



Analysis of friction and wear performance of Y_2O_3 -doped Si_3N_4 ceramic using the estimation of stress

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Received: 19 November 2022 / Accepted: 10 February 2023 / Published online: 30 March 2023
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Abstract

A variety of combinations of Y_2O_3 and Al_2O_3 were used as sintering aids in the fabrication of Si_3N_4 ceramics via gas pressure sintering (GPS). Based on the prediction of the residual thermal stress at the interface, the influence of crystal phases on the wear properties of Si_3N_4 ceramics was analyzed. As a result, with the increase of Y_2O_3 addition from 1 to 9 wt%, there are four kinds of crystal phases being firmly discovered in the sample, $Y_2Si_2O_7$, $YSiO_2N$, $Y_4Si_2O_7N_2$, and $Y_2Si_3O_3N_4$. The crystal phases of $Y_2Si_2O_7$, $YSiO_2N$, and $Y_4Si_2O_7N_2$ inhibit the particle flaking during the friction process, which greatly inhibited the progress of the abrasive wear, and optimize the wear resistance of silicon nitride ceramics. According to Selsing's model, it can be predicted that the interface residual thermal stress caused by the intergranular phase $Y_2Si_3O_3N_4$ is 22–35% higher than that caused by the other three intergranular phases. The crystal phase $Y_2Si_3O_3N_4$ intensifies the shedding of grains during wear, and reduces the wear performance of silicon nitride ceramics. In addition, the silicon nitride ceramics with 5wt% Y_2O_3 show better wear resistance, and the wear rate is $1.8 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$.

Keywords Silicon nitride · Crystal phases · Residual thermal stress · Wear properties

1 Introduction

Si_3N_4 is an all-purpose ceramic with desirable mechanical properties, including excellent strength and low creep at high temperatures, thermal shock resistance, wear resistance, corrosion resistance, and appreciable theoretical thermal conductivities, which is ideal material for the preparation of wear-resistant basic parts [1–3]. At the same time, wear becomes the main reason for the failure of these components. Substantial efforts have been made to improve the wear resistance of silicon nitride ceramics [4–7]. Previous research has indicated that the wear properties of silicon nitride ceramic are greatly affected by the hardness ratio between the abrasive particles and the material matrix, and the specific wear rate is maximum when the abrasive particles are harder than the matrix [8]. Since the

β - Si_3N_4 particles (20 Gpa) that exfoliated during the wear process are much harder than the Si_3N_4 (16–17 Gpa) ceramic matrix, the effective ways to improve the wear properties of silicon nitride ceramics are controlling the interfacial debonding. Francisco found that the existence of residual thermal stress at the interface promoted interface debonding [9]. Xue et al. [10] found that the residual thermal stress originating from the thermal expansion coefficient mismatches between the β - Si_3N_4 and the crystal phase.

Y_2O_3 is the most frequently used sintering aid in investigating and exploring the mechanical properties of silicon nitride. Quaternary Y–Si–O–N compounds are the frequently detected crystal phase in investigating Si_3N_4 ceramic when Y_2O_3 is used as sintering additive. It has been reported that there are six quaternary crystal phase being firmly discovered in the Y–Si–O–N family: Y_2SiO_5 , $Y_2Si_2O_7$, $Y_5Si_3O_{12}N$, $YSiO_2N$, $Y_4Si_4O_7N_2$, and $Y_2Si_3O_3N_4$ [11, 12]. Moreover, the thermal expansion coefficients of these crystalline phases are obviously different [13–15].

However, to the authors' knowledge, little information is available on the relationship between the wear properties of silicon nitride ceramics and Y_2O_3 . Therefore, the present study explores the wear performance of silicon nitride doped with Y_2O_3 , focusing on the influence of crystal phase on

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wear performance of silicon nitride ceramics. This study provides technical guidance for optimizing the preparation process of Si₃N₄ ceramic.

2 Estimation model of interface residual thermal stress

2.1 Generation of interface residual thermal stress

Figure 1 shows the transformation process of the α phase to the β phase in Si₃N₄. SiO₂ is an inevitable phase in Si₃N₄ powders. The sintering aid reacted with the SiO₂ film on the surface of the Si₃N₄ powder in an oxidation–reduction manner, thereby removing SiO₂ and generating a liquid phase. α-Si₃N₄ dissolves in the liquid phase generated by the sintering aid, and when the solution reaches saturation, it precipitated out again as β-Si₃N₄ and existed in the form of rod-like β-Si₃N₄ after cooling. In this process, the residual thermal stress caused by thermal expansion mismatch between crystal phase and β-Si₃N₄ may promote interface debonding of Si₃N₄ ceramics.

2.2 Thermal expansion coefficient of crystal phase

The thermal expansion coefficient of each crystal phase in the SiO₂–Y₂O₃–Si₃N₄ system was calculated using the bond valence model. For a multicomponent compound

system with n types of binary systems, its thermal expansion coefficient can be obtained on the basis of the definition by the following equation [16]:

$$\alpha = \sum_{\mu=1}^n v_l^{\mu} \alpha_l^{\mu}, \tag{1}$$

v_l^{μ} and α_l^{μ} can be calculated as

$$v_l^{\mu} = \frac{(n^{\mu} R_{ij, \mu}^3)^{1/3}}{\sum_{\mu=1}^n (n^{\mu} R_{ij, \mu}^3)^{1/3}} \tag{2}$$

$$\alpha_l^{\mu} = \frac{1.35kR_{ij}}{\gamma \left(\frac{8S_{ij}}{3}\right)^{3/2} \left(\frac{1}{b} - \frac{2}{R_{ij}}\right)} \tag{3}$$

$$S_{ij} = \left(\frac{R_0 - R_{ij}}{b}\right), \tag{4}$$

where a is the linear thermal expansion coefficient of the intergranular phase, $\gamma = 23nN\text{\AA}^2$ (electrons)⁻², R_{ij} is the length of the bond between the two atoms i and j , b is a universal constant equal to 0.37 Å, R_0 is the bond valence parameter, k is the Boltzmann constant, μ represents the bond type in the unit cell, and n represents the number of binary systems in the unit cell. The binary structural units,

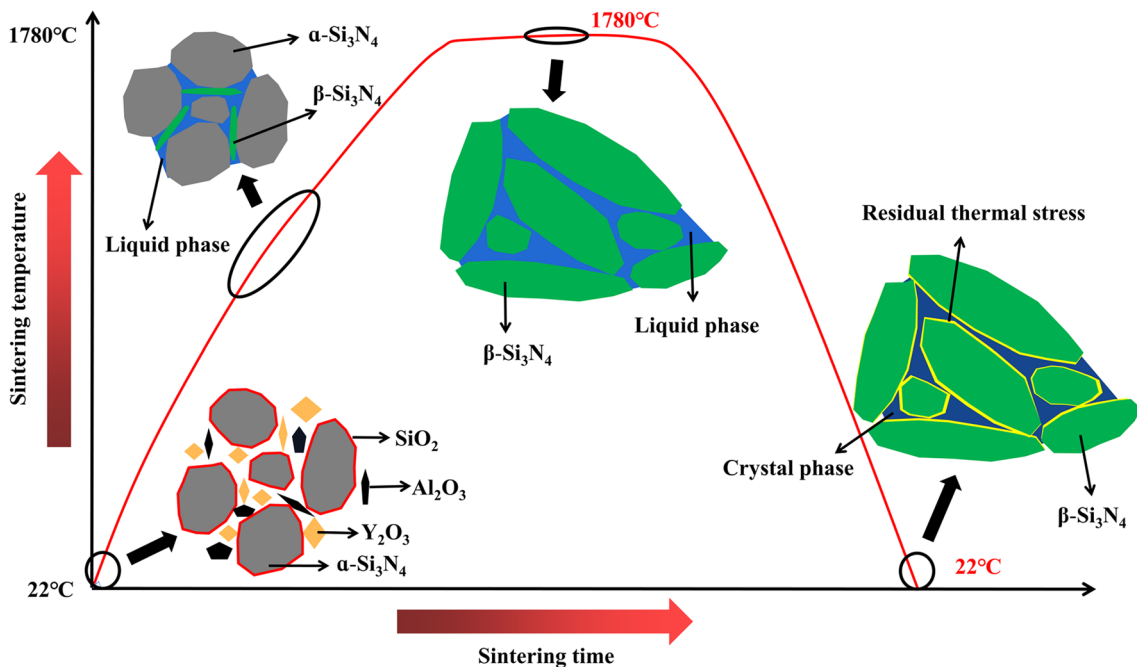


Fig. 1 Schematic diagram of α → β phase transition in silicon nitride sample

Table 1 Structural units, calculated average bond lengths (Å), and bond valence parameter (*R*₀) of Y(O/N)_{*n*} polyhedra and Si(O/N)₄ tetrahedra of the six Y–Si–O–N quaternary compounds [14, 17]

Compounds	Structural units	Average bond length (Å)		<i>R</i> ₀
		Y(O/N) _{<i>n</i>}	Si(O/N) ₄	
Y ₂ Si ₂ O ₇	Y–O ₆ Si–O ₄ O ₁ –Si ₂	2.028	1.624	0.37
Y ₂ SiO ₅	Y ₁ –O ₇ Y ₂ –O ₆ O ₁ –Y ₄	2.019	1.622	0.37
Y ₅ Si ₃ O ₁₂ N	Y ₁ –O ₈ N Y ₂ –O ₇ Y ₃ –O ₆ N Y ₄ –O ₇	2.378	1.612	0.37
YSiO ₂ N	Y ₁ –O ₆ N ₂ Y ₂ –O ₆ N ₂ Y ₃ –O ₆ N ₂	2.438	1.648	0.37
Y ₄ Si ₂ O ₇ N ₂	Y ₁ –O ₅ N ₂ Y ₂ –O ₇ Y ₃ –O ₅ N Y ₄ –O ₅ N ₂	2.331	1.643	0.37
Y ₂ Si ₃ O ₃ N ₄	Y ₁ –O ₃ N ₅ Y ₂ –O ₅ N ₃ Si ₁ –ON ₃ Si ₂ –ON ₃ Si ₃ –O ₂ N ₂	2.478	1.671	0.37

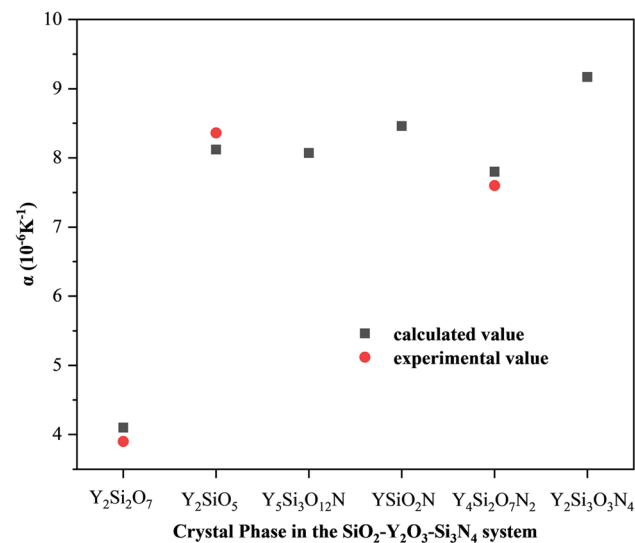


Fig. 2 Thermal expansion coefficient of crystal phase

bond length, and bond valence parameters of the six crystal phases are shown in Table 1.

Figure 2 shows the calculated and experimental values of the thermal expansion coefficients of the six crystal phases of the SiO₂–Y₂O₃–Si₃N₄ system. The experimental values were obtained from literatures [13–15]. Here,

all the experimental thermal expansion coefficients are those at or near room temperature in the available literatures. Wherein, the calculated thermal expansion coefficients of crystal phases Y₂SiO₅, Y₂Si₂O₇, Y₅Si₃O₁₂N, YSiO₂N, Y₄Si₂O₇N₂, and Y₂Si₃O₃N₄ are 4.1 × 10^{−6} K^{−1}, 8.1 × 10^{−6} K^{−1}, 8.07 × 10^{−6} K^{−1}, 8.46 × 10^{−6} K^{−1}, 7.8 × 10^{−6} K^{−1}, and 9.17 × 10^{−6} K^{−1}, respectively. The calculated values of thermal expansion coefficient were compared with the experimental data, and the good agreement demonstrates the predictive power of Eq. (1).

2.3 Residual thermal stresses

According to Selsing's model [18, 19], the interfacial thermal residual stress between the crystal phase and silicon nitride could be calculated by

$$\sigma_r = \frac{\Delta\alpha \cdot \Delta T}{\frac{1-2\nu_1}{E_1} + \frac{(1+\nu_2)}{2E_2}}, \tag{5}$$

where, σ_r , $\Delta\alpha$, ΔT , ν , and E are the interfacial thermal residual stress, difference in coefficient of thermal expansion (CTE), temperature range over which stress is not relieved, Poisson's ratio, and elastic modulus, respectively. The subscripts 1 and 2 refer to the crystal phase and silicon nitride, respectively. The physical properties of the β -Si₃N₄ and the crystal phase are shown in Table 2.

Figure 3 shows the residual thermal stress at the interface between crystal phase and β -Si₃N₄. The residual thermal stress around crystal phases of Y₂SiO₅, Y₂Si₂O₇, Y₅Si₃O₁₂N, YSiO₂N, Y₄Si₂O₇N₂, and Y₂Si₃O₃N₄ are 37.53 MPa, 453.23 MPa, 524.44 MPa, 529.25 MPa, 442.64 MPa, and 678.4 MPa, respectively. The residual thermal stress of the samples with introducing crystal phases of Y₂Si₂O₇, Y₄Si₂O₇N₂, Y₂SiO₅, Y₅Si₃O₁₂N, and YSiO₂N shows a continuous upward trend. At the same time, the generation of the crystal phase Y₂Si₃O₃N₄

Table 2 Physical properties of β -Si₃N₄, Y₂SiO₅, Y₂Si₂O₇, Y₅Si₃O₁₂N, YSiO₂N, Y₄Si₂O₇N₂, and Y₂Si₃O₃N₄

Compounds	Young's modulus (<i>E</i> , GPa)	Poisson's ratio (ν)	Thermal expansion (α , K ^{−1})
β -Si ₃ N ₄	300[20]	0.28 [21]	3.5 × 10 ^{−6} [21]
Y ₂ Si ₂ O ₇	155 [14, 15]	0.27 [14]	4.1 × 10 ^{−6}
Y ₂ SiO ₅	124 [14, 15]	0.31 [14]	8.1 × 10 ^{−6}
Y ₅ Si ₃ O ₁₂ N	204 [22]	0.29 [22]	8.07 × 10 ^{−6}
YSiO ₂ N	196 [22]	0.27 [22]	8.46 × 10 ^{−6}
Y ₄ Si ₂ O ₇ N ₂	191 [15, 22]	0.28 [22]	7.8 × 10 ^{−6}
Y ₂ Si ₃ O ₃ N ₄	244 [22]	0.27 [22]	9.17 × 10 ^{−6}

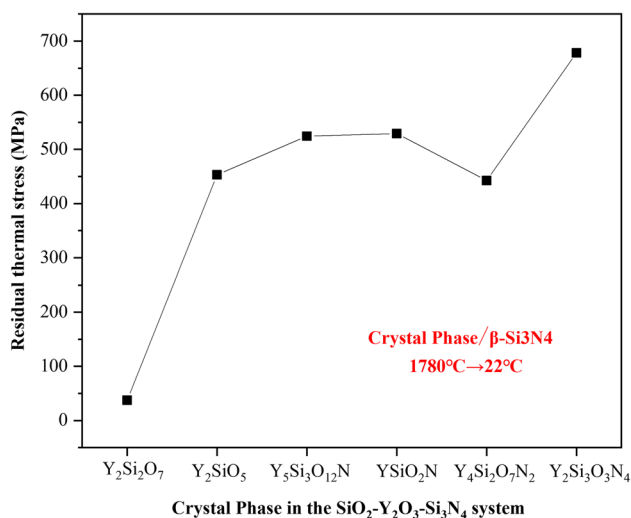


Fig. 3 Residual thermal stress between the crystal phase and silicon nitride

significantly increases the residual thermal stress at the interface, which may aggravate interface peeling during the wear process and reduce the wear performance of silicon nitride ceramic.

3 Materials and methods

3.1 Raw materials and experimental procedure

Si₃N₄, (E10 α-Si₃N₄, UBE, Japan), Y₂O₃, and Al₂O₃ powders (Shanghai Naiou Nanotechnology Co., Ltd.) were used as raw materials in this study. The raw materials were mixed according to the ratios given in Table 3.

The α-Si₃N₄ powder was mixed with Y₂O₃ and Al₂O₃ with agate balls in a nylon jar for 12 h in a planetary mill, using ethanol as a mixing medium. The slurries were dried at 80 °C. The mixed powder was milled and sieved through 80-screen sieve. After being uniaxially dry pressed at 50 MPa and cold isostatically pressed at 230 MPa, the green samples were sintered at 1780 °C under 100 bar

Table 3 The composition for each sample

Designation	Si ₃ N ₄ (wt%)	Y ₂ O ₃ (wt%)	Al ₂ O ₃ (wt%)	Sintering method	Temperature (°C)
1Y	94	1	5	Gas pressure sintering	1780
3Y	92	3	5		
5Y	90	5	5		
7Y	88	7	5		
9Y	86	9	5		

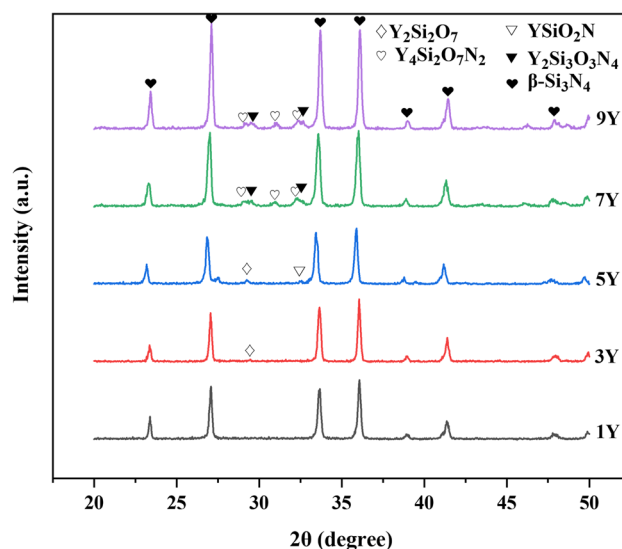


Fig. 4 XRD results of Si₃N₄ samples

nitrogen pressure, followed by furnace cooling down to room temperature (22 °C).

3.2 Characterization

An X-ray diffractometer was used to characterize the phase structure of sintered samples. The step size was set at 0.03° in a diffraction angle range of 20°–50°, and the scanning rate of 1°/min.

The wear properties of the samples were tested using the Rtec multifunctional friction and wear tester (MFT-5000, USA). Polished commercial Si₃N₄ balls (Sinoma High-tech Nitride Ceramics Co., Ltd.) with a diameter of 5.56 mm were used as friction pairs. At room temperature (22 °C), the friction pairs moves is a reciprocating relative motion. The load, stroke, and frequency of reciprocation were all set to 40 N, 4 mm, and 3 Hz, respectively.

After the wear test, the worn surface morphology of the sample was observed using scanning electron microscopy (SEM).

Meanwhile, the depth of the worn surface was measured using laser confocal microscopy.

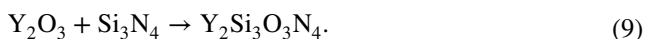
Finally, a VHX6000 ultradepth-of-field 3D contour graph (Keyence Corporation, Japan) was used to measure the topography profile curves of worn after the test. Then, using the formula $k = A/2(P \times f \times t)$ to calculate wear rates. A is the area of topography profile of worn surface which determined by ultradepth-of-field 3D contour graph, P is the applied load, f is sliding frequency, and t is total sliding time.

4 Results and discussion

4.1 Phase composition

Figure 4 shows the XRD patterns obtained for the as-sintered samples with different Y₂O₃ content. It can be seen that all main peaks are characteristic β-Si₃N₄ peaks, which indicates that the α-phase has been completely transformed into the β phase during the sintering. For Y₂O₃ content of 1 wt%, no other peaks were observed except for the characteristic β-Si₃N₄ peaks. For the sample with 3 wt% Y₂O₃, in addition to the β-phase peaks, low-intensity peaks characteristic for Y₂Si₂O₇ were detected. In the samples with 5 wt% Y₂O₃, the Y₂Si₂O₇ and YSiO₂N phase was found. For Y₂O₃ content of 7 and 9 wt%, the Y₄Si₂O₇N₂ phase was found.

The chemical reactions for the formation of the Y₂Si₂O₇, YSiO₂N, Y₄Si₂O₇N₂, and Y₂Si₃O₃N₄ phases are as follows [23–26]:



It can also be seen from the above that when the ratio of Y₂O₃/SiO₂ is 1/2, 2/1, 4/1, and 1/0, the corresponding crystal phases are Y₂Si₂O₇, YSiO₂N, Y₄Si₂O₇N₂, and Y₂Si₃O₃N₄, respectively.

4.2 Wear behavior

Figure 5 shows the wear surface morphologies of the samples. When the Y₂O₃ content was 1%, recurrent loads induced surface fatigue and crack, as shown in Fig. 5a. As the wear progressed, the cracks propagated and the scale debris accumulation layer were exfoliated, and then, a new surface was exposed. The surface morphology of the Si₃N₄ ceramics with 3wt% and 5wt% Y₂O₃ after Wear test is shown in Fig. 5b, c, respectively. The wear debris adhered to each other during the wear process. Most area of the surface remains smooth, and surface fatigue and crack occurred in a few isolated areas. When the Y₂O₃ content increased to 7%, wear took away the debris accumulation layer to expose a new surface, and Si₃N₄ particles' exfoliation resulted in the wear intensified, as shown in Fig. 5d. When the Y₂O₃ content increased to 9%, accompanied by large debris accumulation layer removed, the wear surface

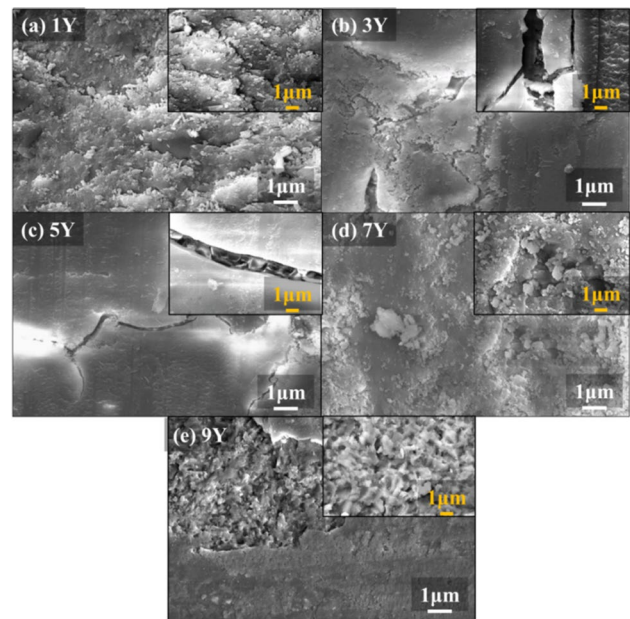


Fig. 5 Scanning electron micrographs (SEM) of the wear surface morphologies on the Si₃N₄ tested at 22 °C under a load of 40 N

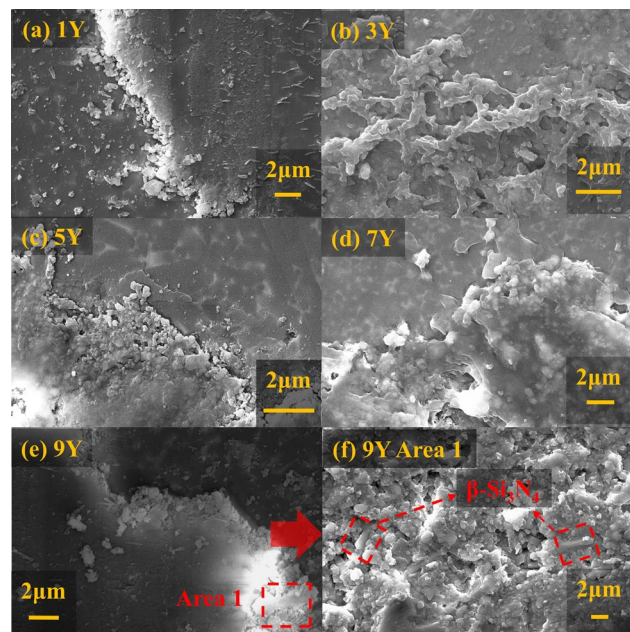


Fig. 6 Wear debris morphology of wear surface

has the appearance of a typical fracture surface of silicon nitride, indicating a possible intergranular fracture, as shown in Fig. 5e. This observation suggests that interface peeling is the predominant mode of wear.

To analyze wear mechanism of Si₃N₄ ceramics by adding Y₂O₃, the debris morphology of wear surface was also observed and analyzed. The morphology of the wear debris

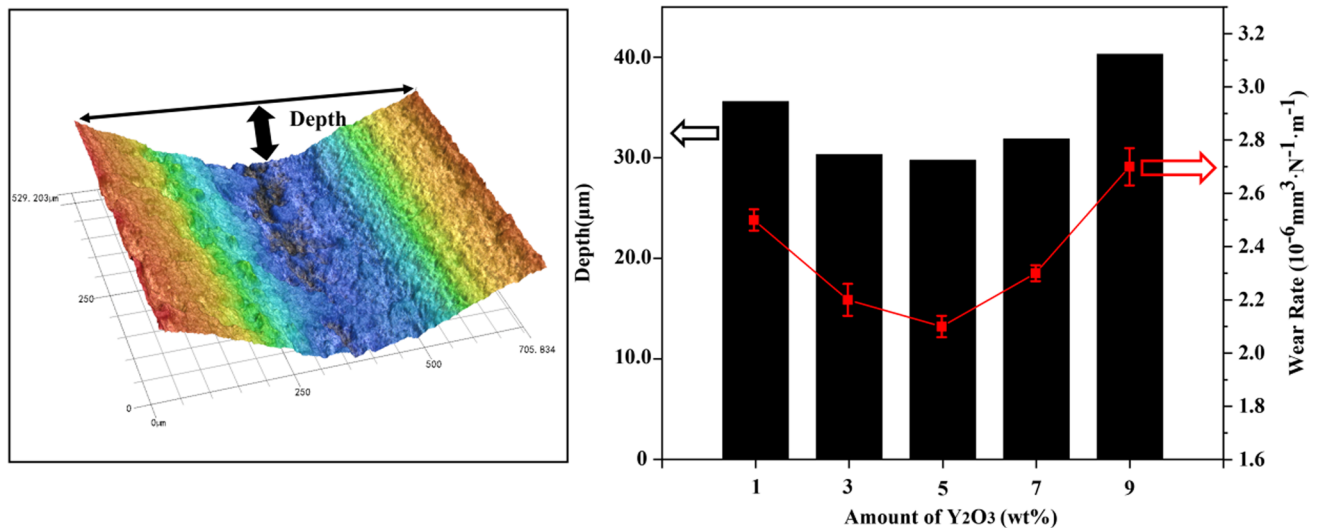


Fig. 7 Cross-sectional depth of the Si₃N₄ ceramic sample wear track and wear rate

of the Si₃N₄ ceramic with 1, 3, and 5 wt% Y₂O₃ is block-like debris, as shown in Fig. 6a–c. This showed that surface fatigue is the dominant wear mechanism in Si₃N₄ ceramics. For the Si₃N₄ ceramic with 7 and 9 wt% Y₂O₃, the morphology of the wear debris is particle-like debris and β-Si₃N₄, as shown in Fig. 6d–f. This showed that abrasive wear is the dominant wear mechanism in Si₃N₄ ceramics. Based on the above analysis, the existence of Y₂Si₃O₃N₄ phases caused a large residual thermal stress at the interface, and thus increased the β-Si₃N₄ fracture of subsurface during the wear process, which greatly promoted the progress of abrasive wear.

4.2.1 Wear surface depth and wear rate

The wear depth and the wear rate of the silicon nitride ceramics are shown in Fig. 7. When the Y₂O₃ content increased from 1 to 5%, the wear depth decreased from 35.6 μm to 29.8 μm, and the wear rate of the silicon nitride ceramics decreased from $2.5 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ to $1.8 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$. The wear depth and wear rate increased by 35.2% and 53.8% after the Y₂O₃ content increased from 5 wt% to 9 wt%. Obviously, the silicon nitride ceramics with 5 wt% Y₂O₃ shows better wear resistance. Since the less residual thermal stress is helpful to improved the fracture toughness of the silicon nitride ceramics [27], thereby inhibiting particle flaking during the friction process. As discussed in Sects. 2.3 and 4.2, the crystal phases of Y₂Si₂O₇, YSiO₂N, and Y₄Si₂O₇N₂ inhibit the particle flaking during the friction process, which greatly inhibited the progress of the abrasive wear and optimize the wear resistance of silicon nitride ceramics. However, the residual thermal stress at the interface caused by the crystal phase Y₂Si₃O₃N₄ was

22–35% higher than that caused by the above three crystal phases. The interface between crystal phase Y₂Si₃O₃N₄ and Si₃N₄ matrix would be debonded due to the excessive residual thermal stress, intensifies the shedding of grains during wear, and reduces the wear performance of silicon nitride ceramics.

5 Conclusions

A variety of combinations of Y₂O₃ and Al₂O₃ were used as sintering aids in the fabrication of Si₃N₄ ceramics via gas pressure sintering (GPS). Based on the prediction of the residual thermal stress at the interface, the influence of crystal phases on the wear properties of Si₃N₄ ceramics was analyzed. The following conclusions were drawn:

1. The crystal phases, Y₂Si₂O₇, YSiO₂N, Y₄Si₂O₇N₂, and Y₂Si₃O₃N₄, were formed in silicon nitride ceramics during sintering. The interface residual thermal stress caused by the crystal phase Y₂Si₃O₃N₄ is 22–35% higher than that caused by the other three crystal phases.
2. The crystal phases of Y₂Si₂O₇, YSiO₂N, and Y₄Si₂O₇N₂ inhibit the particle flaking during the friction process, which greatly inhibit the progress of the abrasive wear and optimize the wear resistance of silicon nitride ceramics.
3. The interface between crystal phase Y₂Si₃O₃N₄ and Si₃N₄ matrix would be debonded due to the exces-

sive residual thermal stress, intensifies the shedding of grains during wear, and reduces the wear performance of silicon nitride ceramics.

Acknowledgements This work was supported by Science and Technology Major Project of Shanxi Province, China (20181102015), and Science and Technology Project of Taiyuan, Unveiled Project of Taiyuan.

Author contributions QW and CZ: wrote the main manuscript text; CY: analyzed the data; RH and YY: experiment.

Data and code availability The raw/processed data required to reproduce these findings cannot be shared at this time as the data also form part of an ongoing study.

Declarations

Conflict of interest There is no conflict of interest with others.

Ethics approval No human tissue or animal subjects were involved in this study.

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