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The effect of adding nano-nickel oxide on the physical properties of (Al-SiO_2) by powder method

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Original Research

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Abstract:

There are a wide range of methods used to manufacture the materials, and one of these methods is powder technology, which is usually easy to manufacture at the lowest possible cost. Aluminium was used as a base material with a percentage of 3% wt. SiO_2, and nano-nickel oxide was added in different volume ratios, namely (2, 4, 6, 8, 10)% wt. The different powders were mixed using a locally made powder, and a pressing mould with a diameter of 10 mm was prepared. The mixed powders were pressed using a hydraulic press at 5 Ton. The samples resulting from the pressing process were heat treated in an oven at a temperature of 600 °C for two hours. A number of physical tests were conducted on the resulting samples before and after thermal sintering, including scanning electron microscopy (SEM) and X-ray diffraction spectroscopy (EDS). The true density, true porosity and water absorption were also tested, as well as the hardness, compressive strength and wear rate. The results showed that the best mixing ratio is 10% wt. before and after thermal sintering, which gave high surface homogeneity, hardness and compressive strength with the lowest wear rate. We also found an increase in the values of true density and porosity with a decrease in water absorption.

Keywords: Adhesive strength; Cermet; SEM; Silica; Wear rate

1. Introduction

Powder metallurgy plays an important role in the manufacture of materials due to its many applications, especially in space, aviation and the manufacture of various parts [1]. This technology has entered the manufacture of electronics that require high-precision materials, as is the case with shape memory alloys [2, 3]. There are many methods through which materials can be manufactured, for example, the casting method, which provides products with high hardness and quality. However, the resulting samples require subsequent machining processes, which reduces its importance compared to the powder method, as the samples do not need to be modified or coordinated in the external shape but are regular and of equal dimensions [4–6].
This technology is of great importance and is considered a new innovation in the science of engineering materials, as it can produce different alloys at high speeds and low cost [7]. In addition, the resulting samples are characterised by high strength in terms of hardness, resistance to compression and shocks, and a relatively low wear rate. The mechanical properties of products can be improved by thermal sintering, which effectively contributes to raising the mechanical and structural properties of these materials [8]. In the field of aviation, powder technology has emerged effectively as it was of low economic cost, such as in the manufacture of turbine blades that can withstand high temperatures and are resistant to external conditions [9, 10]. This technology is also characterised by light weight, which contributes to reducing the weight of some structural parts in aircraft. This in turn contributes to reducing the financial cost during maintenance and in the long term [11].
Powder metallurgy has historically developed since the beginning of the nineteenth century, when powders were used to manufacture many materials, such as pottery and tools, gradually developing and entering the field of metallurgy [12]. This development also contributed to the manufacture of alloys and materials based on pressure and thermal sintering. Therefore, this method improved to include a wide range of manufacturing, such as 3D printing and the manufacture of some parts of aircraft and cars [13]. The development of materials continued to enter the field of nanotechnology, and many metals such as nickel, bronze and iron emerged here. In the twentieth century, powder metallurgy became an integral part of the aviation and alloy

industries [14].
The importance of powder metallurgy in terms of modern innovations has had a great impact on medicine, space and aviation [10]. It has also played a significant role in the manufacture of metal and cermet alloys, which are a mixture of ceramics and metals [15, 16]. The strength and hardness of metals such as aluminium, nickel, copper, graphite and others can be increased by adding metal oxides to them, thus creating a cermet alloy with properties and specifications that are not found in both metals and oxides [17, 18].
The current research article aims to improve the properties of aluminium by adding silica at a fixed rate of 3% wt., in addition to reinforcing the base material with nano-nickel oxide. The properties required to be improved are mechanical, structural and physical.

2. Materials and method

The base material was aluminium from Oxford Lab. Chem. with a particle size of 63 µm and purity of ($\geq 99.4\%$), while the reinforcement material was silica from Sigma-Aldrich with a particle size of 75 µm and purity of ($\geq 99.8\%$), and the second reinforcement material was nano-nickel oxide from Changsha Santech Co. with a particle size of (30 ± 5 nm) and purity of ($\geq 99.8\%$).
The above powders were mixed in volumetric ratios due to the different densities between the materials. The ratios for the base material of aluminium were (95, 93, 91, 89, 87)% wt., while the first reinforcement material was silica with a fixed ratio of 3% wt. and the second variable reinforcement material was nano-nickel oxide with ratios (2, 4, 6, 8, 10)% wt. The powder mixing process is essential to ensure the homogeneity of the mixtures at each ratio. For this purpose, a locally manufactured micro mill was used. To compress the powders, a Turkish-made (HALIM USTA) hydraulic press with a maximum pressure of 20 Ton was used. The powders were compressed at each ratio at 5 Ton for one minute. The pressing mould was prepared with a diameter of 10 mm and made of high-pressure steel. The samples, which have a weak structure and need thermal sintering to strengthen the structural structure, were pressed using a Korean-made (Muffle) thermal furnace. The sintering was at a temperature of (600 °C) for two hours. The samples were left inside the furnace until the next morning to eliminate any possible thermal stress and complete the stability of the thermal sintering and thermal distribution. Then, the samples were taken out and thoroughly cleaned of any laboratory contaminants to be ready for structural, mechanical and physical tests.

3. Theoretical part

The structural tests used included scanning electron microscopy (SEM) and X-ray diffraction spectroscopy (EDS), where a MIRA3 TESCAN device was used, which has a radiation stress range of $(0.1-30)$ Kv and high magnification ranges of up to one million, with high-resolution image quality.
The mechanical tests included micro-Vickers hardness using a French-made device with a probe angle of 136°, with a pressure force of 10 kg. The hardness was tested on five different areas of the sample surface before and after thermal sintering. Equation (1) was used to calculate the hardness values H_V [19].

$$H_V = \frac{\sin(\frac{136}{2}\cdot)2F}{d^2} \tag{1}$$

where H_V: Vickers hardness (Kg/mm^2), F: Applied force (Kg), d: Diameter of the trace produced by the diamond probe (mm^2).
The compressive strength values were calculated by an American-made device, where the sample was placed between the jaws of the device and pressure was applied to it until the sample failed, i.e. it broke completely or partially. The test was repeated a number of times to ensure the accuracy of the results obtained. Relationship (2) was used to calculate the amount of compressive strength [20, 21].

$$\text{Compression strength}(\varepsilon) = \frac{F}{A} \tag{2}$$

where ε: Compressive strength (MPa), F: Applied load force (N), A: Sample surface area (m^2).
While the sliding wear rate was calculated using the pin-on-disc method with a Swedish device, the samples to be measured for their wear rate were weighed before being placed in the device, and this represents the first weight m_1. After placing them in the device and applying a load of (25 N) to them via an upper disc holding the sample and a lower disc rotating at a speed of 350 rpm and rotating for 10 minutes, the sample was extracted to represent the second weight m^2. The relation (3) was used to determine the value of the wear rate, and the value of the distance travelled by the disc during rotation (r) was also determined using the relation (4) [22, 23].

$$\text{Wear rate}(W) = \frac{m_1 - m_2}{r} \tag{3}$$

where W: Frictional wear rate (g/cm), m_1, m_2: Weight of the sample before and after work (g), r: Distance the rotating disc moves (cm).

$$r = 2\pi an \tag{4}$$

where a: Radius of the piston (model) (cm), n: Number of revolutions the disk rotates.
Some physical properties were calculated to include (true density, true porosity, water absorbency), and for this purpose Archimedes' method was used in the laboratory, where the samples were weighed when they were dry it is m_d. Then, the samples were immersed in distilled water for 24 hours and weighed after removing them from the water to represent the weight of the sample saturated with water with the symbol ms. Next, the samples were immersed in distilled water (dry samples) and suspended in a sensitive balance and weighed while suspended to represent the symbol m_i. After that, the true density, true porosity and water absorbency were calculated with the relations (5)-(7) respectively [24–26].

$$\text{True density}(\rho) = \frac{\text{Mass material}(M)}{\text{Volume of compressed powders}(V)} \tag{5}$$

$$\text{True porosity}(\%) = \frac{\text{T.D.} - \text{B.D.}}{\text{T.D.}} \times 100\% \tag{6}$$

$$\text{Water absorption}(\%) = \frac{m_s - m_d}{m_s - m_i} \times 100\% \tag{7}$$

where M: Represents the weight of the compressed particles (g), V: The volume of the compressed particles (cm^3), T.D.: The theoretical density (g/cm^3), B.D.: The bulk density (g/cm^3), m_s: The weight of the sample saturated with distilled water (g), m_d: The weight of the dry sample (g), m_i: The weight of the suspended sample (g).

4. Results and discussions

The results were obtained through the theoretical part above based on the change in the percentages of the added material of nano-nickel oxide before and after thermal sintering. The scanning electron microscope (SEM) test was conducted for the samples prepared after thermal sintering and at all added percentages as shown in Fig. 1 (1-a) represents the percentage of adding 2% wt. of nano-nickel oxide, Fig. 1 (1-b) is 4% wt., Fig. 1 (1-c) is 6% wt., Fig. 1 (1-d) is 8% wt. and Fig. 1 (1-e) is 10% wt., respectively. We found that after each addition of the nanomaterial there is an improvement in the surface structure of the composite, as there is a spread of oxide particles across the aluminium surface in a uniform manner, and this spread increases gradually with each addition. Therefore, this interaction between the composites improves the physical properties of the pressed alloy. We found that the best mixing ratio that gave a consistent and homogeneous surface is at 10% wt., which indicates an improvement in the properties, including the mechanical ones, as the nano oxide contributed to enhancing the hardness and compressive resistance, and the effect of the nanomaterial is an effect in increasing the surface area of interaction between the components. As for the reasons for the pores that appear as in sample Fig. 1 (1-b), they can be attributed to the result of the interaction between nickel oxide and aluminium. In general, the addition of nano-nickel oxide contributed to increasing the surface texture and smoothness with each addition, to be the best at Fig. 1 (1-e), and this indicates that the addition of nano to aluminium helped to increase the surface smoothness, which in turn reduces the friction values. This is especially true when calculating

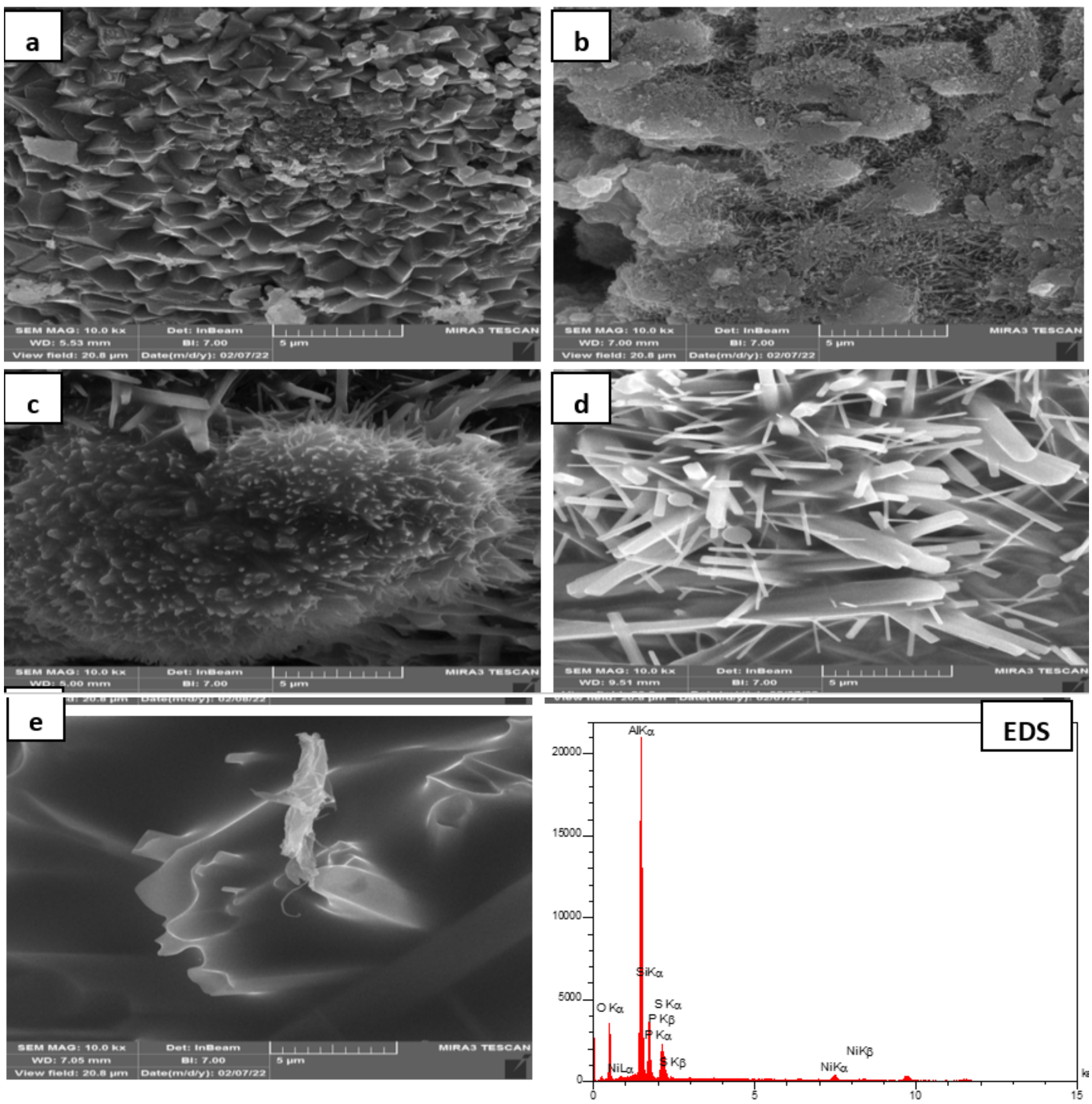

Figure 1. Scanning electron microscope (SEM) and X-ray diffraction spectroscopy (EDS) images after sintering and at (a-2, b-4, c-6, d-8, e-10) %wt.

the wear rate, as the smooth surface reduces frictional wear and any possible deformations, unlike the rough surface, which in turn affects the hardness and compressive strength [27–30]. In Fig. 1 (1-EDS), we can see a clear appearance of the materials used for each of aluminium, silicon and nano-nickel oxide, which gives an idea of the purity of the materials used in the practical aspect, as any impurities negatively affect the properties of the pressed materials [31].
The results of the Vickers hardness values were calculated according to the relation (1) and as shown in figure 2 before and after sintering and for different reinforcement ratios of nano oxide. It was found that the hardness value was from (30.1 kg/mm^2) to (135.5 kg/mm^2) at a reinforcement ratio of (2% wt.) to (10% wt.) respectively before sintering, while the hardness was from (55.1 kg/mm^2) to (148.9 kg/mm^2) at a reinforcement ratio of (2% wt.) to (10% wt.) respectively after sintering. We found that there was an increase in hardness values with the increase in the percentages of different nano additions before and after sintering. This increase is attributed to the fact that nanomaterials have a significant effect on increasing the interatomic reinforcement of the composite as well as forming bonding areas between nano atoms and aluminium atoms, therefore increasing strong bonds, including mechanical ones, which contributes significantly to increasing hardness values. Thus, nano works to prevent any sudden movements within the crystal lattice of the base material, which reduces any possible deformations and increases the hardness percentage [32, 33]. We also found that the effect of sintering has a clear change in increasing hardness values, because sintering works to reduce pores, which are considered a crystal defect, and heat also works to change the mineral phases, which may produce materials with different compositions [34].
The compressive strength test was also conducted for the pressed samples, where we found from figure 3 the relationship between the compressive strength and the reinforcement values of different nano-nickel oxide before and after sintering. So, we found that the compressive strength was (51 – 90 MPa) when reinforced (2 – 10% wt.) before sintering while the results after sintering were (75 – 115 MPa) when reinforced (2 – 10% wt.). Therefore, we noticed a clear increase in the compressive strength values with each addition of nano-nickel oxide, especially at the best of them, 10% wt., and this is attributed to the importance of the nanoparticles spread throughout the aluminium matrix. These, in turn, act as reinforcement and cohesion particles for the system, as nano has an effect in terms of the small size of the grains that work to distribute any potential stresses throughout the system, reduce porosity and increase density, thus improving the physical properties significantly [35, 36]. Thermal sintering also has an effect on improving the structural properties and transforming the phases into phases with high hardness and compressive strength [37].
The friction wear rate values were also calculated within the mechanical properties using the relations (3), (4), as we notice in figure 4 the relationship between the friction

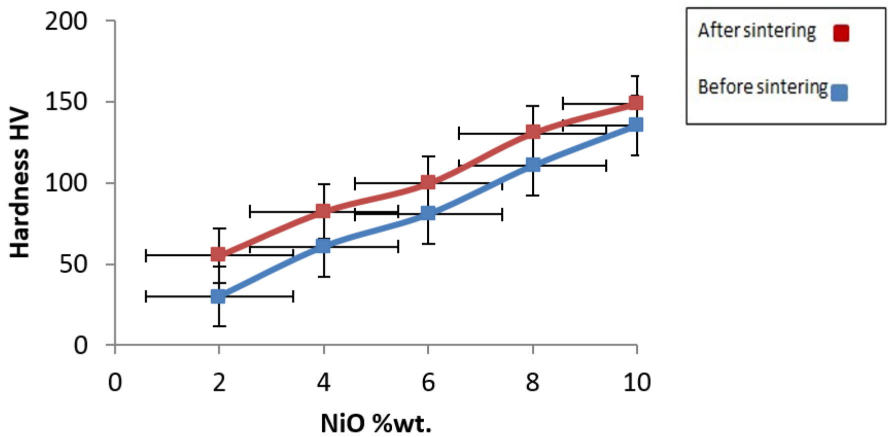

Figure 2. The relationship between Vickers hardness and the percentage of added nano-nickel oxide before and after sintering.

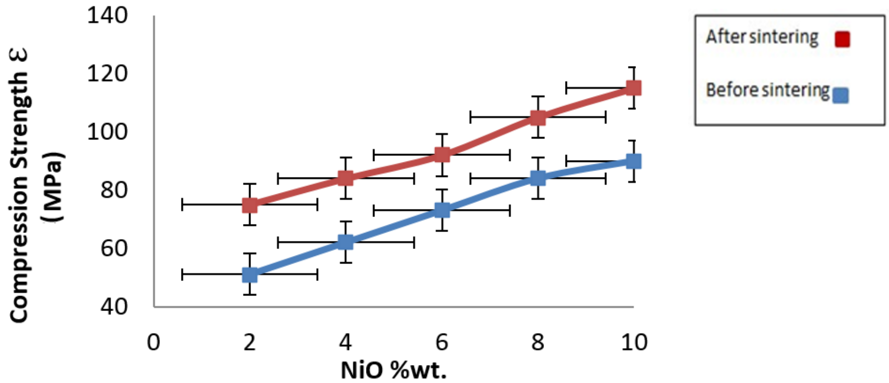

Figure 3. The relationship between compressive strength and the percentage of added nano-nickel oxide before and after sintering.

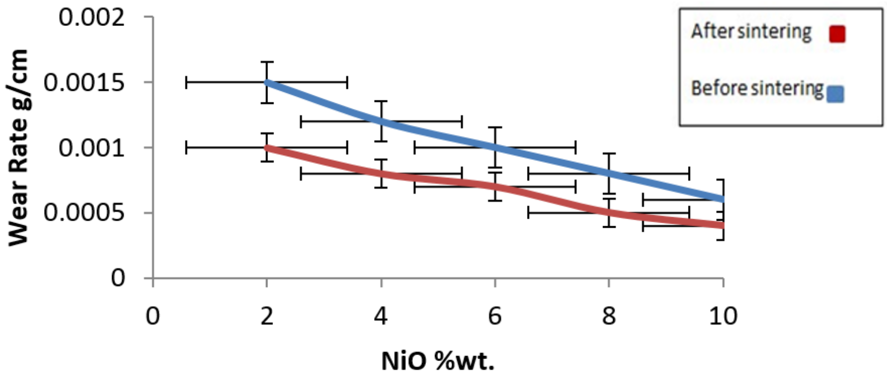

Figure 4. The relationship between the friction wear rate and the percentages of added nano-nickel oxide before and after sintering.

wear rate and the percentages of nano-nickel oxide before and after sintering. We found that the value of the wear rate before sintering is (0.0015 – 0.0006 g/cm) at percentages (2 – 10% wt.), while the wear rate after sintering was (0.001 – 0.0004 g/cm) at percentages (2 – 10% wt.). Therefore, we found that the behaviour of the wear rate results is similar to what we obtained from the results of hardness and compressive strength, because the effect is similar to the nanoparticles in terms of their effect on the composite and improving its resistance to any crystalline stresses. Also, nano-nickel oxide has an effect on improving the surface properties in terms of increasing smoothness and surface resistance to external influences, thus improving the system as a whole in terms of corrosion resistance and reducing the wear rate values [38]. The thermal effect on the wear rate is clear from the decrease in the wear rate values after thermal sintering [39]. This can be attributed to the fact that the high temperature on the particles contributes to a mechanical interaction between the nanomaterials and the base alloy, which in turn produces strong mechanical bonds that contribute to reducing the wear rate [40, 41].
The mathematical relationship (5) was also used to calculate the values of the true density results, as we find in figure 5 the relationship between the true density and the values of the different additions of nano-nickel oxide before and after thermal sintering. We found that the true density values are (2.3 – 3.4 g/cm^3) at nano-reinforcement ratios (2 – 10% wt.) before sintering, while the density was (2.85 – 3.85 g/cm^3) at (2 – 10% wt.) reinforcement after thermal sintering. This increase is clearly attributed to the fact that nano-nickel oxide has a high density, i.e. higher than aluminium and silicon oxide, so any addition works to increase the true density value of the composite as a whole, and the small size of the nano-granules will work to block the largest possible number of pores, which results in an increase in the density values [42]. Also, the thermal effect by sintering works to reduce the pores, which is the main factor in increasing the density, thus increasing the density of the total alloy after thermal sintering [43]. Another important factor to improve the density values is the way the three powders are mixed together, which in turn contributes to the even distribution of nanoparticles within the aluminium granules [44]. High density values improve the mechanical properties of the composite in terms of bearing loads and high pressure [45]. The true porosity was calculated from the relationship (6), while figure 6 gives the relationship between the actual porosity and the different addition ratios of nano oxide before and after sintering. We found that the porosity before sintering is (8.56 – 17.97%) at nano reinforcement ratios (2 – 10% wt.), while after sintering it was (6.78 – 13.24%) at reinforcement ratios (2 – 10% wt.). The presence of pores within the alloy is considered a structural defect and greatly affects the mechanical properties because it represents the number of voids or pores relative to the total volume of the alloy, and its increase leads to failure of the alloy [46]. Therefore, we found that adding nano helped to reduce the

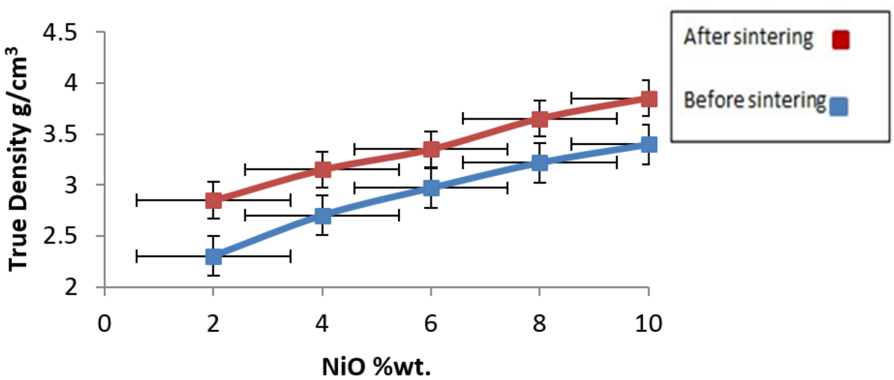

Figure 5. The relationship between the true density and the percentages of added nano-nickel oxide before and after sintering.

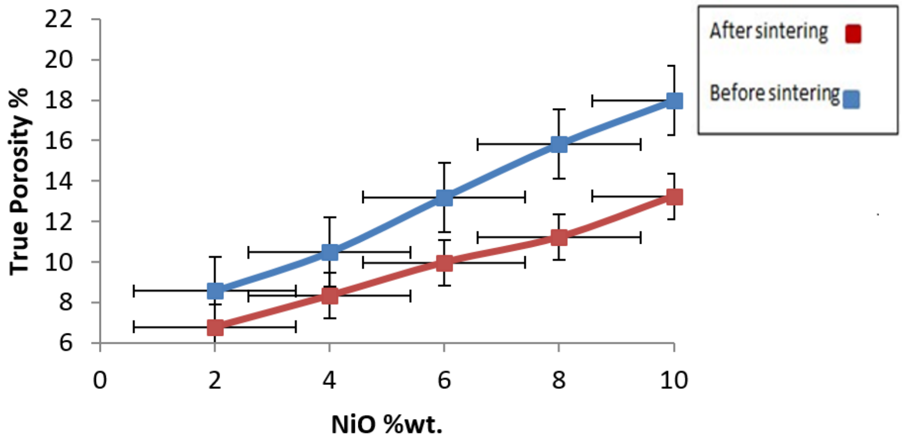

Figure 6. The relationship between the true porosity and the percentages of added nano-nickel oxide before and after sintering.

pore ratios by building molecular connections between the interacting particles, which in turn reduced the presence of interstitial voids or resulting gaps. Sintering also has an effect on improving and reducing the pores by increasing the atomic cohesion and strengthening the structure of the alloy, which in turn reduces the pore ratios throughout the entire system [47, 48].

Water absorbance was also calculated according to the relationship (7), as figure 7 shows the relationship between water absorbance and the reinforcement ratios with nano-nickel oxide before and after sintering. We found that the absorbance value is $(0.56-1.1\%)$ at reinforcement ratios $(2-10\%$ wt.) before sintering, while the absorbance was $(0.43-0.97\%)$ at reinforcement ratios $(2-10\%$ wt.) after thermal sintering. Therefore, it is clear that the behaviour of the alloy resulting from pressing and after thermal sintering is the best in terms of low water absorption ratio, which indicates that the alloy behaves like a vitrified behaviour, i.e. the absorption ratio is less than 1% [49]. This shows the effect of adding nano-nickel oxide on forming a layer that prevents water from entering the base material of aluminium and silicon oxide, as the nano contributed effectively to increasing the density of the alloy as a whole, so this increase in density values with the presence of pressure and temperature worked to fill the generated gaps and reduced the alloy's water absorption ratio [50**?**]. Nano composites also have the advantage of producing hydrophobic surfaces, i.e. water-repellent, which means that the surface will absorb less water [51].

5. Conclusion

The important conclusion of the work is the possibility of compressing nano powders with micronisation in the (Al-3wt.%SiO_2-NiO) system, as the addition of nano has a great effect on improving the structural properties as well as the mechanical and physical properties. The values of density, hardness and compressive strength improved significantly with the addition of nano-nickel oxide, while the values of friction wear rate, porosity and water absorption decreased with each addition, and the best results were at 10% wt. after thermal sintering at 600 °C for two hours. Also, the surface structure improved and became more homogeneous and interlocked with an increase in its smoothness, as shown by the results of the scanning electron microscope (SEM).

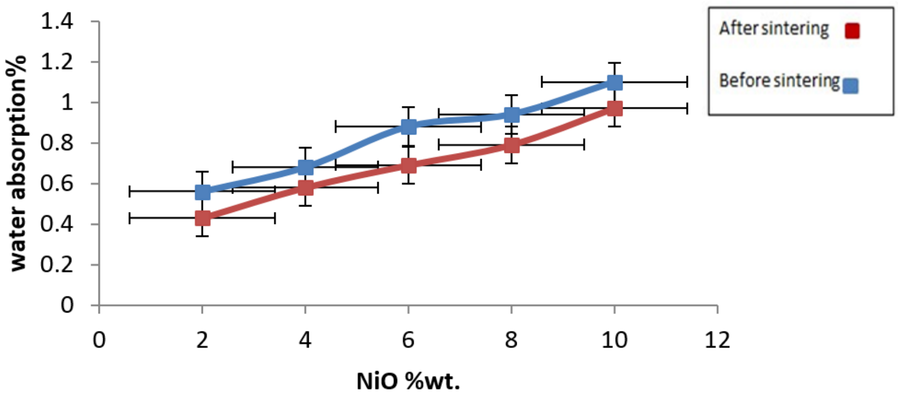

Figure 7. The relationship between water absorbency and the percentage of added nano-nickel oxide before and after sintering.

Authors Contribution
Zuheer N. Majeed and Salih Y. Darweesh wrote the main manuscript text and Salih Y. Darweesh prepared all figures. All authors reviewed the manuscript.

Availability of data and materials
The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflict of interests
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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