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ORIGINAL STUDY

Calculation of Thermal Conductivity, Mechanical Properties Values at Adding Copper to a Base of Zirconia by Powder Method

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Abstract

Powder metallurgy has a wide range of industrial applications in the manufacture of tools, brushes, filters, and others. As a base of zirconia metal was used and supported by silicon at a constant rate of 3% Si with different reinforcement percentages of copper, which are (3,6,9,12,15)%. The pressing process was carried out at a pressure of 5 tons with a hydraulic press. A pressing mold with a diameter of 10 mm was used. The samples resulting from the press were sintered at 1000 °C for one hour, and the thermal, mechanical, and structural properties were calculated after sintering, which included thermal conductivity, thermal diffusion, heat flow, heat capacity, thermal resistance, Vickers hardness, compressive strength, wear, and scanning electron microscopy. Under ideal conditions of thermal sintering at 1000 °C, and a mixing ratio of 15%Cu, encouraging results were obtained, with a thermal conductivity of 66.34 W/m.K, a thermal diffusivity of 2.2416 mm²/sec, a heat flux of 420.424 W^{1/2}/m².K, and a heat capacity of 440.2512 J/kg.K, with a thermal resistance of 0.00103 m².K/W. While the Vickers hardness was 602Kg/mm², the compressive strength was 71 MPa, and the wear and tear was 3 × 10⁻⁸ g/cm. As for the synthetic results, which included the scanning electron microscope, it showed that the best homogeneity and reticular consistency was at the addition rate of 15%, and it was also noted that the spread of zirconia on the surface of copper was significant.

Keywords: Composite materials, Heat flow, Thermal resistance, SEM

1. Introduction

Powder technology has recently spread among industrial applications because of its advantages over other methods of manufacturing materials. This method is characterized by the production of materials with high resistance and does not require final operations, as is the case in methods of casting and welding. Through this technique, it is possible to manufacture models with international specifications and according to the actual need, as it was possible to design press molds with complex shapes and perform pressing and then thermal sintering of the resulting samples [1,2]. It is also possible through this method to perform pressing and pressing operations in the presence of heat or cold, according to the application used, because there are some applications that need complex shapes, such as those found in

internal combustion engines and other engines. Powder mixing requires a statistical methodology before selecting the appropriate type of equipment to be designed [3]. Only in this way is a satisfactory result obtained with the distribution among the components of the mixture as close as possible to the ideal, since the mixing process is an essential part for the production of cermet compounds that meet the purpose used [4]. The quality and characterization of the mixture require taking several samples and randomly analyzing them to find out the type of materials and their basic characteristics [5]. Before starting the powder mixing process, classification and possibly separation of particles must be carried out, as the sifting process is to separate a mixture of particles of different sizes into two or more parts, through the sifting surface, which acts as a scale that allows particles of certain sizes to pass only. The finished pieces consist of

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particles of more uniform size than the particles of the original mix [6,7].

Powder metallurgy is generally characterized by the fact that it is possible to mix powders with different bases, including metal-based materials, ceramic-based materials, and even polymers, carbides, and glass [8]. As for the reinforcement materials, they may be a mixture of two materials or one material from the same base or something else, and the best feature of the reinforcement materials is the combination of metal-based particles supported by ceramic materials, or vice versa, as materials are a mixture between two powders of ceramic and metal, which are known as materials cermet compound. There are three basic mechanisms in mixing solids: diffusion, convection, and shear. Shear can be thought of as convection, and effective mixing through diffusion and convection mechanisms must be combined. It generates a pure diffusion process between the mixed powders however, the high mixing efficiency of individual particles occurs at a low rate [9,10]. The convection process is quick but less effective and shows ineffectiveness in the final mix. For solids, the diffuse mixture will only occur by mechanical activation, the particles will change their collective or individual relative positions, and particle separation may also occur, and this occurs when particles of different sizes, shapes, or densities are mixed [11]. A good mixture occurs when particles are homogeneous with each other. Determining and evaluating a powder mix is difficult, But a certain amount of solids measurements can help estimate the performance of the mixer. Therefore, the resulting powders can be subjected to many tests to determine the type of elements included, such as X-ray spectroscopy (EDX) [12]. The work aims to calculate the values of the structural results, thermal and mechanical results of a system based on a zirconia ceramic material supported by a fixed percentage of silicon and a different percentage of copper, and know the effect of the different addition of metal to the base material.

2. Raw materials

The materials mentioned in Table 1 were used according to the values of density, particle size,

Table 1. The raw materials used.

No.	Materials	Density g/cm ³	Purity %	Grin Size	Company	Origin
1	Zr ₂ O ₃	5.89	99.5	63 μm≥	Fluka	German
2	Si	2.32	99.5	63 μm≥	CDH	Indian
3	Cu	8.96	99.99	75 μm≥	CDH	Indian

purity, and origin, which were mainly made of zirconia and supported by a fixed primary material of silicon, and a variable secondary material of copper.

3. Practical part

The process of preparing the presses was carried out using the powder method, by mixing the three powders, which are the base of zirconia with percentages of (94,91,88,85,82)%, silicon as a primary support material with a constant rate of (3%), and the second support material of copper metal with a percentage of (3,6,9,12,15)%. Mixed powders are usually exposed to laboratory-acquired moisture, so this problem is eliminated by drying them in an oven at 100 °C for half an hour. After that, the pressing mold is prepared, which is made of reinforced iron with a diameter of 10 mm shown in Fig. 1. The powder is placed inside the mold, and the pressing process is carried out using a Turkish hydraulic press at a pressure of 5 tons. Fig. 2 gives the pressed samples after thermal sintering. The resulting samples suffer from weak structural structure and have a green density. They are heat treated using a kiln of Korean origin of the type Muffle at a temperature of 1000 °C for a time of one hour. The presses produced from the convection furnace need a simple mechanical process to prepare them for the thermal, mechanical, and structural tests. The pistons were taken according to each mixture of the three materials above, and the smoothing process was carried out using 2000-degree smoothing paper, then a polishing process with a special paste, and finally, they were placed with a special show solution to show the crystalline borders of the pistons. Also, the pistons must be kept clean and not be held by hand for fear of getting dirty or the arrival of other materials on their outer surface, which causes their appearance during the structural tests by the EDX examination. The tests



Fig. 1. The die used to press with a diameter of 10 mm.



Fig. 2. Samples obtained from 15% compression after thermal sintering.

carried out on the pistons included thermal tests of thermal conductivity, heat flow, thermal diffusion, heat capacity, and thermal resistance. The mechanical tests were Vickers hardness, radial compressive strength, and frictional wear. The structural tests were scanning electron microscopy (SEM) and X-ray spectroscopy (EDX), through which the basic elements involved in the crystal structure of the resulting alloy can be known.

4. Tests used

4.1. Thermal tests

Thermal conductivity is a property of a material that describes its ability to conduct heat. Thermal conductivity is measured in units of (w/m.k). In the past few years, a great deal of attention has been paid to thermal conductivity, especially in composite and thermally insulating materials, as well as in mineral-supported insulator composites, where the addition of minerals greatly improves the thermal, electrical, and mechanical properties of the composites [13,14]. The great difference in the values of thermal conductivity imposes the classification of objects into two categories, objects of poor thermal conductivity and objects of good thermal conductivity. What good conduction such as metals is used in the manufacture of walls that permeate heat such as radiators and heat exchangers, while the objects of poor conductivity, which are many and different, they are used to improve thermal insulation. In fact, there is no material that completely insulates heat [15].

The thermal conductivity can be calculated using the following relationship [16,17]:

$$Q = \frac{dH}{dt} = -\frac{\lambda A dT}{dx} \quad (1)$$

whereas:

Q: the amount of heat per unit of time (watt). H: temperature (J), . t: time (sec), . λ : thermal conductivity (w/m.k), T: temperature (K), X: height of test specimen (m), A: cross-sectional area of specimen (m²).

The equation for thermal diffusion (m²/s) δ is:

$$\delta = \frac{\lambda}{C_p \cdot \rho} \quad (2)$$

C_p: specific heat capacity (J/g.k), ρ : density of the sample (g/cm³).

And the heat flux relation \mathcal{E} (Ws^{1/2}/m².K) is:

$$\mathcal{E} = \sqrt{\lambda \cdot \rho \cdot C_p} \quad (3)$$

The R thermal resistance (m².K/W) is:

$$R_{thermal} = \frac{dX}{\lambda} \quad (4)$$

The device that was used to measure the thermal conductivity is Mathis (TCi) type (MPTS) depends on the level of transmission modulation source, that is, there is a sensor that reflects the heat between the two surfaces of the probe and the sample on one side. The thermal conductivity and heat flow are measured directly, giving a detailed presentation of the thermal properties of the sample materials, which may be in the form of solids, liquids, powders, or paste [18,19]. The Mathis TC i device also provides multiple graphs and tables directly from during its programme. The rest of the thermal properties can be calculated by mathematical relations, as the device gives the following properties:-1 Heat flow 2- Thermal diffusion 3- Thermal conductivity 4- Heat capacity 5- Thermal resistance 6- Heat transferred per unit time.

4.2. Mechanical properties

Mechanical tests were carried out, which included micro-Vickers hardness, compressive strength, and frictional wear. Where the mechanical tests are considered one of the most important parameters that give the resulting alloy specifications in terms of resistance, and the cohesion strength between the composites mixed [20]. The Vickers hardness device was used to calculate the hardness values for the compressed models, where a stitching tool was used from a high-hardness prism at an angle of 136° in the presence of an applied load, and the value of the impact diameter resulting from the stitching tool is calculated, through which the micro-hardness values can be known according to the relationship (5) [21], Different test areas for hardness are taken to take the average of the resulting value to reach the best hardness value.

$$HV = 1.854 \frac{P}{d_{av}^2} \quad (5)$$

Where: H_V : Vickers hardness, P : shed load (N), d_{av} : mean impact diameter.

While the values of the diametrical compressive strength were calculated using the pressure method and in mega Pascal units, where the samples or pistons are placed between the two bases of the device and pressure is applied to the samples until failure and fracture of the models occurs, the device will record the values of the relationship between stress and strain, and thus the compressive strength is calculated through the relationship below [22].

$$\sigma_D = \frac{2F}{\pi dh} \quad (6)$$

Where: σ_D : represents the compressive, strength in units of (MPa), F represents the amount, of force applied in units of N, d : represents the diameter, of the pressed unit in (mm), h : represents the height, of the pressed unit in (mm).

As for the frictional wear rate, it can be calculated using a disc-on-disc method, where a constant load of 20 N is applied to the samples, and the disc rotates at a constant speed of 200 m/s. The weight of the pistons is calculated before and after operation. By identifying the difference between the two weights and the relationships below, the wear rate can be calculated [23,24].

$$\text{Wear Rate} = \frac{w_1 - w_2}{d} \quad (7)$$

Where: w_1 = Weight Before Work, w_2 = Weight After Work, d = Distance Traveled by the Disk During the Test.

$$d = 2\pi r n \quad (8)$$

Where: r = The radius of the Coating Samples, n = The Number of Turns.

4.3. Compositional tests

Structural tests including scanning electron microscope and X-ray spectroscopy were carried out to identify the surface morphology and surface composition of the prepared pistons and to identify the smoothness and consistency of the surface. Electron microscopes are characterized by the ability to magnify atoms thousands of times compared to optical microscopes. French TESCAN, model MIRA3.

5. Findings and discussions

5.1. Scanning electron microscopy and X-ray spectroscopy

Fig. 3 represents the results of scanning electron microscopy and X-ray spectroscopy, with a depth of (10 μm) and a magnification (500KX), as the figure gives pictures of each concentration of added copper and its apparent effect on the surface of zirconia, as shown by the fusion of the three granules with each other, forming one integrated alloy. Also, the addition of the metal led to a widespread through the ground of the ceramic material of zirconia, and we also found that the spread of copper and the presence of high temperatures led to a large overlap between the voids and pores formed between the ceramic material because one of the characteristics of minerals is their vulnerability to temperatures, especially temperatures up to 1000 °C. We find that the best percentage of certain homogeneity is at 15% of copper, and the extent of homogeneity and cohesion between the three components becomes clear, this is because the minerals have an effect on diffusion with each subsequent increase, and this effect extends to include thermal and mechanical examinations of them [25], so we find with each addition an improvement. It is evident in the thermal conductivity, which in turn gives a positive effect to the rest of the thermal parameters, and the hardness increases and the wear rate decreases [26].

5.2. Thermal tests

Figs. 4–8 give the thermal properties, which included thermal conductivity, thermal diffusion, heat flow, heat capacity, thermal resistance with copper percentages added, respectively, and after thermal sintering at 1000 °C. As we note that there is a significant improvement in the thermal properties through the continuous additions of the metal to the ceramic material of zirconia in volumetric proportions due to the difference in densities between the stamped elements, that this improvement came as a result of adding a material with a body thermal conductivity, which is copper, to a material with very little thermal conductivity, which is zirconia. Also, the thermal sintering, which greatly contributed to the increase in the fusion of particles and the convergence of atoms, which improved the surface area of the presses, and thus there will be suitable distances for thermal conductivity. The pressing pressure at 5 tons also has a significant impact on the homogeneity and cohesion of the particles that will need a few distances for the purpose of reaching

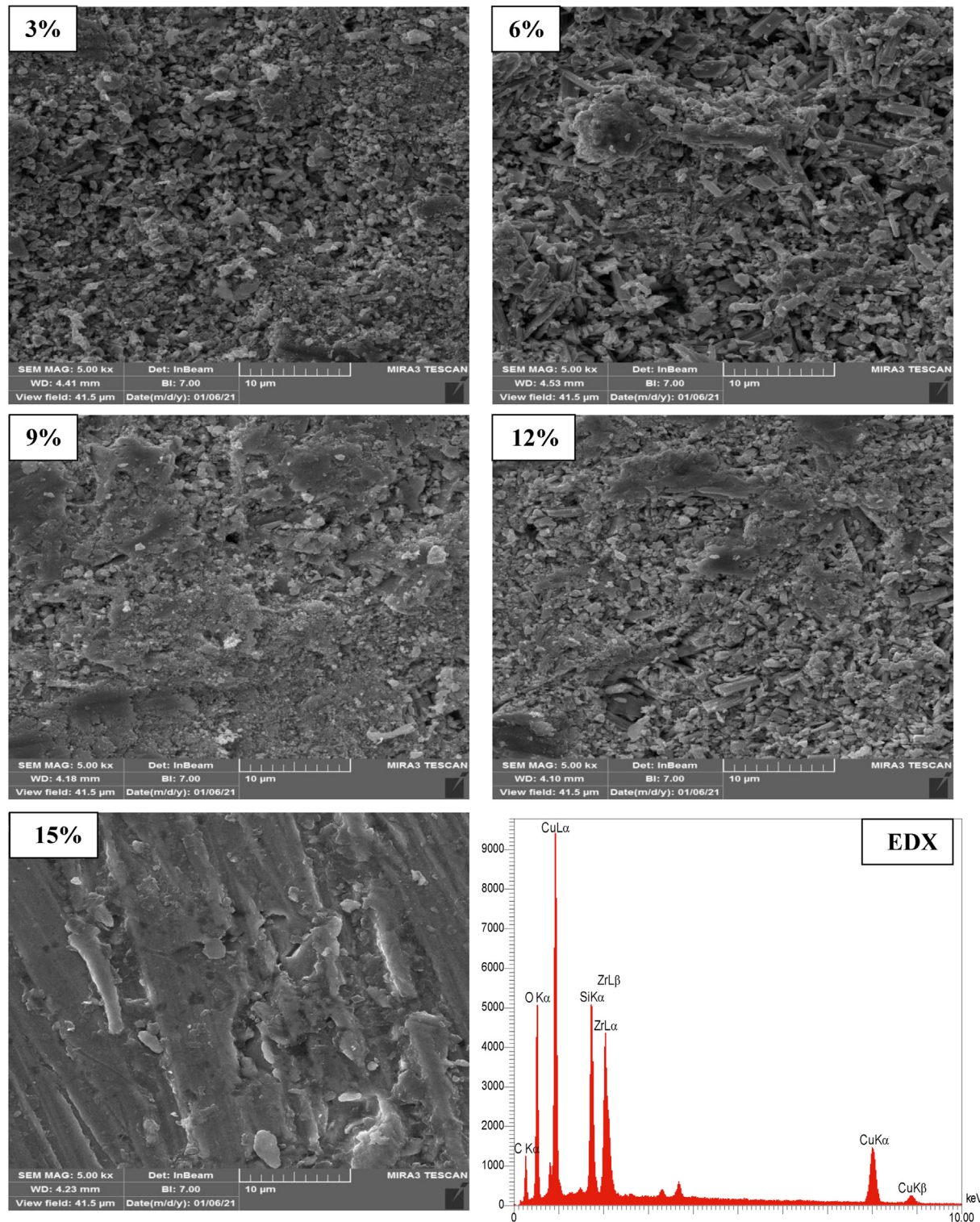


Fig. 3. Scanning electron microscopy and X-ray spectroscopy at reinforcement percentages (3, 6, 9, 12, 15)% of copper after thermal sintering.

a good state of thermal conductivity, and the decrease in thermal conductivity and its parameters at low reinforcement ratios is due to the lack of copper at these ratios and thus the increase in pores

and crystalline defects Which negatively affects the characteristics as a whole [27]. This is the case with the heat flow, which also improved with the increase in copper percentages, the best of which was

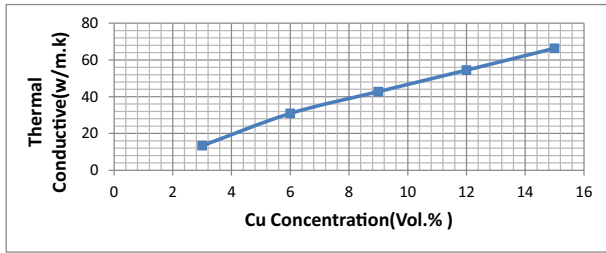


Fig. 4. The relationship between increasing the volumetric ratio of copper particles and thermal conductivity.

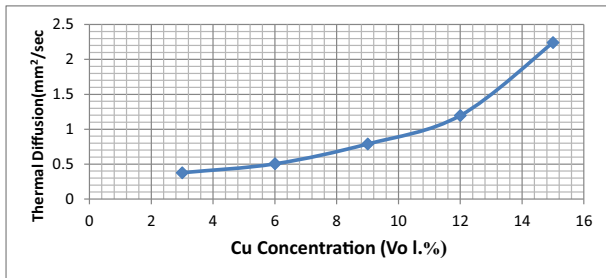


Fig. 5. The relationship between increasing the volumetric ratio of copper particles and thermal diffusion.

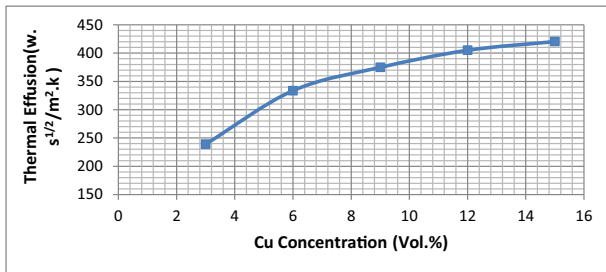


Fig. 6. The relationship between increasing the volumetric ratio of copper particles and heat flow.

at 15%, and this is similar to what was obtained with thermal conductivity. As for the thermal diffusion, it increased as shown in the scanning electron microscope, and a significant improvement with each increase of copper metal [28]. As for the results of

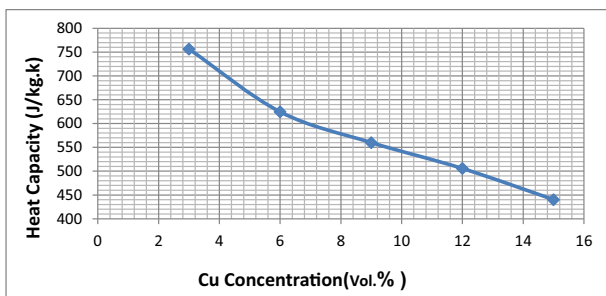


Fig. 7. The relationship between increasing the volumetric ratio of copper particles and heat capacity.

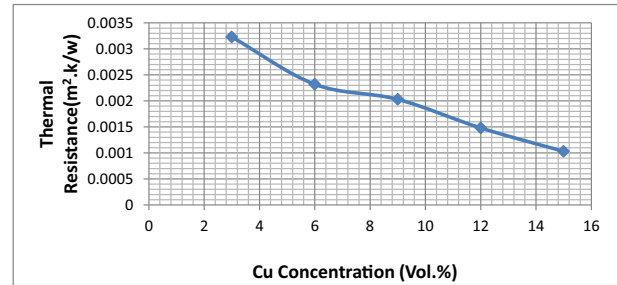


Fig. 8. The relationship between increasing the volumetric ratio of copper particles and thermal resistance.

the heat capacity, it showed a decrease in its amount. Thus, the system behaves like a semiconductor because the combination between metal and ceramic is similar to what happens in semiconductors [29]. As for the thermal resistance, we find that it takes a decrease in the system with each increase, because the insulators show resistance to heat transfer through them, but by increasing the content of the support material (copper), i.e. a conductive material that works to bridge the voids between the particles, which facilitates the work of a physical path for heat transfer through the compound, i.e. directly proportional to the porosity and inversely with thermal conductivity. Thus, the small proportions of the reinforcement material give weak thermal properties, and these properties and their parameters increase and improve with each increase of copper metal, to be the best at a rate of 15% of copper metal.

5.3. Mechanical tests

Figs. 9–11, which gives the relationship of each of the Vickers hardness, compressive strength, wear and tear with the added volumetric copper ratios after thermal sintering at 1000 °C, as we find through Fig. 9 that the hardness values gradually increase with each addition of the support material

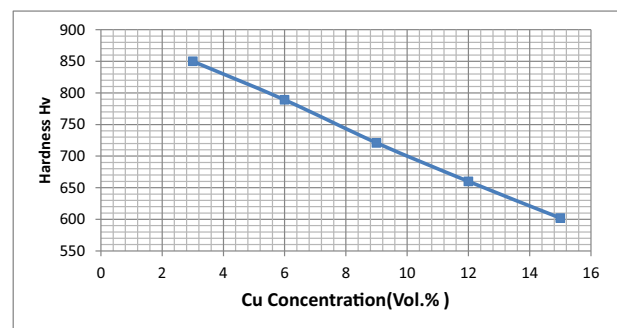


Fig. 9. The relationship between the increase in the volumetric ratio of copper particles and the Vickers hardness after sintering.

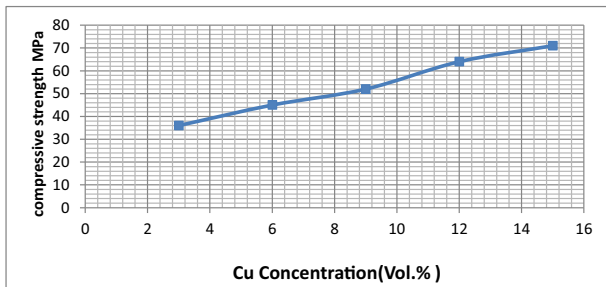


Fig. 10. The relationship between increasing the volumetric ratio of copper particles and compressive strength after sintering.

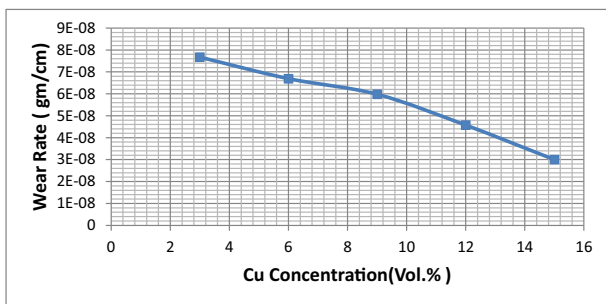


Fig. 11. The relationship between the increase in the volumetric ratio of copper particles and the wear rate after sintering.

of copper metal until reaching the maximum value of hardness with a reinforcement rate of 15%, and the increase in hardness is attributed to the effect of copper metal after sintering and rarefaction between the atoms of zirconia and silicon, thus forming a strong and cohesive alloy because the merging of the metal atoms with the rest of the compounds will give a cermet material with high hardness. The sintering temperature also has a clear effect by increasing the adhesion of particles with each other, which in turn produces hard and soft models that withstand shocks, to be used as an application in many applications such as cutting tools and some machines [30]. As for Fig. 10, we find that the diagonal compressive strength begins to increase with each volumetric addition of copper metal, thus we obtain the best values for the compressive strength at the best mixing ratio, which is 15% of copper, and this is due to the heat factor that affected all mechanical parameters where the materials suffer. Ceramic has a weak crystalline structure, thus adding minerals that have the ability to combine with ceramic atoms, thus forming an alloy with crystalline homogeneity and strong mechanical interlocking. As for Fig. 11, which gives the results of frictional wear after thermal treatments, and usually the wear values of metal reinforcement decrease with each increase, because the ceramic materials

suffer from an increase in wear values, but the addition of minerals works to strengthen the ceramic and reduce wear relatively [31].

6. Conclusions

The conclusion in the article deals with the most important thermal parameters that have been improved with the continuous additions of copper to the system (Zr_2O_3 –%Si), where the thermal conductivity values have improved at the ideal parameters, which are thermal sintering at 1000 °C for a time of one hour, and at a cementing ratio of 15%, and pressing 5 tons, it was found that the thermal conductivity is 66.34 W/m.K, the thermal diffusivity is 2.2416 mm²/s, the heat flow is 420.424 W^{1/2}/m². K, the heat capacity is 440.2512 J/kg.K, and the thermal resistance is 0.00103 m² K/W. While the Vickers hardness was 602 kg/mm², the compressive strength was 71 MPa, and the wear and tear was 3×10^{-8} g/cm. As for the synthetic results, which included the scanning electron microscope, it showed that the best homogeneity, lattice consistency, and crystal overlap were at the same ideal parameters above, while the X-ray EDX spectroscopy showed the appearance of all the superimposed elements of zirconia, silicon, and copper.

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