Spark plasma sintering of ZrO₂ reinforced Ni-Cr alloy: Microstructure and mechanical behavior

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Abstract
Ni-Cr-ZrO₂ composites with varying amounts of ZrO₂ additive (5 wt%, 7.5 wt%, 10 wt% and 12.5 wt%) were fabricated using spark plasma sintering method at a sintering temperature of 1000°C, heating rate of 100°C/min, holding time of 5 min, and a pressure of 50 MPa. The effect of ZrO₂ addition on the microstructure, tribological and mechanical properties of the developed composites were studied. The results showed that maximum densification was attained at 10 wt% ZrO₂. Further increase in the fractions of ZrO₂ within the composites results in a decrease in the relative density of the sintered composite. A significant increase in hardness from 433.24 HV to 510.11 HV and elastic modulus from 252.67 GPa to 294.6 GPa was observed in the fabricated samples as the ZrO₂ content increase from 5 to 12.5 wt%. An appreciable improvement in the wear performance of the sintered samples was obtained with increasing ZrO₂ content. The observed improvement in the properties of the sintered composites was attributed to the presence of the hard dispersoids of ZrO₂ and formation of solid solution strengthening and hard Cr₃Ni₂ phases within the matrix of the sintered composites.

Keywords
sintering, Ni-Cr-ZrO₂ composites, microstructure, nanoindentation hardness, wear

Introduction
The demand for lightweight materials for the production of some of the components of aircraft engines has influenced the continuous research and development of novel materials suitable for such applications.¹ Nickel-based alloys and composites are special type of materials with a unique combination of physical, chemical, and thermo-physical characteristics. These unique attributes results in their wide usage in different engineering applications such as automotive, aerospace, power, and nuclear reactors.² The ability of nickel-based alloys to maintain high strength at elevated temperatures, high resistance to wear, corrosion, and fatigue; and high toughness are distinctive attributes which increase their demand in the combustion chambers of aircraft, and power generation.³⁻⁸ Nickel-based alloys with a chromium content of 15–30% weight fractions are commonly utilized in land turbine and aircraft turbine engines where high temperatures and strong mechanical loads are encountered. This is due to their ability to maintain their strength at high-temperature, and also display good resistance to corrosion and oxidation at high-temperature.⁹

As a result of their high efficiency at high temperatures, nickel-chromium (Ni-Cr) compounds are increasingly being evaluated for applications at extreme temperatures.¹,¹⁰ The unique mechanical properties of Ni-Cr based alloys, such as excellent resistance to oxidation and hot corrosion, have classified them amongst the popular superalloys with a wide range of engineering applications and thus, are used for important structural materials.¹¹,¹² Nevertheless research has shown that their extensive usage in aero-engine is

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limited by their high specific density. However, it has been reported that Ni-Cr alloys can be reinforced with hard particles like carbides and oxides to reduce their specific density and widen their usage in aero-engines. Carbides and oxides reinforcements are utilized because of their unique properties, such as high melting point, high resistance to plastic deformation and heat, physical and chemical stability. These ceramics confer on the resulting composites outstanding properties such as toughness and wear resistance, which has made them useful in machining, cutting blades, and other applications.

In recent years, the use of zirconia particles as reinforcement has gained tremendous popularity because of its key qualities such as increased mechanical properties, high melting point, excellent chemical and heat resistance, and compatibility with living tissues. Ni-ZrO₂ is a feasible alternative for metal-ceramic composites since the coefficients of thermal expansion (TE) of nickel and zirconia are close to each other. Several studies focused on ZrO₂ reinforced composites have been reported in the literature. Srivastava et al. investigated the influence of ZrO₂ addition on the chemical and mechanical properties of Ni-Co alloys through electrodeposition method. They discovered that the presence of ZrO₂ nanoparticles altered the corrosion behavior of the sintered composite and enhanced its wear resistance. The development and characterization of Ni-ZrO₂ composite by pulse electrodeposition was studied by Wang et al. It was discovered that Ni-ZrO₂ composite possessed better hardness properties and wear resistance when compared with pure nickel metal. Plazanet et al. studied the effect of reinforcement on the mechanical properties of NiAl by comparing SiC and ZrO₂ particles, they reported that the addition of ZrO₂ better enhanced the fracture toughness and flexural strength of NiAl matrix composite better than the SiC. The synthesis and mechanical properties of Ni-W-ZrO₂ nanocomposite coating fabricated by pulse electrodeposition was studied by Zhang et al. It was reported that adding ZrO₂ to the Ni-W matrix composite positively influence the particle size, harness properties, corrosion resistance and roughness of the sintered composite.

However, few report have been found on ZrO₂ reinforced nickel matrix composites consolidated through the powder metallurgical route. Several powder metallurgical processes such as hot pressing (HP), hot isostatic pressing (HIP), cold isostatic pressing and compaction as well as spark plasma sintering (SPS) are employed in the fabrication of metal matrix composites (MMCs). However, the advantages of SPS which include accelerated heating rate, reduced sintering temperature, shorter sintering time, high sintered densities and reduced grain growth makes the process a preferred method of fabricating MMCs.

Until now, only a limited study has been conducted on Ni-Cr alloys reinforced with zirconia using the spark plasma sintering process in the literature. Hence, this work focused on spark plasma synthesis of Ni-Cr-ZrO₂ composites. The effect of ZrO₂ addition on the microstructural, mechanical and tribological characteristics of the developed Ni-Cr-ZrO₂ composites is critically investigated.

### Experimental procedure

#### Material and methods

Nickel powder (99% purity), Chromium powder (99% purity) and, Zirconium oxide powder (99% purity) used in this study were supplied by Sigma Aldrich and Alfa Aesar, South Africa. The powders were accurately weighed to form the desired stoichiometry composition. The percentage weight (wt%) composition of the composite is shown in Table 1.

#### Experimental procedures and characterization

Nickel, Chromium and zirconium oxide powders of the desired stoichiometry of Ni-20Cr-XZrO₂, where X is 5, 7.5, 10, and 12.5 wt% (Table 1) were mixed in a tubular mixer (model T2F Nr.100, 462, Muttenz Switzerland) for 8 h at a rotating speed of 49 r/min. Pre-alloying was done with a ball-to-powder ratio (BPR) of 10:1, to aid the uniform distribution of the elemental powders. The pre-alloyed powders were poured into a graphite die of 20 mm diameter and 50 mm height, and then compacted manually. The inside of the graphite die was lined with a 0.2 mm thick graphite sheet, this was necessary to prevent a reaction between the powders and the graphite die. The graphite die-powder-punch assembly was then charged into the furnace of the spark plasma sintering machine (model HHPD-25, FCT Germany) operated at a sintering temperature of 1000°C, heating rate of 100°C/min, holding time of 5 min and pressure of 50 MPa to produce a cylindrical disc of Ø 20 mm × 7 mm. Prior to the selection of these sintering parameters, an initial trial-study was performed which revealed that a temperature exceeding 1000°C resulted in melt-out. The sintered samples were sandblasted, to get rid of any residual graphite layer on the surfaces of the samples. Then the relative density was determined using Archimedes’ principle.

A scanning electron microscope (JEOL JSM-7600F) equipped with EDS detector with INCA X-Stream 2 pulse analyzer was used to analyse the microstructure of the sintered samples, while the phases present in the sintered composites were evaluated using an X-ray diffraction (XRD) equipment (PANanalytical). The sintered samples were sectioned into smaller pieces of 5 mm × 5 mm and 10 mm × 10 mm each with the aid of a cutting machine. The sectioned surface was grounded and polished using grit sizes of 320, up to 2400, to get a mirror-finished surface.
Anton-Paar TTX-NHT3 Nanoindentation tester equipped with a diamond Berkovich three-sided pyramid indenter (Oliver-Pharr method) was employed to determine the Vickers hardness, microhardness, and elastic modulus of the samples. The experiment used a 400 mN indentation load that imprints grids on the surface of the samples using a diamond Berkovich indenter. A minimum of 12 indentations was applied per load during the indentation for each sample with a pause duration of 20.0 s.

Anton Paar TRB3 pin-on-disc tribometer was used to perform the wear test on the sintered sample of dimension 10 mm × 10 mm. An alumina ball with a diameter of 6 mm was used as a counterface material. The test was conducted at a rotational speed and radius of 300 r/min and 3 mm respectively under varying loads of 10 N, 15 N, and 20 N and a frequency of 15 Hz. During the test, the stationary counterface ball was made to slide against a rotating disc comprising the test samples in a reciprocating motion. The coefficient of friction as a function of time for the composites was continuously recorded by the tribometer. The wear test was carried out three times on each sample to guarantee repeatability of the results. The wear rate was determined and the morphology of the worn surface of the samples was also analysed.

Result and discussion

Microstructural and phase analysis of the sintered composites

The SEM images of the sintered Ni-Cr-ZrO₂ composites at different ZrO₂ contents are shown in Figure 1(a)–(d). From Figure 1, dark and white coloured phases were observed within the matrix of the developed composites. The EDS analysis showed the preponderance of elemental Cr and Ni in the dark coloured regions while the predominance of Zr and O was observed in the white coloured regions within the matrix of the composites. This suggests these phases to be Cr₃Ni₂ and ZrO₂ compounds respectively. The presence of pores could be seen within the microstructure of the Ni-Cr-5ZrO₂ composite (Figure 1(a)). Decreasing pores with increasing ZrO₂ content was observed in Figure 1(b)–(d) due to the presence of homogeneously distributed ZrO₂ particles within the composites matrix. This is because there is a uniform dispersal of ZrO₂ particles across the Ni-Cr boundary, and no significant accumulation of ZrO₂ particles was found. It has also been reported that incorporation of ZrO₂ as sintering additive improves the sinterability of metal matrix composites. This is in agreement with a study conducted and reported by Shuan Ma. The Zr-O rich phase (white phase) is not as noticeable in Figure 1(a) as it is in Figure 1(d), where the percentage composition of ZrO₂ is highest and uniformly dispersed throughout the matrix.

Figure 2 depicted the EDS mapping of the sintered Ni-Cr-12.5ZrO₂ composite. The figure shows homogeneous distribution of elements within the composite. The blue colour regions represent the Ni-rich spots. Expectedly, nickel being in highest percentage composition is conspicuously present in all the phases. The green colour areas represent the Cr-spots. However, the brightly coloured spots in this matrix signify high concentration of Cr which correspond to the EDS quantitative analysis in Figure 1(b). The purple and pink colour regions represent the Zr-rich and O-rich spots in the matrix of the composite.

The XRD diffractographs of the sintered Ni-Cr-ZrO₂ composites fabricated at different ZrO₂ contents are shown in Figure 3. The XRD patterns showed the formation of solid solution phase of (Cr, Ni) and a hard tetragonal phase, Cr₃Ni₂. The presence of undissolved, hard ZrO₂ phase within the composites matrix was also observed. This can be ascribed to the lower sintering temperature employed in this work (1000°C) compared to the melting temperature of ZrO₂ (2680°C). Thus confirming that the dark and white coloured phases observed in Figure 1 as Cr₃Ni₂ and ZrO₂ phases respectively. As reflected in the pattern, the peaks are indexed to the cubic structure of nickel according to Wyckoff et al. and Lundqvist. In Figure 3, three noticeable peaks of Ni at Bragg’s angles (2θ) of 44°, 52° and 77° were observed throughout the sintered composites. It was revealed that pure nickel had a preferential orientation along the (111), (002), and (022) crystal planes. The prominent peak corresponding to Ni (111) and (Cr, Ni), was found at a Bragg’s angle (2θ) of 44°, and at Bragg’s angle (2θ) of 52° the peak corresponds to Ni (002). However, Ni (022) and Cr peak was found at Bragg’s angle (2θ) of 77° while only Ni was found on the peak as the ZrO₂ content increased to 12.5 wt% in the matrix. Some minor peaks correlated to Cr₃Ni₂ can be noticed in XRD diffractograph. More peaks belonging to ZrO₂ were observed with the increasing ZrO₂ content in the matrix of the sintered composite. This can be attributed to increasing fraction of undissolved ZrO₂ in the matrix. It is noticed that the intensity of the Ni peaks reduced, and the base broadens, while the intensity of other phase peaks increases with increase in ZrO₂ contents (Figure 3). The reduction in peak intensity indicates increased dislocation density as a result of flaws caused by grain refining capacity of ceramic particles within matrix. This is responsible for the increase in the hardness.

### Table 1. Percentage weight composition of Ni-Cr-ZrO₂ composite.

| Elements | Ni | Cr | ZrO₂ |
|----------|----|----|------|
| Composition (wt%) | Balance | - | 20 | 5 |
| - | - | 7.5 | 10 | 12.5 |
| Particle size (μm) | ≤44 | ≤44 | ≤44 | ≤44 | ≤44 |
Figure 1. Scanning electron micrographs of Ni-Cr-ZrO₂ composites sintered at different ZrO₂ content. (a) 5 wt% (b) 7.5 wt% (c) 10 wt% (d) 12.5 wt%.

Figure 2. EDS mapping and element distribution of Ni-Cr-12.5ZrO₂ composite sintered at 1000°C.

| Elements | Weight |
|---------|--------|
| Ni      | 64.8   |
| Cr      | 18.9   |
| Zr      | 14.6   |
| O       | 1.1    |
| C       | 0.6    |
property of the sintered composite as the ZrO2 content increases.

The increase in the intensity of the diffraction line of ZrO2 indicates a reduction in the weight percentage of nickel in the metal matrix. This is due the increasing the ZrO2 content, which also increases the crystallinity of the fabricated composite. Thus, signifying the ordered arrangement between the Ni-rich phase and ZrO2.

**Densification and hardness of the sintered composites**

Figure 4 shows the relative density and microhardness values as a function of the zirconium oxide content. Dicated in Table 2 are relative density values for the developed samples using the Archimedes’ principle. From these tables, it was observed that increasing the ZrO2 content in the Ni-Cr matrix led to a corresponding increase in the relative density to the maximum value of 98.21% at 10 wt% ZrO2. Subsequent addition of 12.5 wt% ZrO2 to the Ni-Cr matrix signal the onset of a decreasing trend of the relative density. According to Rominiji et al.23 and Baghery et al.,36 homogeneous dispersion of reinforcement within composite matrix enhance densification while non-uniform distribution of reinforcement within the composite matrix results in agglomeration and this strongly reduce the density of the resulting composite. Thus it can be inferred that the uniform distribution of ZrO2 particles in the composite matrix was achieved up to 10 wt% beyond which the density begin to decline due agglomerations and porosities in the matrix of the composite. It is worth noting that density plays a major role in improving the mechanical properties of material because of its direct effects on the properties of the material (Table 3).

From Table 2, it was observed that the actual densities of all the fabricated composites were slightly less than the theoretical density. This showed that there is a strong
adhesion between the Ni-matrix and the ceramic particles of ZrO2.3

The variation in hardness of the sintered Ni-Cr-ZrO2 composites with increasing ZrO2 addition is presented in Figure 4. As expected, the relative density of the samples has common characteristics as the hardness which is a function of the ZrO2 addition. An increasing trend of microhardness values with increasing ZrO2 addition was observed in the sintered composites (Figure 4). It is worth noting that densification influence the mechanical properties of metallic composites, metals and alloys. Hence, composites with higher densities are known to have better mechanical properties, and reinforcing such composites and alloys with ceramic particulates will directly impart improve densification and mechanical properties.37,38 Also, the presence of hard, Cr3Ni2 phase and hard particles of ZrO2 in the composite matrix inhibit dislocation propagation.39 This subsequently resulted in increasing microhardness value as the reinforcement content increased in the composite matrix as shown in Figure 4. Thus the highest microhardness value of 510.11 HV was obtained for composite containing 12.5 wt% ZrO2 and the least microhardness value of 433.24 HV was recorded for composite reinforced with 5 wt% ZrO2. Similar work conducted by Li et al.40 on Ni-Mo-ZrO2 composites reported that the addition of ZrO2 nanoparticles influenced the hardness value of the sintered composites.

The force-depth curves

The nanoindentation experiments were carried out in order to examine the time-dependent nanoscale mechanical properties of the Ni-Cr-ZrO2 composites at room temperature. Figure 5 represents the force-depth curves of the sintered composites. It can be seen from the graph that the curves of all the sintered samples skewed to the left, signifying a reduction in the indenter maximum penetration depth with a force of 400 mN. The skewness of the curves of the composites indicated that the material was attempting to recover from the distortion.41 However, the indentation depth of Ni-Cr-5ZrO2 composite of 1990 nm was greater than that of Ni-Cr-12.5ZrO2 composite by 301 nm. The variation of zirconia content in the composites also influenced the indentation depth. At 12.5 wt% ZrO2, the indentation depth is observed to reduce by about 15.1%, and hardness increase with about 31%, thus, the composite display enhanced hardness properties. Composite materials with a higher ZrO2 content are more resistant to plastic deformation than those with a lower wt% of ZrO2 due to inhibition of dislocations movement in the lattice.40,41 The decrease in the penetration depth indicated an increase in

| Composition variation (wt%) | Theoretical density (g/cm³) | Relative density (g/cm³) | Relative density (%) |
|-----------------------------|-----------------------------|--------------------------|----------------------|
| Ni-Cr/5% ZrO2               | 8.27                        | 8.12                     | 98.15                |
| Ni-Cr/7.5% ZrO2             | 8.16                        | 8.01                     | 98.17                |
| Ni-Cr/10% ZrO2              | 8.06                        | 8.06                     | 98.21                |
| Ni-Cr/12.5% ZrO2            | 7.96                        | 7.81                     | 98.20                |

| Composition variation (wt%) | Microhardness (HV) | Relative density (%) | Nanohardness (GPa) | Elastic modulus (GPa) |
|-----------------------------|--------------------|---------------------|---------------------|-----------------------|
| Ni-Cr/5% ZrO2               | 433.24 ± 2.17      | 98.15 ± 0.002       | 4.9 ± 0.07          | 252.67 ± 1.26         |
| Ni-Cr/7.5% ZrO2             | 470.60 ± 2.35      | 98.17 ± 0.002       | 6.1 ± 0.09          | 281.91 ± 1.40         |
| Ni-Cr/10% ZrO2              | 506.49 ± 2.53      | 98.21 ± 0.002       | 6.8 ± 0.10          | 292.36 ± 1.46         |
| Ni-Cr/12.5% ZrO2            | 510.11 ± 2.55      | 98.20 ± 0.002       | 7.1 ± 0.11          | 294.60 ± 1.47         |
nanohardness of the material. Rodriguez et al.\textsuperscript{42} also reported that the indentation depth is a major factor that determines the nanohardness property of a material. As a result, a decrease in penetration depth as observed in Figure 5 is frequently associated with an increase in nanohardness of a material.\textsuperscript{43}

\textbf{Nanoindentation hardness and elastic modulus}

Figure 6 shows the relationship between the approximated elastic modulus (E) and the nanoindentation hardness (GPa) values with variation in ZrO\textsubscript{2} content. The plot revealed that the addition of ZrO\textsubscript{2} greatly influenced the hardness properties of the composites. The nanohardness value and the elastic modulus increased with increasing ZrO\textsubscript{2} content. At 5 wt\% ZrO\textsubscript{2}, the nanohardness value is 4.9 GPa, and the elastic modulus is 252.67 GPa, and at 12.5 wt\% ZrO\textsubscript{2}, the nanohardness and elastic modulus increased to 7.1 GPa and 294.6 GPa respectively. An increase in ZrO\textsubscript{2} content in the Ni-Cr matrix improved both the elastic modulus and the hardness properties of the sintered sample composite as seen in Figure 6. It is known that a higher amount of reinforcing particles in the matrix would result in more dislocations inhibition that result in an increase in the hardness of the composite.\textsuperscript{44} Thus, this increase is caused by the high hardness property of the reinforcing elements, and the improvement in the properties of the composite could be due to changes in the microstructure, as the presence of hard ZrO\textsubscript{2} can serve as a dislocations movement barrier. Therefore, a higher amount of energy would be needed to ensure the dislocation movement and this leads to an increase in the hardness of the composite.\textsuperscript{30}

\textbf{Wear rate and the average coefficient of friction}

The values of the wear rate and the average coefficient of friction with varied applied load (10N, 15N, 20N) obtained for the sintered composites are shown in Figure 7(a) and (b), respectively. From Figure 7(a), it can be generally observed that the wear rate of the composite across the applied loads decreased with increasing ZrO\textsubscript{2} content in the composites matrix. Under an applied load of 20 N, the material reinforced with the lowest fraction of ZrO\textsubscript{2} reinforcement (5 wt\%) displayed the highest wear rate of $2.709 \times 10^{-4}$ mm\textsuperscript{3}/Nm, while the lowest wear rate of $8.73 \times 10^{-5}$ mm\textsuperscript{3}/Nm was obtained for sintered composite containing 12.5 wt\% of ZrO\textsubscript{2}. The observed trend in the wear rate of the composites is in tandem with the hardness results as presented in Figure 4. This agrees with the Archard's equation of wear which states that the hardness of a material is inversely proportional to the volume loss/wear rate.\textsuperscript{45-47} Hence, expectedly, the least wear rate observed in composite containing 12.5 wt\% ZrO\textsubscript{2} across the applied loads can be attributed to the highest hardness of this sample as shown in Figure 4.

Figure 7(b) represents the average coefficient of friction (COF) of the sintered composites. It can be seen from this figure (Figure 7(b)) that the average coefficient of friction increase across the applied loads with the increasing ZrO\textsubscript{2} content in the composites matrix. This behaviour can be attributed to the ease of plastic deformation and pulling out of the hard particles of ZrO\textsubscript{2} from the surface of the composite at lower reinforcement content during dry sliding wear. Thus the high surface roughness which resulted in increasing average COF as observed in Figure 7(b). However, increase in the volume of hard phases in the composite matrix with increasing ZrO\textsubscript{2} content inhibit plastic deformation thereby ensuring relative surface smoothness of the composite during dry sliding wear. This result in decreasing average COF with the least observed in composite reinforced with 12.5 wt\% ZrO\textsubscript{2}. The different phase distribution and the mechanical action due to frictional movement of the surfaces during the rotational motion caused the ceaseless formation and damage of the friction-reducing layer. The response of the material to wear is also determined by the type of the microstructural features i.e. phase distribution, which is not density-dependent.\textsuperscript{48} The fluctuation in the average coefficient of friction could also be attributed to the formation of tribofilms at the friction interface due to the percentage composition of ZrO\textsubscript{2}.\textsuperscript{29}

\textbf{Analysis of the worn surface}

Figure 8(a)–(d) shows the SEM micrographs of the worn surfaces of the sintered composite under applied load 20N and dry sliding wear condition. Shallow grooves (Figure 8(a) inset) and delamination as well as the presence...
of debris as flakes were observed on the worn surfaces (Figure 8(a)). Under a high applied load of 20N, the wear debris forms a compressed transfer layer and spread over the sliding surface area (Figure 8(b)). This suggests the formation of a protective layer in the form of oxide layers on the rubbing surface due to the interaction between the counterface alloy and the surface of the sample, this significantly lowered the wear rate of the composites as the percentage content of ZrO2 increased (Figure 7(a)). Thus, the surface of the composites without delamination in Figure 8(c) and (d), revealed that as the ZrO2 content in the composites increased, the two predominant wear mechanism are abrasion with mild plastic deformation and adhesive wear.29 Reinforcing with ZrO2 could have influenced the hardness property and caused a decrease in plastic deformation by inhibiting the sintered samples from undergoing additional flow stress when the sliding surfaces come in contact with each other. This is in conformity with what was reported in the literature.49–51 Hence, the enhanced wear rate of the composites with increasing ZrO2 content.

**Conclusion**

In this work, Zirconium oxide reinforced Ni-Cr matrix composites were successfully developed using the SPS process at 1000°C. The influence of ZrO2 additions on the microstructural, mechanical and tribological properties of the sintered composites were examined. The microstructural result revealed that reinforcing Ni-Cr matrix composites...
with ZrO$_2$ enhanced the formation of new phases that improved the hardness property of the composites. Increasing relative density with increasing ZrO$_2$ content were observed up to 10 wt% ZrO$_2$. The microhardness, nanoindentation hardness and the wear properties of the sintered composites were found to improve considerably with the addition of ZrO$_2$. According to this study, Ni-Cr-ZrO$_2$ composites possess strong potential as an enduring material for load bearing applications.

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**Availability of data and materials**
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**References**
1. Niu X, Zhang H, Pei Z, et al. Measurement of interfacial residual stress in SiC fiber reinforced Ni-Cr-Al alloy composites by Raman spectroscopy. *J Mater Sci Technol* 2019; 35(1): 88–93.
2. Makena MI, Shongwe MB, Ramakokovhu MM, et al. Effect of sintering parameters on densification, corrosion and wear behaviour of Ni-50Fe alloy prepared by spark plasma sintering. *J Alloys Compd* 2017; 699: 1166–1179.
3. Fattahi Z, Sajjadi SA, Babakhani A, et al. Ni-Cr matrix composites reinforced with nano- and micron-sized surface-modified zirconia: Synthesis, microstructure and mechanical properties. *J Alloys Compd* 2020; 817: 152755.
4. Cooper D, Blundell N, Maggs S, et al. Additive layer manufacture of Inconel 625 metal matrix composites, *Reinforcement Mater Evaluat* 2013; 213(12): 2191–2200.
5. Zickler GA, Schnitzer R, Radis R, et al. Microstructure and mechanical properties of the superalloy ATI Allvac® 718Plus™. *Mater Sci Eng A* 2009; 523(1–2): 295–303.
6. Borkar T and Banerjee R. Influence of spark plasma sintering (SPS) processing parameters on microstructure and mechanical properties of nickel. *Mater Sci Eng A* 2014; 618: 176–181.
7. Pollock TM and Tin S. Nickel-based superalloys for advanced turbine engines: chemistry, microstructure and properties. *J Propulsion Power* 2006; 22(2): 361–374.
8. Ogunbiyi OF, Jamiru T, Adesina OT, et al. Effect of nickel powder particle size on the microstructure and thermophysical properties of spark plasma sintered NiCrCoAlTiW-Ta superalloy. IOP Publishing.
9. Babalola BJ, Shongwe MB, Obadele BA, et al. Densification, microstructure and mechanical properties of spark plasma sintered Ni-17% Cr binary alloys. *Int J Adv Manufacturing Technol* 2019; 101(5–8): 1573–1581.
10. Sun D, Liang C, Shang J, et al. Effect of Y2O3 contents on oxidation resistance at 1150°C and mechanical properties at room temperature of ODS Ni-20Cr-5Al alloy. *Appl Surf Sci* 2016; 385: 587–596.
11. Briggs SA, Barr CM, Pakarinen J, et al. Observations of defect structure evolution in proton and Ni ion irradiated Ni-Cr binary alloys. *J Nucl Mater* 2016; 479: 48–58.
12. Xu Y, Yan J, Sun F, et al. Effect of further alloying elements on corrosion resistance of Ni-Cr alloys in molten glass. *Corrosion Sci* 2016; 112: 647–656.
13. Shen M, Zhao P, Gu Y, et al. High vacuum arc ion plating NiCrAlY coatings: Microstructure and oxidation behavior. *Corrosion Sci* 2015; 94: 294–304.
14. Maoquan X. Effect of graphite addition on oxidation behavior of Ni-Cr-based superalloy at 900 C. *Rare Metal Mater Eng* 2009; 38(7): 1146–1149.
15. Lopez M, Jimenez J and Corredor D. Precipitation strengthened high strength-conductivity copper alloys containing ZrC ceramics. *Composites Part A: Appl Sci Manufacturing* 2007; 38(2): 272–279.
16. Irshad HM, Hakeem AS, Ahmed BA, et al. Effect of Ni content and Al2O3 particle size on the thermal and mechanical properties of Al2O3/Ni composites prepared by spark plasma sintering. *Int J Refractory Metals Hard Mater* 2018; 76: 25–32.
17. Ding S, Zhang K and Wang C. Pulse electrodeposition and nonoindentation test of ZrO 2/Ni nanocomposite. *J Wuhan Univ Technology-Mater Sci Ed* 2007; 22(3): 462–465.
18. Lopez-Esteban S, Rodriguez-Suarez T, Esteban-Betegon F, et al. Mechanical properties and interfaces of zirconia/nickel in micro-and nanocomposites. *J Mater Sci* 2006; 41(16): 5194–5199.
19. Srivastava M, Srinivasan A and V. K WG. Influence of zirconia incorporation on the mechanical and chemical properties of Ni-Co alloys. *Am J Mater Sci* 2011; 1(2): 113–122.
20. Wang W, Hou FY, Wang H, et al. Fabrication and characterization of Ni-ZrO2 composite nano-coatings by pulse electrodeposition. *Scripta Mater* 2005; 53(5): 613–618.

21. Plazanet L, Tetard D and Nardou F. Effect of SiC and ZrO2 particles on the mechanical properties of NiAl. *Composites Sci Technol* 1999; 59(4): 537–542.

22. Zhang W, Ji C and Li B. Synthesis and properties of Ni–W/ZrO2 nanocomposite coating fabricated by pulse electrodeposition. *Results Phys* 2019; 13: 102242.

23. Rominiyi AL, Shongwe MB, Tshabalala LC, et al. Spark plasma sintering of Ti–Ni–TiCN composites: microstructural characterization, densification and mechanical properties. *J Alloys Compd* 2020; 848: 156559.

24. Jeje SO, Shongwe MB, Maledi N, et al. Synthesis and characterization of TiN nanoceramic reinforced Ti–7Al–1Mo composite produced by spark plasma sintering. *Mater Sci Eng A* 2021; 807: 140904.

25. Ogungbii O, Sadiku R, Adesina O, et al. Microstructure and mechanical properties of spark plasma-sintered graphene-reinforced inconel 738 low carbon superalloy. *Metallurgical Mater Trans A* 2021; 53: 1–15.

26. Obadele BA, Ige OO and Olubambi PA. Fabrication and characterization of titanium-nickel-zirconia matrix composites prepared by spark plasma sintering. *J Alloys Compd* 2017; 710: 825–830.

27. Ogungbii O, Jamiru T, Sadiku R, et al. Optimization of spark plasma sintering parameters of inconel 738LC alloy using response surface methodology (RSM). *Int J Lightweight Mater Manufacture* 2020; 3(2): 177–188.

28. Ogungbii O, Jamiru T, Sadiku R, et al. Influence of sintering temperature on the corrosion and wear behaviour of spark plasma–sintered Inconel 738LC alloy. *Int J Adv Manufacturing Technol* 2019; 104(9): 4195–4206.

29. Ogungbii O, Jamiru T, Sadiku R, et al. Spark plasma sintering of graphene-reinforced Inconel 738LC alloy: wear and corrosion performance. *Metals Mater Int* 2020; 28: 1–15.

30. Oke SR, Falodun OE, Okoro AM, et al. Effect of ZrO2 addition on densification and properties of Spark plasma sintered Ti6Al4V-Ni. *Mater Today Proc* 2019; 18: 2454–2460.

31. Ma S, Li A, Zhou S, et al. Microstructure and mechanical properties of nickel strengthened by Y2O3 through roll-milling and spark plasma sintering. *J Alloys Compd* 2018; 750: 911–916.

32. Asharaf Ali S, Karthikeyan S, Deivanai M, et al. Zirconia: properties and application a review. *Pakistan Oral Dental J* 2014; 34(1).

33. Wyckoff RWG and Wyckoff RW. *Crystal structures*. New York, NY: Interscience publishers.

34. Lundqvist D. X-ray studies on the ternary system Fe-Ni-S. *Arkiv for Kemi. Mineralogi Och Geologi* 1947; 24(22): 12.

35. Falodun OE, Oke SR, Obadele BA, et al. Influence of SiAlON ceramic reinforcement on Ti6Al4V alloy matrix via spark plasma sintering technique. *Metals Mater Int* 2021; 27(6): 1769–1778.

36. Baghery P, Farzam M, Mousavi A, et al. Ni-TiO2 nano-composite coating with high resistance to corrosion and wear. *Surf Coat Technol* 2010; 204(23): 3804–3810.

37. Asl MS, Ahmad Z, Parvizi S, et al. Contribution of SiC particle size and spark plasma sintering conditions on grain growth and hardness of TiB2 composites. *Ceramics Int* 2017; 43(16): 13924–13931.

38. Mampuru KM, Ajenifuju E, Popoola A, et al. Effect of silicon carbide addition on the microstructure, hardness and densification properties of spark plasma sintered Ni-Zn-Al alloy. *J King Saud University-Sci 2019; 31(4): 1122–1126.

39. Obadele BA, Andrews A, Olubambi PA, et al. Effect of ZrO2 addition on the dry sliding wear behavior of laser clad Ti6Al4V alloy. 2015; 328: 295–300.

40. Li N, Xu H, Li X, et al. Tribological properties and corrosion resistance of porous structure Ni-Mo/ZrO2 Alloys. *Coatings* 2020; 10(8): 767.

41. Dada M, Popoola P, Mathe N, et al. Investigating the elastic modulus and hardness properties of a high entropy alloy coating using nanoindentation. *Int J Lightweight Mater Manufacture* 2021; 4(3): 339–345.

42. Rodriguez R and Gutierrez I. Correlation between nanoindentation and tensile properties: influence of the indentation size effect. *Mater Sci Eng A* 2003; 361(1–2): 377–384.

43. Kashavate V, Praveen Kumar B, Singh J, et al. Analysis of indentation size effect (ISE) in nanoindentation hardness in polycrystalline PMN-PT piezoceramics with different domain configurations. *Ceramics Int* 2021; 47(9): 11870–11877.

44. Efe GC, Ipek M, Zeytin S, et al. An investigation of the effect of SiC particle size on Cu–SiC composites. *Composites Part B: Eng* 2012; 43(4): 1813–1822.

45. Rominiyi AL, Shongwe MB, Jeje SO, et al. Microstructure, tribological and oxidation behaviour of spark plasma sintered Ti-Ni-xTiCN composites. *J Alloys Compd* 2022; 890: 161857.

46. Hutchings IM. *Tribology: friction and wear of engineering materials*. 1st ed. London: Edward Arnold; 1992.

47. Falodun OE, Obadele BA, Oke SR, et al. Influence of spark plasma sintering on microstructure and wear behaviour of Ti6Al4V reinforced with nanosized TiN. *Trans Nonferrous Metals Soc China* 2018; 28(1): 47–54.

48. Xu Z, Shi X, Zhai W, et al. Preparation and tribological properties of TiAl matrix composites reinforced by multilayer graphene. *Carbon* 2014; 67: 168–177.

49. Ogungbii O, Jamiru T, Sadiku R, et al. Corrosion and wear behaviour of spark plasma-sintered NiCrCoAlTiW-Ta Superalloy. *J Bio- and Tribo-Corrosion* 2020; 6(1): 1–13.

50. Zhen J, Li F, Zhu S, et al. Friction and wear behavior of nickel-alloy-based high temperature self-lubricating composites against Si3N4 and Inconel 718. *Tribology Int* 2014; 75: 1–9.

51. Lavella M and Botto DJW. Fretting wear characterization by point contact of nickel superalloy interfaces. *Wear* 2011; 271(9–10): 1543–1551.