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The Effects of Nano Titanium Oxide Addition on the Structural and Mechanical Properties of a Ni-Al₂O₃ System Using the Powder Method

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تأثير اضافة اوكسيد التيتانيوم النانوي بطريقة المساحيق على الخصائص التركيبية والميكانيكية لنظام (Ni-Al₂O₃)

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KEYWORDS

الكلمات المفتاحية

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ABSTRACT

The powder method is a modern technique used in the manufacturing of composites. Thus, it was employed in this study to prepare composite samples from nickel as a matrix, with a constant alumina content of 10% and titanium oxide (TiO₂) with different proportions (0, 5, 10, 15 and 20%). The powders were milled and stirred for one hour using a homemade grinder containing steel balls. Afterward, the samples were compacted at a pressure of five tons for one minute. The prepared samples were sintered at 1300°C for two hours. Tests with the scanning electron microscope (SEM) on mechanical properties (hardness and diagonal compressive strength) and some physical properties (density and porosity) were conducted before and after sintering. The SEM results show a clearly defined microstructure, with the best homogeneity and crystalline cohesion of TiO₂ at 15%. The greatest Vickers hardness was measured at 15%, reaching 170.82Hv. The greatest diagonal compressive strength value was also found at 15%, reaching 49.5 MPa, while density decreased with the increase in reinforcement content, reaching its minimum at 20% of TiO₂. However, porosity increased with the increase in titanium oxide content and decreased after the sintering process.

المخلص

تعتبر طريقة المساحيق من التقنيات الحديثة والمستخدمة في تصنيع عدد القطع وبعض المواد الهندسية، حيث اشتملت الدراسة الحالية على تصنيع نماذج بتلك الطريقة، حيث تم تحضير العينات من اخذ النيكل كمادة اساس و الالومينا بنسبة ثابتة (10%) اما اوكسيد التيتانيوم (TiO₂) كان بنسب (5,10,15,20,0%) حيث تم طحن المساحيق و خلطها باستخدام طاحونة محلية الصنع تحوي على كرات فولاذية لزمن ساعة واحدة بعد ذلك تم كبس النماذج عند ضغط (5 Ton) ولزمن دقيقة واحدة. لبنت العينات المحضرة عند درجة حرارة (1300°C) ولزمن ساعتين فقط. تم اجراء الفحص التركيبي باستخدام المجهر الالكتروني الماسح (SEM) وكذلك الفحوص الميكانيكية (الصلادة ومقاومة الانضغاط القطرية) وبعض الفحوص الفيزيائية (الكثافة والمسامية) وجميعها قبل وبعد التلييد. بينت نتائج المجهر الالكتروني الماسح الحصول على بنية مجهرية واضحة المعالم ويكون افضل تجانس وتماسك بلوري لها عند النسبة (15% TiO₂) اما بالنسبة للصلادة فقط اعطت افضل صلادة لفيكرز عند النسبة 15% TiO₂ ووصلت الى (170.82 Hv)، اما مقاومة الانضغاط القطرية فكانت افضل قيمة لها عند نفس النسبة ايضا وبلغت (49.5 MPa) اما قيم الكثافة تتناقص مع زيادة نسب التدعيم لتصل اقلها عند النسبة 20% TiO₂. اما بالنسبة للمسامية تلاحظ ازديادها مع زيادة محتوى اوكسيد التيتانيوم وتقل بعد اجراء التلييد.

1. Introduction

In the past few decades, interest has grown in the production of composites from various engineering materials to improve strength, stiffness, wear resistance and tolerance to high temperatures. Moreover, composites contain components that share covalent bonds, such as Al₂O₃, Si₃N₄, SiC and C, which can be produced with different techniques, including powder technology and methods of thermal, chemical and physical deposition. Composites are classified according to their matrix into a metal, ceramic or polymeric matrix. The reinforcement phase can be metal, ceramic or polymeric (Crawford, 1987; Clyne *et al.*, 2003); also, the Ni-Al₂O₃ system is resistant to high heat and corrosion as it can be used in motors to increase the wear resistance and hardness of metal surfaces (Bogdan *et al.*, 2005). Many modern technologies require a set of materials with distinct properties that cannot be obtained when using conventional alloys. Ceramics and polymers have been used as engineering materials in aerospace, aqueous and transport applications. For example, aircraft engineers seek synthetic materials that have high strength, impact resistance, rubbing resistance and corrosion resistance and low density, which are rarely found in one component. Consequently, composites have been the ideal solution for combining these contrasting properties (William *et al.*, 2001). Composites have received considerable attention in developmental research to meet the requirements of technical progress. One of the most important advantages of composites is design flexibility, as the engineer can add reinforcement materials with different forms and then design them. For example, adding fibres toward the loads results in the optimal use of the material and thus a greater decrease

in the final composition weight. The main disadvantage of composites from an engineering perspective is their lack of dynamic impact load tolerance, which often causes stratification of the material (Daniel *et al.*, 2001). With scientific and technical development, materials that could not be replaced with other materials have emerged, the most important of which is graphite. Graphite is one of the most important materials known to mankind because of its thermal, mechanical and chemical properties and low friction coefficient and wear rate. Therefore, it is applicable to various uses, including the production of the brushes used in electric motors. It is also used as a neutron moderator in nuclear reactors and as a lubricant. Hence, powder metallurgy is a key technology for using graphite in the electrical and thermal industries (Lee, 1998). Previously, nanoparticle titanium dioxide was not combined with nickel in engineering applications, so the oxide was added to test its effect on the mechanical and physical properties. This study aims to explain the effects of nano-titanium oxide on nickel in terms of structural and mechanical properties, which facilitates the selection of useful engineering materials, such as for engines, magnets and coins.

2. Experiment

2.1. Materials:

Nickel (Ni) of German origin from Merck Co. was used as a matrix, with a grain size of ≤ 53 and a purity of 99.95%. Also, constant alumina content (Al₂O₃) of German origin from Fluka Co. was used with a grain size of ≤ 63 and a purity of 99.99%. The reinforcement material was made of 30 ± 5 nm nano titanium oxide (TiO₂) of

Chinese origin from Changsha Santech Materials Co., with a purity of ≥99.8%.

2.2. Materials Preparation:

The powders were dried at 200°C for two hours to remove moisture and other volatile substances. Then, the mixture weights were prepared for each component by following the weighted ratios so that the percentage of alumina was constant with a content of 10% for all mixtures, but titanium oxide was at different ratios (0, 5, 10, 15 and 20%). A Japanese-origin Sartorius balance of 0.0001 g, precision calibrated by the Central Organisation for Standardisation and Quality Control, was used for weights. After accomplishing the milling and stirring processes and obtaining a homogeneous powder, the samples were formed through the application of a uniaxial compaction in a hardened steel mould with a hardness of 70HRC. The stirred mixture was placed inside the compaction mould carefully to prevent the movement of the parts of the mould. Then, five tonnes of pressure was applied for one minute to avoid the possibility of elastic strain (Jin *et al.*, 2011; Samal *et al.*, 2012). For this purpose, a Turkish-type (HALIM USTA) hydraulic press with a capacity of 20 tonnes calibrated by the Central Organisation for Standardisation and Quality Control was used to obtain cylindrical samples with a diameter of 10 mm and height of 6 mm. After the pressing process, the samples are not yet ready for testing and have little green strength, requiring care during transporting and handling while the sintering process is taking place. The sintering process was carried out using a German-made CARBOLITE oven at a temperature of 1300°C for two hours. Inert Alarcon gas was pumped into the oven to eliminate oxidation and obtain distinct samples.

2.3. Examinations and Practical Measurements:

2.3.1. Scanning Electron Microscope (SEM)

After sintering, the surface topology of the resulting samples was examined to determine their external surface and coherency. An SEM was used for this purpose.

2.3.2. Vickers Hardness (Hv)

Hardness is an important mechanical property and is defined as surface indentation (Chawla *et al.*, 2012). It was examined using the Vickers method by indenting a square-based diamond pyramid using bulk of 500 gm for 10 seconds. By calculating the resulting indentation diameters, the Vickers hardness of compacts can be found by applying the following equation (Chawla *et al.*, 2012):

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

where P is the force applied (Kg), and D_v is the mean diameter of the pyramidal indentation resulting from the force applied to the surface.

2.3.3. Diagonal Compressive Strength

Compressive strength was tested using a universal testing machine of Chinese origin (HOYTOM). First, the sample was placed on the examination platform. Then, force was applied to the sample diameter until failure occurred, and the maximum load was shown on the tool's digital screen. It should be noted that the tool can store the maximum value of the force before failure. Compressive strength is calculated using the following equation (Jonsen, 2006):

$$\sigma_D = \frac{2F}{\pi dh} \text{ --- (2)}$$

where σ_D is compressive fracture strength (MPa), F is the force

applied (N), d is the sample diameter (mm), and h is the sample thickness (mm).

2.3.4. Practical density

One of the most important metallurgical tests of compounds produced with the powder metallurgy technique is the density test. Hence, green density is one of the important characteristics of powders and has a great impact on densification. Improving green density helps obtain soft and regular compacts. It can be determined with the weights and dimensions of compacts using the following equation (Jianzhong *et al.*, 2009; Berger, 2010; Dutta *et al.*, 2012; Lowell *et al.*, 2013; Mossa, 2013):

$$\rho = \frac{M}{V} \text{ --- (3)}$$

where ρ represents green density (g/cm^3), M compact bulk (gm) and V compact size (cm^3).

2.3.5. Porosity

Practical porosity was tested with the Archimedes principle based on international standard ASTM C373 -88 using an electric balance with a precision of 0.0001g, following the following steps:

Compacts were dried for one hour at a temperature of 150°C using a Memmert electric oven and left to cool inside the oven. They were weighed after being removed from the oven to obtain the dry weight (W_d).

Distilled water was provided from the ampoules division station at the General Company for the manufacture of medicines and medical supplies in Samarra, processed in four stages. It was boiled for five hours, and the compacts were immersed in it. Then, the compacts were set in containers of distilled water for 24 hours at room temperature. Afterward, the compacts were removed, and the suspended water remaining on the surface was carefully discharged. Then, the compacts were weighed to obtain the saturated weight (W_s).

The compacts were weighed while suspended and immersed in distilled water using a portable electronic scale found in the mineral laboratory of the Department of Mechanical Engineering at Tikrit University in order to obtain the suspended weight (W_i).

Theoretical density is the following (Berger, 2010):

$$\rho_{th} = \sum_0^n (\rho_i * X_i) \text{ --- (4)}$$

where ρ_{th} is theoretical density (g/cm^3), ρ_i is the theoretical density of the single compact components (g/cm^3), and X_i is the volumetric ratio of each compact element.

Green density and bulk density are the following (Berger, 2010; Lowell *et al.*, 2013):

$$\rho = \frac{W_d}{W_s - W_i} * \rho_w \text{ --- (5)}$$

where ρ is the green density representing volumetric density (g/cm^3), ρ_w is the density of the fluid used (1g/cm^3), W_d is the compact dry weight (gm), W_i is the compact in suspended weight (gm), and W_s is the compact saturated weight (gm).

Apparent density is the following (Lowell *et al.*, 2013):

$$\rho_A = \frac{W_d}{W_d - W_i} * \rho_w \text{ --- (6)}$$

Practical porosity is the following (Berger, 2010; Lowell *et al.*, 2013):

$$T.P \% = \frac{\rho_{th} - \rho}{\rho_{th}} * 100 \text{ --- (7)}$$

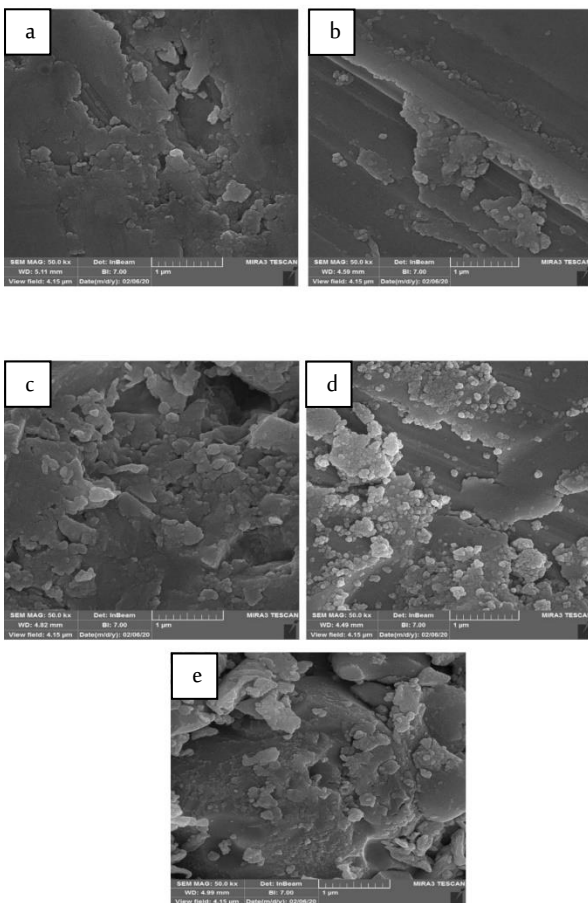
where $T.P\%$ is the percentage of practical porosity, ρ_{th} is theoretical density (g/cm^3), and ρ is green or volumetric density.

3. Results and Discussion

3.1. Scanning Electron Microscope (SEM):

Figure 1 – a, b, c, d and e – illustrates SEM images at $1 \mu\text{m}$ of composite compacts after the sintering of the samples. In a and b, there is a homogeneous distribution of titanium oxide nanoparticles on the nickel surface, forming a continuous metal net as well as recrystallisation, as shown in c. Titanium oxide nanoparticles \ attract porosity, whether before or after sintering. Based on electronic.

Fig. 1: SEM after sintering at 1300°C for two hours with a-0%, b-5%, c-10%, d-15% and e-20% of TiO_2



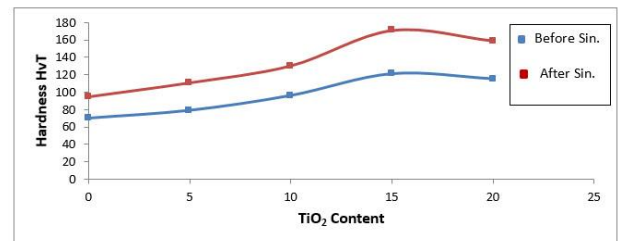
In the images, there is clear and homogeneous diffusion in the solid state with great bonding between composite elements, as shown in sample d, i.e. at 15% of TiO_2 . After the sintering process, it was observed that the microstructure is more homogeneous, with a stronger bond, and that there is an overlap between the particles of all elements. In addition, there are no cracks or divisions that indicate the positive effect of appropriate thermal treatment on the elements' convergence with strong and clear bonding between grain boundaries as a result of this strong correlation and homogeneity between composite components. As for sample e, dislocations and pores begin to emerge, demonstrating that the best reinforcement contents are at 15%.

3.2. Effect of Increasing Titanium Oxide Nanoparticle

Content on Vickers Hardness:

Figure 2 depicts the hardness behaviour of nickel-alumina composite 10% TiO_2 – titanium oxide with respect to increasing TiO_2 nanoparticle content at a sintering temperature of 1300°C . It was observed that adding 5% titanium oxide nanoparticle content does not have a significant impact on hardness values, while an increase of 15% leads to a significant increase in hardness. After that, the values either decrease or stabilise when increasing titanium oxide nanoparticle content to 10% and 20%. Hardness values then significantly decrease until the titanium oxide nanoparticle content reaches its maximum at 20%. The reason for the decrease in hardness values may be due to the occurrence of clusters when increasing the ratio of nanoparticles to this percentage, which in turn decreases the hardness. It should be noted that before and after sintering, composite hardness at 0% increased from 70 to 94.4, then increased for the remaining percentages – 5%, 15% and 20% of TiO_2 nanoparticles, respectively. Figure 2 also shows the greatest value of hardness at 15%, when it reached 170.82 after sintering and then decreased to 158.74 at 20% titanium oxide nanoparticle content. The decrease in composite hardness at 20% is due to the increase in the accumulation of titanium oxide, which facilitates its indentation by indenter of the hardness method used, in addition to its lowest solubility in nickel (the lack of hardness in solid solution hardening, the lack of interaction between nickel and titanium oxide and the bonding between them is limited to the mechanical bond) (Gan *et al.*, 2008).

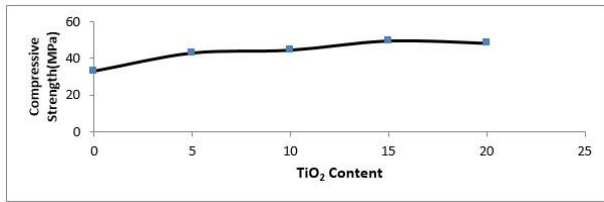
Fig. 2: The relationship between Vickers hardness and nano titanium oxide content before and after the sintering process



3.3. Effect of Titanium Oxide Nanoparticle Content on Diagonal Compressive Strength:

Figure 3 clarifies a change in compression strength with the change in the content of titanium oxide nanoparticles for the nickel-alumina composite (10%) – titanium oxide after sintering. It was observed that the increase in the content of titanium oxide nanoparticles causes a significant increase in the compression strength of the composite, which increased from 32.9 MPa to 49.5MPa at 0% to 15%, respectively, of titanium oxide nanoparticle content when sintering at 1300°C , reaching the highest value of compression strength and then decreasing to 48.8 MPa at 20% titanium oxide nanoparticle content. This behaviour can be attributed to several reasons, the most important of which is high composite fragility with the increase of titanium oxide nanoparticle content. Also, increasing titanium content coats and segregates nickel particles from each other, which prevents complete integration and impedes partial integration between them. This is consistent with the findings of Gan *et al.* (2008) and Samal *et al.* (2013). Moreover, the increase in titanium oxide content was accompanied by a decrease in volumetric density and an increase in the practical porosity of the composite. All these factors have led to this significant decline in compression strength at 20%. The decrease in the hardness values may also be due to the occurrence of clusters within the nanoparticles when increasing them to this percentage, which in turn decreases the hardness.

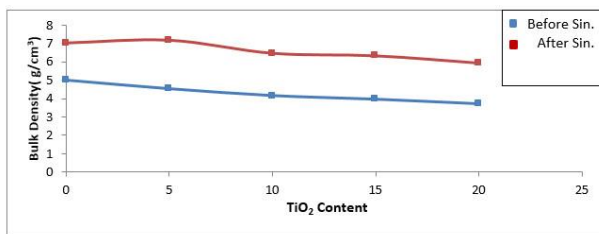
Fig. 3: The relationship between titanium oxide content and compressive strength after sintering



3.4. Effect of Titanium Oxide Nanoparticle Content on Density:

Figure 4 shows the relationship between different contents of titanium oxide nanoparticles and practical density in nickel composite (10%) alumina-titanium oxide. It is observed that increasing titanium oxide content has led to a decrease in practical density for all contents of titanium oxide in composites. Hence, they decrease from 5.026 g/cm³ at 0% of titanium oxide content to 3.698 g/cm³ at 20% content before sintering. As shown in Figure 4, the density increases to 7.021 g/cm³ at 0% of titanium oxide content after sintering and then begins to decrease until it reaches 5.935 g/cm³ at 20% content. This can be attributed to the increase in the cold forming rate by sintering and the accompanying increase in hardness and flow stress, and a decrease in ductility, which is the criterion of plastic formation (Ziemkowska *et al.*, 2014). This, in turn, leads to an increase in powder compaction strength and plastic formation with sintering, and thus the density of the compacted composite decreases. The titanium oxide content also plays an important role in decreasing practical density by increasing sintering. The reason for the decrease in the hardness values may be due to the occurrence of clusters with the nanoparticles when increasing them to this percentage, which in turn decreases the hardness and a density below that of nickel matrix, which in turn leads to a decrease in density (Ziemkowska *et al.*, 2014).

Fig. 4: The relationship between titanium oxide nanoparticle content and practical density before and after sintering



3.5. Effect of Titanium Oxide Nanoparticle Content on Porosity:

Figures 5 and 6 show the relationship between porosity and titanium oxide nanoparticle content before and after sintering at 1300 °C, respectively. It should be noted that the increase in porosity occurs with increasing titanium oxide nanoparticle content. For example, it increases from 6.71% at 0% and 20% of titanium oxide, respectively, before performing sintering composite samples, as shown in Figure 7. Then, the porosity value decreases from 6.71% before sintering to 5.34% after sintering at the same content of 0%, then decreases from 16.95% before sintering to 13.54% after sintering at the same content of 20%. This behaviour is fully compatible with the total porosity behaviour. This indicates that the effect of titanium oxide content on both open and closed porosity is of one nature, the factors governing closed porosity are similar to those governing open porosity and that the mechanisms forming these two types of porosity are the same. It was also noticed

that titanium oxide nanoparticle content and its effect mechanisms in the formation of porosity determine apparent porosity and not sintering (Darweesh *et al.*, 2019; Stipniec *et al.*, 2020).

Fig. 5: The relationship between titanium oxide nanoparticle content and porosity before sintering

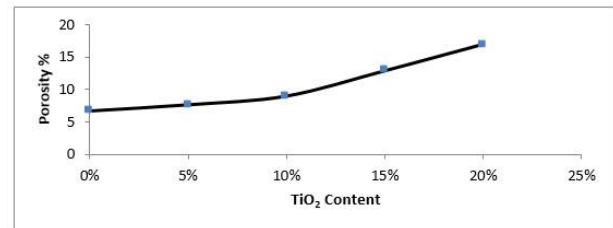
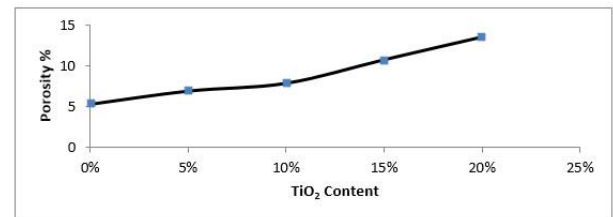


Fig. 6: The relationship between titanium oxide nanoparticle content and porosity after sintering



4. Conclusion

This study concluded the possibility of manufacturing composite samples of nickel-alumina- titanium with a constant alumina content of 10% and different reinforcement contents of titanium oxide nanoparticles. It was observed that there was a significant increase in the values of hardness and compression strength with increasing reinforcement content; however, there was a decrease in practical density values and an increase in porosity values with increasing reinforcement content. Also, it is found that the ideal reinforcement content is 15%, which has a significant mechanical effect compared to the remaining contents. As for the electron microscope, the surface morphology indicates that the most ideal content is 15%.

Bios

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