_w - - - (4)$$

Since: ρ = green density representing the volumetric density (g / cm^3), = ρ_w the density of the liquid used (1g / cm^3)(

W_d = the dry weight of the pressed in grams, W_i = the suspended weight of the pressed in grams, W_s = the saturated weight of the pressed in grams

: The Apparent Density is

$$\rho_A = \frac{W_d}{W_d - W_i} * \rho_w - - - (5)$$

True Porosity is:

$$T.P \% = \frac{\rho_{th} - \rho}{\rho_{th}} * 100 - - - (6)$$

Since: T.P% = the percentage of true porosity, ρ_{th} = theoretical density (g / cm^3), ρ = green density or bulk density.

4. X-ray diffraction calculations:

The X-ray diffraction of the compressors was examined using a Dutch-origin X'Pert HighScore Plus device. The tube used was (Cu) kα, and the tests were conducted at room temperature. Table (1) shows the test data.

Table (1) specifications of the X-ray diffraction inspection device.

Voltage	40 KV
Current	30 mA
Scan Speed	5 deg/min
Scan Range	20$^{°}$ - 80$^{°}$
Wavelength	1.54060 A$^{°}$

We were provided with the interactions values of the atomic levels (d) for each compressor and the phases were obtained by comparing the values of the intervals (d) with the intensity of the rays and the angle (2θ) according to standard tables for the materials used in Miller's coefficients.

Results and Discussion

First: The effect of volume ratios and sintering on Vickers hardness

Figure (1) shows the relationship between the change in the size ratios of zirconia nanoparticles and the hardness of Vickers before and after the sintering process. It is noticeable that the hardness increases with the increase in the content of Nano scale zirconia. Furthermorem, the hardness increased before sintering from (38.4-54.3) and then decreased to (43). The content of Nano crystalline zirconia from (0-20)%, and the hardness increased after sintering from (58.75-90.81) and then decreased to (60.5) when the content of nano zirconia ranged from (0-20)%. The increase in hardness before and after sintering is due to the addition of zirconia nanoparticles despite the decrease in density and the increase in the porosity ratio to the high hardness of the zirconia nanoparticles. The abundance of interfaces was formed as a result of the addition of the reinforcing particles (SiC). The increased resistance to plastic deformation and internal stress remained due to the difference in thermal expansion coefficient between the base material and the hardening particles resulting in a lot of dislocations. Such dislocations led to an increase in the hardness of the composites since the high hardness nanoparticles of zirconia acted as obstacles to deforming the base material. The increase in hardness after sintering, however, is due to recrystallization, granular growth

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and removal. The stressesin the composites are because of the rise in the sintering temperature to 900°C for a period of two hours [13].
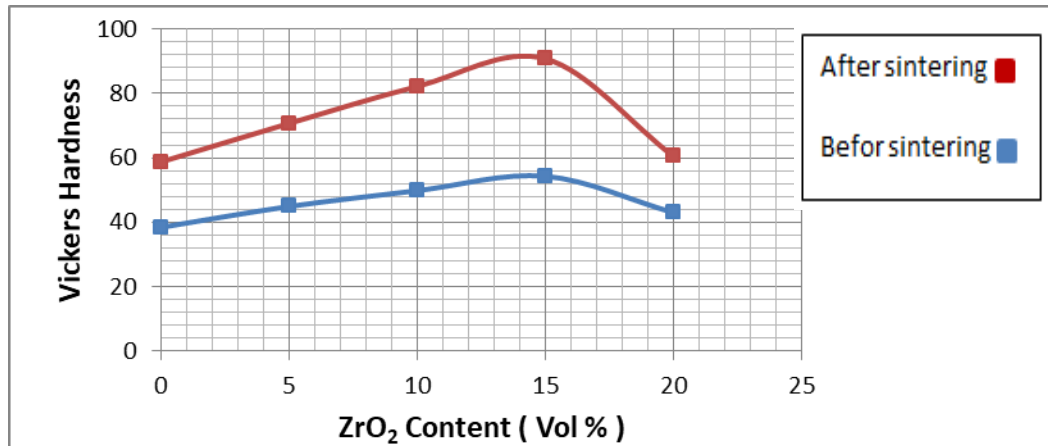

Figure (1) The relationship between the change in the size ratios of zirconia nanoparticles and the Vickers hardness before and after the sintering process.

Second: The effect of volume ratios and sintering on the true density

Figure (2) shows the relationship between the change in the size ratios of the zirconia nanoparticles and the green density before the sintering process. it also decreased to (6.652-5.493) g/cm^3 when the content of nanostructured zirconia increased from (0%) to (20%). The figure also shows the relationship between the change in the volume ratios of nanoparticles of zirconia and the bulk density after the sintering process at a temperature of 900 °C for a period of two hours. If decreased from (8.397-7.897) g/cm^3 to (7.01) g/cm^3,the content of nanostructured zirconia increased from (0%) to (20%). The decrease in green density and bulk density is due to the lower density of Nano scale zirconia compared to copper density. Therefore, it is normal for a decrease in the green density and bulk density of the (Cu-SiC-ZrO$_2$) compound when adding zirconia nanoparticles and this is consistent with what was reached in. It is also noted that the density values after sintering have increased due to the increase in the bond strength between copper and nanostructured zirconia as a result of the sintering temperature of 900 °C for a period of two hours, which in turn helps to improve the interface of the composites and the bonding between the particles. However, the cold compression process itself results in composites with a green density that is much less than the theoretical density, so it is logical that the bulk density is higher than the green one as the volumetric density approaches the theoretical

as a result of the occurrence of fusion between the particles of the superposition during the sintering process [14].
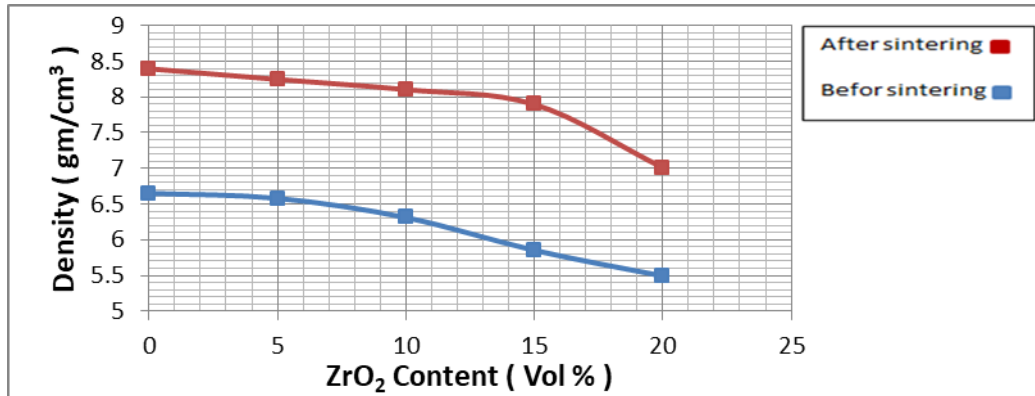

Figure (2) The relationship between the change in the size ratios of zirconia nanoparticles and the density before and after the sintering process.

Third: The effect of volume ratios and sintering on true porosity

Figure (3) shows the relationship between the change in the size ratios of the nanoparticles of zirconia and the real porosity before sintering. It is observed through the figure that the increase in the volume ratios added to the Nano scale zirconia led to a decrease in the true porosity, as the percentage of true porosity decreased from (35.729-20.809)%. Then, it increased to (30.606)% when the content of nanostructured zirconia increased from (0%) to (20%). The figure also shows the relationship between the change in the size ratios of nanoparticles of zirconia and the real porosity after the sintering process. This went up to (22.5)% when the content of nanostructured zirconia ranges from (0%) to (20%). The reason for the increase in true porosity by increasing the content of Nano crystalline at a ratio of (20%) is due to the fact that the porosity is the inverse result of density, and since the density decreases with the increase in the content of Nano scale zirconia, it is necessary that the pore ratio be high when increasing the content of Nano scale zirconia despite the lack of moisture between the metal. The basis and the hardening particles, in addition to the lack of complete fusion between the particles of the composites prior to sintering. Regarding the true porosity ratio after sintering and its comparison with its percentage before sintering, it is noted that the true porosity percentage has decreased after the sintering process, and this apparent decrease in the true pore ratio is due to the improvement of the adhesion between the base metal and the hardening particles during the sintering process in addition to the increase in the apparent density values. The hydrostatic stress works to close the pores

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during the plastic deformation between copper and zirconia. The sintering process has a major role in shrinking the size of the pores in the overlays and reducing it by increasing the fusion as a result of the diffusion process in the solid state this is supported by what is indicated by [13-15].
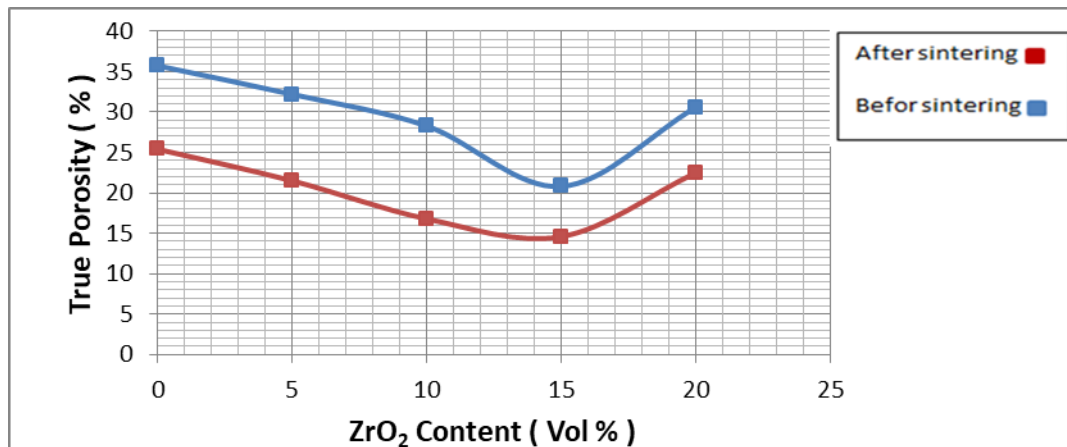

Figure (3) The relationship between the change in the size ratios of zirconia nanoparticles and the true porosity before and after the sintering process.

Fourth: The Results of X-ray Diffraction

The study of research samples by means of x-rays is an important means to identify the nature and composition of composite materials. First, the phase changes that occur when changing the volumetric addition ratios from the bonding material are monitored. Second, The materials are compressed and treated thermally at (900 °C) for two hours. In figure (4), X-ray diffraction (XRD) was examined by the compositions and at different support ratios of Nano scale zirconia% (0,5,10,15,20) and after sintering. The result was as following, first, the base material (copper) at the international card number (Cubic , Cu card No. 96-500-0217) and a crystalline cubic phase at Miller's coefficients (111), (200) and (220) was appeared, and this is fully compatible with the international standard. Second, the fixed support material silicon carbide (SiC) at the international card number (Cubic, SiC card No. 96-900-8857) and the combination of two (Cubic) at Miller's coefficients (111), (200), (220), (311) and (222) was disappeared, and this is also completely compatible with the global measurement. Third, there is the emergence of the modified modulating zirconia Nano composite (ZrO_2) at the international card number (Monoclinic, ZrO_2 card No. 96-900-5834) and with a cubic (Monoclinic) composition at Miller transactions (011), (110), (1-11) , (111), (002), (10-2), (112), (013) where there is a rise in the tops of both the base

material and the reinforcing material as well as in the reinforcement ratios, which indicates the spread of the support material through the base material and more regularity in the atoms. This was confirmed by the improved mechanical and physical results with increasing volumetric reinforcement ratios[16].

Figure (4) X-ray diffraction of (Cu-SiC-ZrO₂) system at different support ratios and after sintering.

Conclusion
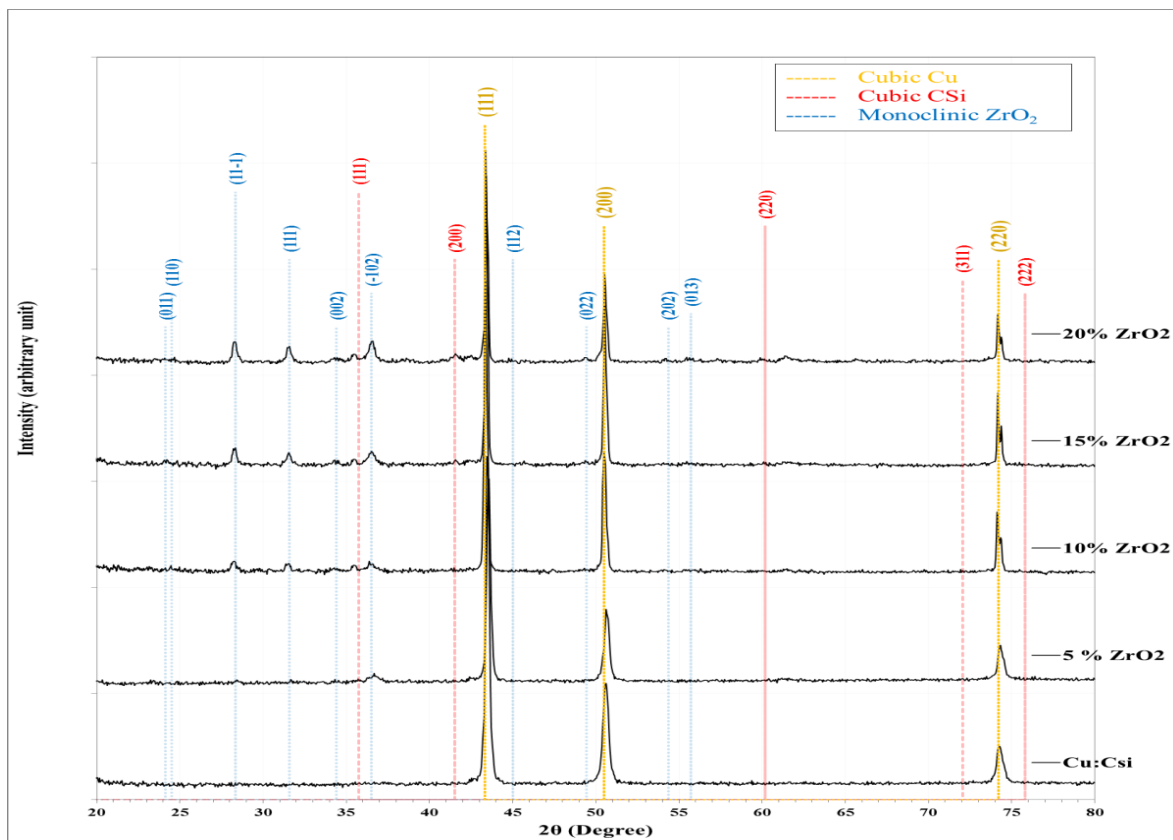
It can be concluded that it is possible to combine the three particles of the compound (Cu-SiC-ZrO₂) in order to obtain encouraging results such as a hardness that reached a maximum of (15% ZrO₂) with Hv (90.81), and a decrease in density. As a result of adding the supported nanomaterial, the true porosity was increased when the support percentage increased to 15% given the lowest at (14.6%) and the support ratio of 15%. The X-ray diffraction results showed that the materials used were of the base material (copper) with a cubic phase (Cubic) crystalline according to Miller's coefficients (111), (200) and (220), as well as the emergence of the fixed support material silicon carbide (SiC) and the structure of cubic (Cubic) at Miller's coefficients (111) and (200) and (220) and (311) and (222), as well as the emergence of

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the variable reinforced zirconia nanoparticles (ZrO_2) with a monoclinic structure at Miller's parameters (011), (110), (1-11), (111), (002), (102), (112), (013). We note the appearance of the materials that were used within the article, which are both the base material and the stiffeners, and at phases installed within the drawing of the X-ray diffraction .

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