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EFFECTS OF Ni CONTENT ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ALUMINA/SILICON CARBIDE TOOL COMPOSITES

VPLIV VSEBNOSTI NIKLJA NA MIKROSTRUKTURO IN MEHANSKE LASTNOSTI KOMPOZITOV NA OSNOVI Al_2O_3/SiC ZA REZALNA ORODJA

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Alumina-based ceramic composites are widely used for cutting tools owing to their high strength and oxidation resistance. However, their brittle nature and low toughness adversely affect their use and performance. In this study, alumina/silicon carbide (Al_2O_3/SiC) composites with different mass fractions of Ni (5 %, 10 %, 15 %, and 20 %) were prepared via vacuum hot-press sintering to optimise their strength and toughness. The structural and mechanical properties of the composites were characterised using various techniques. The hardness and bending strength of the Ni-doped Al_2O_3/SiC composites increased with the Ni content up to 10 % and decreased with a further increase in the Ni content. The maximum values of the hardness, bending strength, and fracture toughness were 7.19 GPa, 871.25 MPa, and 7.90 MPa·m$^{1/2}$, respectively. Thus, the addition of Ni improved the mechanical properties overall and provided a toughening effect.

Keywords: Nickel, ceramic composite, toughness

Keramični kompoziti na osnovi aluminijevega oksida se veliko uporabljajo za rezalna orodja, ker imajo visoko trdnost in odpornost proti oksidaciji pri visokih temperaturah. Vendar pa zaradi njihove naravne krhkosti in nizke žilavosti niso dovolj učinkoviti. V tem članku avtorji opisujejo študijo kompozitov na osnovi Al_2O_3/SiC z različnim masnim deležem dodatka Ni. Avtorji so pripravili kompozite s 5 %, 10 %, 15 %, in 20 % Ni s postopkom vakumskega vročega sintranja pod tlakom, da bi optimizirali njihovo trdnost in žilavost. Sledila je strukturna in mehanska karakterizacija izdelanih kompozitov z različnimi tehnikami. Trdota in upogibna trdnost z Ni dopiranih Al_2O_3/SiC kompozitov je naraščala z vsebnostjo dodanega Ni do 10% in nato je začela počasi padati z nadaljnjim naraščanjem vsebnosti Ni. Optimalne dosežene vrednosti trdote, upogibne trdnosti in lomne žilavosti pri dodatku 10% Ni so bile 7,19 GPa, 871,25 MPa in 7,90 MPa·m$^{1/2}$. Na ta način so avtorji dokazali, da je z dodatkom niklja možno izboljšati žilavost te vrste keramičnih kompozitov.

Ključne besede: nikelj, keramični kompozit, žilavost

1 INTRODUCTION

Alumina (Al_2O_3)-based ceramic cutting tools are widely used owing to their high mechanical strength and oxidation/high-temperature resistance, chemical stability, and cost-effectiveness. However, the high brittleness and low toughness of Al_2O_3 affect their performance and reliability. To improve the performance of ceramic materials,[1,2] toughening methods such as particle-dispersion toughening, whisker toughening, phase-change toughening, and synergistic toughening have been explored.[3] Zhu et al.[4] studied zirconia-toughened Al_2O_3 ceramics and reported that samples sintered at 1650 °C with a relative density of 96.65 % exhibited the best mechanical performance, with a flexural strength of 572.0 MPa, Vickers hardness of 16.2 GPa, and fracture toughness of 7.4 MPa·m$^{1/2}$. Such ceramics have excellent mechanical properties, giving them considerable potential for dental applications. Chin et al.[5] examined the interactions between crack deflection and plastic deformation in Al_2O_3/Ti_3SiC_2 and found that the toughening effect stemmed mainly from crack deflection.

Among these toughened alumina ceramics, silicon carbide (SiC)-reinforced Al_2O_3 matrix composites exhibit outstanding mechanical properties. The particle bridging at the crack tip and the change in fracture mode caused by residual stress and crack deflection are the primary strengthening and toughening mechanisms.[6] Moreover, transgranular SiC particles undergo less shrinkage than the surrounding matrix and provide a hydrostatic "expansion" effect in the core of each Al_2O_3 grain. Such grain expansion tightens the internal Al_2O_3 grain bound-

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aries, shielding both the weakly bonded and unbonded (cracked) grain boundaries.[7]

However, the toughening effect of Al_2O_3/SiC remains limited. The addition of different types of particles to Al_2O_3/SiC composites can further increase their toughness and bending strengths.[8–11] Ni is a useful metal that is added to improve the fracture toughness. Gustafsson et al.[12] reported that the addition of Ni in Al_2O_3-based materials prepared via *in situ* reactions resulted in the formation of dispersed Ni phases at the grain boundaries, which increased the strength and toughness of these materials. Moreover, the incorporation of Ni impedes the grain growth, increasing the hardness and wear resistance of composite materials. Wu et al.[13] researched self-lubricating cutting tools that contained Ni-coated hexagonal boron nitride and reported that a tool with a Ni coating had better densification, wear resistance, and cutting performance than an uncoated tool, as well as superior mechanical properties.

In Al_2O_3-based composite materials, Ni is typically present as a dispersed phase between the matrix and grain boundaries. Studies have demonstrated that an optimal amount of Ni can refine the grain structure, improving both the material strength and hardness. For instance, when Ni is incorporated into Al_2O_3/SiC composites via an *in-situ* reaction, the formation of Ni particles at the grain boundaries effectively immobilises the Al_2O_3/SiC, impeding the grain growth and consequently enhancing the flexural strength and fracture toughness.[14] Martínez et al.[15] reported the outstanding mechanical properties of particle-reinforced Ni–alumina metal matrix nanocomposites. After 10 h of milling, the Al_2O_3 nanoparticles were well-dispersed in the Ni matrix, reinforcing the composites. Firoozbakht et al.[16] researched Al_2O_3-SiC-Ni nanocermets in an air environment and found that the microstructure of sintered samples consisted of a continuous Ni network surrounding Al_2O_3 grains, with no structural defects or Ni oxidation.

The Ni toughening mechanism is effective, but does not fully explain the toughening of the tool composites. To improve the mechanical performance of ceramic tool composites, we prepared Ni-doped Al_2O_3/SiC composites via hot-press sintering. We then studied the effects of the Ni content on their mechanical properties to clarify the mechanism of metal toughening in multiphase ceramics.

2 EXPERIMENTAL PART

High-purity powders of Al_2O_3, SiC, and Ni were obtained from Yijia Metal Products Co., Ltd. (Jiaxing, China), and Al_2O_3 with 5 % SiC (mass fraction) was used as the base material. The Ni powders were added in mass fractions of 5 %, 10 %, 15 %, and 20 % to prepare the composite materials. The physical properties of the raw materials are presented in **Table 1**, and the compositions of the prepared samples are presented in **Table 2**.

Table 1: Properties of the raw materials

Material	Molecular weight (g/mol)	Purity (%)	Particle size (μm)
Al_2O_3	101.96	99.99	65
SiC	40.1	99.99	65
Ni	58.69	99.99	65

Table 2: Compositions of the different samples (g)

Samples (with different Ni contents)	Al_2O_3	SiC	Ni	Gross weight
0 % Ni	95	5	0	100
5 % Ni	90	5	5	100
10 % Ni	85	5	10	100
15 % Ni	80	5	15	100
20 % Ni	75	5	20	100

A planetary ball mill (XQM-4-KL, China) was used to mix the powder samples, with ethanol and Al_2O_3 balls as the milling media. The ball-milling speed was 250 min^{-1}, and the duration of milling was 5 h. The composite materials with different Ni contents were prepared via vacuum hot-press sintering (VHP140/25-2300, China) at 1400 °C and 40 MPa with a heating rate of 10 °C/min and a holding time for sintering of 60 min. Their structures were characterised using X-ray diffraction (XRD; TD-3500, China) with a scanning speed of 6°/min, scanning range of 0°–90°, step size of 0.04°, and sampling time of 0.4 s. The actual densities (ρ_1) of the samples were measured using a density tester (AR-150ME, China). The theoretical densities (ρ_2) were calculated from the theoretical densities of Al_2O_3, SiC, and Ni, and the relative densities (ρ) were calculated using the following equation.

$$\rho = \rho_1/\rho_2 \tag{1}$$

The microhardnesses of the composite materials were evaluated using a Vickers hardness tester (HXD-100TM/LCD, China) under a load of 9.8 N for a duration of 15 s. The bending strength was determined via three-point tests using a universal testing machine (WDW-2.0, China). The sample size was 30 mm × 4 mm × 3 mm. The crossbeam speed of the testing machine was set to 0.5 mm/min, and the bending strength was calculated as follows:

$$\sigma = \frac{3FL}{2bd^2} \tag{2}$$

where L represents the lower span of the fixture, b represents the width of the sample (4 mm), and d represents the height of the sample (3 mm). Scanning electron microscopy (SEM; HITACHI S-3400, Japan) was used to characterise the fracture sections of the bent specimens. The fracture toughness of the material was measured via micro-Vickers hardness tester (HVS-30, China) at a load

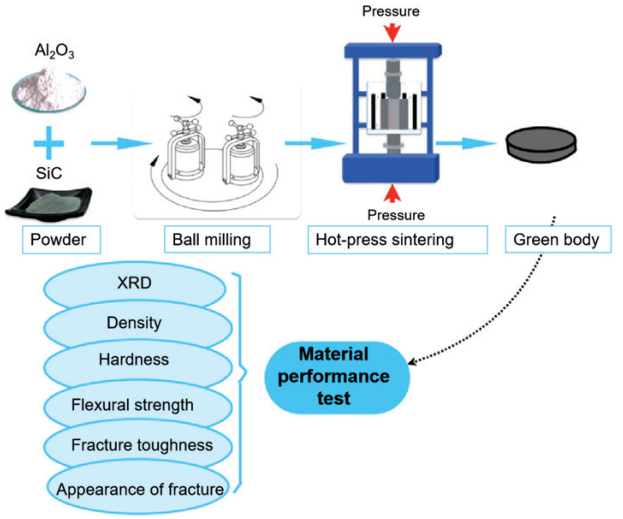

Figure 1: Preparation and characterization of Ni-doped Al_2O_3/SiC composites

of 294 N, which was held for 30 s. It was calculated as follows:

$$K_{IC} = \beta \cdot \left(\frac{E}{H}\right)^{1/2} \left(\frac{F}{C^{3/2}}\right) \quad (3)$$

where K_{IC} represents the fracture toughness, β is the test constant (0.016), E represents the Young's modulus of the material (in GPa), H represents the Vickers hardness of the material (in GPa), F represents the acting load of the Vickers hardness indentation (in N), and C represents the average indentation-crack radius (in mm).

In this study, One-way ANOVA was conducted using the IBM SPSS Statistics 19 software, with results presented as the mean ± standard error (n = 3). For statistical significance, a p-value threshold of less than 0.05 was adopted. In the graphical representations, extremely significant differences ($p < 0.001$), highly significant differences ($p < 0.01$), and significant differences ($p < 0.05$) are denoted by ***, **, and *, respectively.

3 RESULTS AND DISCUSSION

3.1 Structure

Figure 2 shows the XRD patterns of the Al_2O_3/SiC composites prepared with different Ni contents. All the samples exhibit characteristic peaks for standard Al_2O_3. The main peaks are at 25°, 35°, 38°, 44°, 53°, 57°, 66°, and 68°, indicating that the addition of Si, C, Ni did not affect the peak intensity of Al_2O_3. Furthermore, the peak at 45° corresponding to Ni was observed in the samples with Ni contents of 10–20 *w*/%. The intensity of the Ni diffraction peak increased with the Ni content. In addition, peaks corresponding to metallic Ni and Ni compounds, such as $Ni(CO)_4$ at 46°, were observed for the samples with high Ni contents (10 %, 15 %, and 20 %), accompanied by small peaks nearby. At 1400 **?**, Ni is semi-molten, most of the peak sites (Al_2O_3-related peak

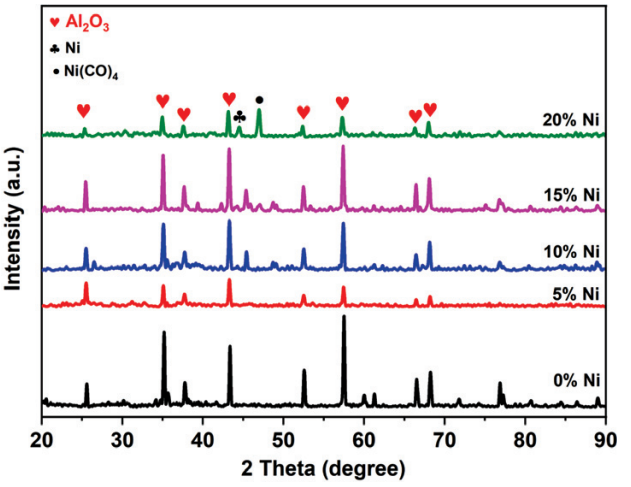

Figure 2: XRD patterns of Al_2O_3/SiC composites with different Ni contents

locations) in the samples are similar, and the reactions between Ni and Al_2O_3 are thought to mainly occur in the first atomic layer of the interface[17]. Fluctuant peaks at 44–50° suggest that the chemical reaction produces Ni compounds at high temperatures. Most of the Al_2O_3-related peaks and Ni-related peaks at 45° reported by Wang et al.[18] were similar. Ni was present as a single phase in the composite materials, and it also reacted with Al and O[19]. These results indicated that Ni was successfully added as a single phase or a dispersion in the composites.

3.2 Relative density

The density of the Al_2O_3/SiC composite without the addition of Ni (0 %) was measured as 95.24 %, and it increased with the Ni content (Figure 3). The density of the composite reached 98.00 % at 15 % Ni. The increase in the density is attributed to the filling of the gaps between the Al_2O_3 and SiC particles by the Ni particles[20,21].

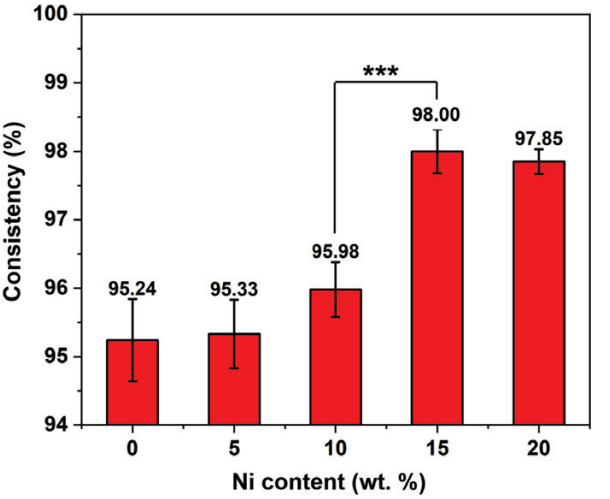

Figure 3: Densities of Al_2O_3/SiC composites with different Ni contents1

The wetting effect of Ni between alumina and SiC reduces the interfacial energy between particles, which facilitates the rearrangement and compact stacking of particles, increasing the density.[22,23] This strengthens the bonding between the particles after sintering. With an increase in the Ni content, more gaps are filled, and the density increases.

3.3 Hardness

As shown in **Figure 4**, the hardness of the Al_2O_3/SiC composite material increases from 6.25 GPa (0 % Ni) to a maximum of 7.25 GPa with the addition of 10 % Ni. With a further increase in the Ni content, the hardness of the composite decreases (e.g., 5.9 GPa at 20 % Ni). As the relative density increases with the Ni content, atoms exhibit a higher degree of compactness in their arrangement.[24] The hardness shows a trend similar to that of the relative density with the addition of 5 % and 10 % Ni. However, when the Ni content surpasses a critical threshold, it may exceed the solubility limit in the matrix metal, resulting in the excessive precipitation of Ni or the formation of coarse intermetallic compounds within the brittle phases[25]. These coarse secondary phases not only fail to effectively enhance the hardness but can also compromise the overall mechanical properties of the material, reducing the hardness. Our results indicated that the hardness first increased and then decreased and that the Al_2O_3/SiC composites with 15 % Ni had the highest hardness.

3.4 Flexural strength

Figure 5 presents the flexural strengths of the Al_2O_3/SiC composites with different Ni contents. Similar to the trend in hardness, with an increase in the Ni content, the flexural strength of the composites first increased and then decreased. When the Ni content was 15 %, the flexural strength of the Al_2O_3/SiC composite reached a maximum of approximately 1175.00 MPa. The addition of Ni may enhance the interface bonding, and the cementing action increases the flexural strength. The phase structure of the material may undergo changes with a further increase in Ni content, resulting in an excess of brittle phases or secondary phases[15], which increases the brittleness of the material.

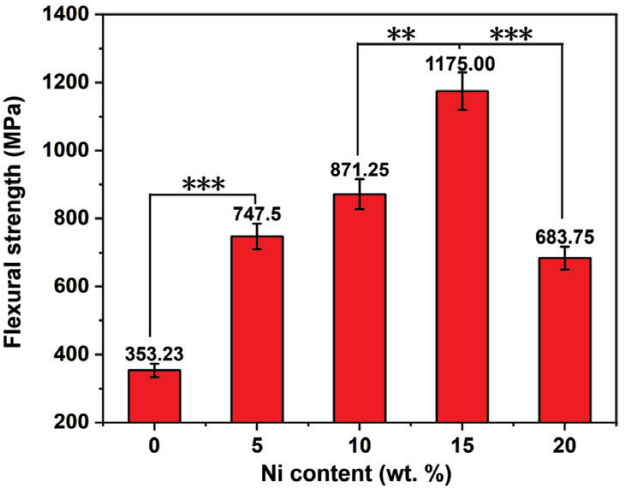

Figure 5: Flexural strengths of the Al_2O_3/SiC composites with different Ni contents

3.5 Fracture toughness

As shown in **Figure 6**, the fracture toughness of the Al_2O_3/SiC composite (6.63 MPa·$m^{1/2}$) increases to a maximum of 7.9 MPa·$m^{1/2}$ at 10 % Ni and then decreases with a further increase in the Ni content. Thus, the addition of 5 %–10 % Ni increase the fracture toughness of the Al_2O_3/SiC composite ceramics. Ni, being a metal, exhibits high ductility and plasticity. The presence of the Ni phase within the ceramic matrix effectively impedes the crack propagation during the cracking process and

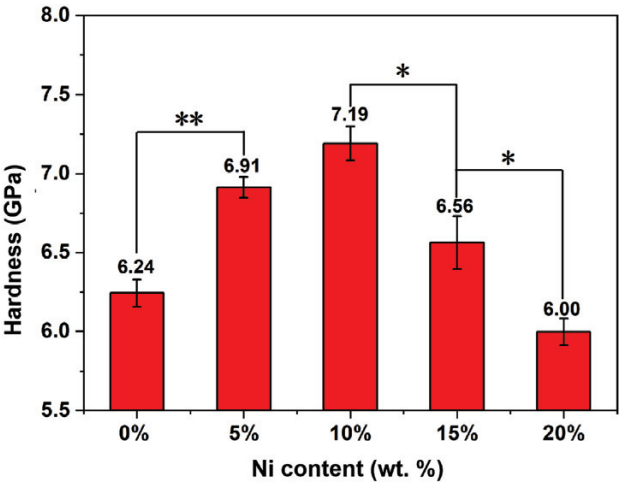

Figure 4: Hardnesses of the Al_2O_3/SiC composites with different Ni contents

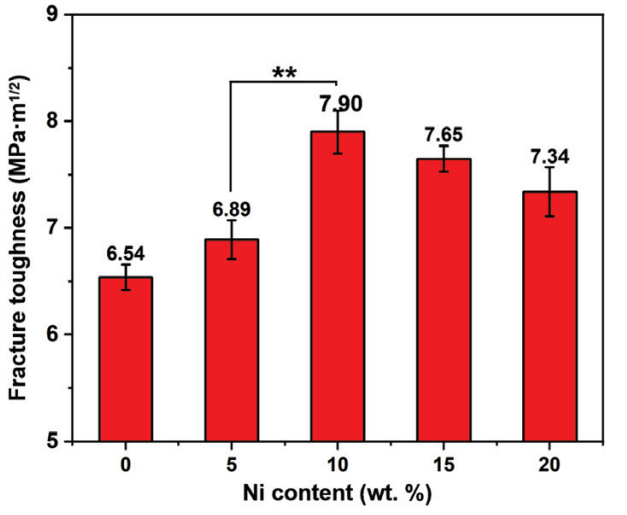

Figure 6: Fracture toughnesses of the Al_2O_3/SiC composites with different Ni contents

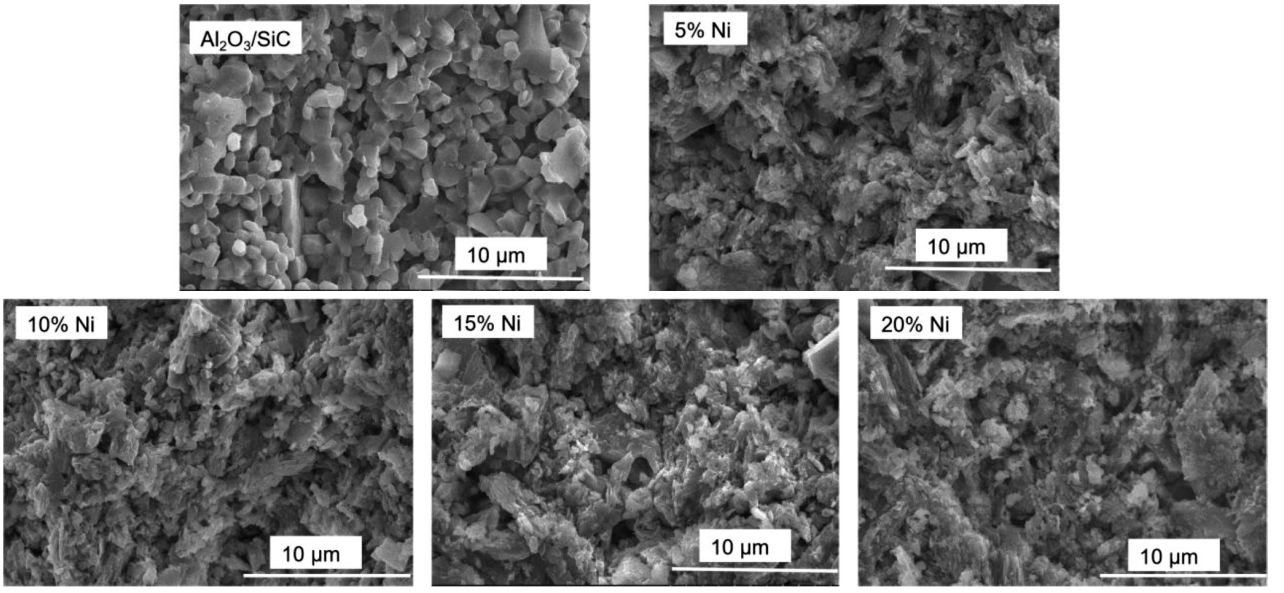

Figure 7: SEM images of the fracture surfaces of Al_2O_3/SiC composites with different Ni contents

increases the energy required for crack expansion,[26,27] which contributes to the fracture toughness. Additionally, the plastic deformation of the Ni enables the absorption of the energy required for crack propagation,[28,29] further increasing the fracture toughness. When the content exceeds 10 %, the fracture toughness of the Al_2O_3/SiC composite material decreases. On one hand, the strength of the interfacial bond between the Ni and alumina may be compromised due to excessive Ni content. High levels of Ni can destabilize or weaken the interfacial bond, leading to an increased likelihood of crack propagation at the interface, thereby reducing fracture toughness. On the other hand, the distribution and morphology of the Ni particles at high concentrations may induce stress concentration. If the Ni particles are unevenly distributed or form large agglomerates within the Al_2O_3 matrix, these regions can become stress-concentration points, further diminishing the overall toughness of the material.

3.6 Fracture morphology

Figure 7 shows SEM images of the fractured sections of the different composites. Long columnar crystals can be observed in the pure Al_2O_3/SiC composites, and regular grain boundaries indicate the mode of intergranular fracture. However, the Ni-doped Al_2O_3/SiC composites exhibit smaller grains with obvious directional arrangement and grain-pulling holes. The addition of a metal element inhibits the grain growth, which results in grain refinement. Meanwhile, an indistinct grain boundary indicates transgranular fracture. Furthermore, the metal particles act as deflection or bridging agents[30] that result in crack deflection and interlocking, toughening the composite material.

According to the literature,[31–33] the grain size of the alumina decreases with an increase in its Ni content. When an Ni-doped Al_2O_3/SiC composite is sintered at a high temperature, the Ni melts and fills the gap between the Al_2O_3 and SiC particles, increasing the density of the material and suppressing the crack formation. In this study, the fracture mode of the composite with 5 % Ni was almost completely intergranular fracture, with little transgranular fracture. However, with an increase in the Ni content, the fracture mode changed from intergranular to transgranular, indicating that the addition of Ni can strengthen the grain boundaries of Al_2O_3 grains, hinder intergranular crack growth, and improve the mechanical properties of the material. Cracks propagate extensively within the grain interior, leading to elongated fracture paths and increased energy consumption. This mode of transgranular fracture necessitates overcoming robust intergranular bonding forces, with an increased fracture toughness.

When a small amount of Ni is added, the sintering activity of the material is improved, the sintering temperature is reduced, and the growth of Al_2O_3 particles[34] is inhibited. This results in grain refinement and grain-boundary strengthening.[35] With an increase in the Ni content to a certain limit, enhanced grain refinement and grain-boundary strengthening can be achieved. After the high-temperature sintering of the Al_2O_3/SiC composite material, Ni no longer exists in the form of a single phase in the composite material; the reaction produces Ni-containing compounds, which are embedded in the Al_2O_3/SiC composite material and enhance its performance.[36,37]

At higher contents, Ni agglomerates and grows into larger particles. These particles impede grain-boundary movement, improving the grain connectivity.[38] Under the effect of an external load, stress concentration around

these particles[39] results in microcracks and expansion that can lead to fracture of the material. Thus, the mechanical properties of the Al_2O_3/SiC material are weakened at high Ni contents.

4 CONCLUSIONS

Al_2O_3/SiC composites with different Ni contents were prepared via vacuum hot-press sintering. The following conclusions are drawn:

When the mass fraction of Ni is increased from 0 % to 15 %, the bonding effect of Ni is enhanced, and the density of Al_2O_3/SiC composite ceramics increases. When the Ni content is increased to 20 %, Ni agglomerates and becomes unevenly distributed in the material, reducing the density, strength, and toughness of the material.

With an increase in the Ni content, the hardness and bending strength of the Al_2O_3/SiC composite first increase and then decrease. The highest hardness is obtained when the Ni mass fraction is 10 %, and the bending strength is maximised at 15 % Ni.

When the mass fraction of Ni is 10 %, the hardness, bending strength, and fracture toughness are 7.19 GPa, 871.25 MPa, and 7.90 MPa·$m^{1/2}$, respectively, i.e., the comprehensive mechanical properties of Al_2O_3/SiC are best at 10 % Ni.

In summary, the addition of Ni increased the density and comprehensively enhanced the mechanical properties of the Al_2O_3/SiC composite material by functioning as a reinforcing and toughening agent. Thus, the addition of metals to ceramic composite materials can improve their performance, which provides a new direction for the development of composites for Al_2O_3/SiC tools.

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