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ARTICLE

Preparation of a Hydrolysis-Resistant Coating on AlN Powder Surface and Its Effect on Thermal Conductivity of AlN Ceramic

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Abstract: Improving the hydrolysis resistance of AlN ceramic powder is crucial to the powder storage and shaping processing. In order to improve the hydrolysis resistance of AlN powder, a hydrolysis-resistant coating was used to act as a barrier layer to prevent water from contacting with the AlN surface. An amorphous Y_2O_3 coating was uniformly deposited and fully wrapped on the surface of AlN powder via a chemical precipitation process. The coating effectiveness and integrity were investigated by TEM, XPS and Zeta potential measurements. The hydrolytic behavior of AlN powder in an aqueous suspension was studied by following the pH vs. time at room temperature. The results show that the coated AlN powder is stable in water within 48 h, indicating that the surface coating treatment with Y_2O_3 can effectively passivate AlN powder against hydrolysis. Moreover, as a way to introduce the sintering additives, the chemical precipitation process can favor an improvement of thermal conductivity of the sintered AlN ceramic, compared with the conventional ball-milling process.

Key words: AlN ceramic; chemical precipitation process; Y_2O_3 coating; hydrolysis; thermal conductivity

Aluminum nitride (AlN) ceramics are promising materials for ceramic substrates and heat sinks in the field of electronic packaging, because of their high thermal conductivity, high electrical resistivity, low dielectric constant, no toxicity, and a coefficient of thermal expansion close to that of silicon[1-3]. However, the hydrolytic reaction of AlN powder poses serious problems concerning the powder storage and shaping processing[4]. When AlN powder is hydrolyzed by water, the formation of undesirable aluminum hydroxides increases the oxygen content, which in turn, lowers the thermal conductivity of AlN ceramic[4,5]. To avoid the reactivity of AlN powder with water, non-aqueous shaping processing is required, or alternatively, hydrolysis resistant AlN powder is used for fabricating AlN ceramic products via aqueous shaping processing methods[6-8]. Due to economic and environmental concerns, the aqueous shaping processing of AlN powder becomes increasingly popular[9]. Therefore, many attempts have been made to passivate AlN powder against hydrolysis[10]. One of the most straightforward approaches is to modify AlN powder surface with organic compounds, such as oleic acid, 8-hydroxyquinoline, sebacic acid[8]. These organic compounds are characteristically hydrophobic and thus prevent water from contacting with the surface of AlN powder. But the hydrophobic ceramic powder cannot be properly dispersed in water without adding wetting agents, which can lead to the extensive foaming of the aqueous suspension[4,11]. In addition, AlN powder can be protected against hydrolysis by inorganic precursors that form impermeable silicate[12] and phosphate[11,13,14] complexes on its surface, where the former cannot provide as complete protection as the latter[10]. However, the use of acid solutions for treating AlN powder is not advisable from the environmental point of view[14], and the protective surface layer can be a trouble for improving the thermal conductivity of AlN ceramic, due to the rising oxygen content on the surface of AlN powder supplied by the protective phosphorous

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layer[11,15,16]. Moreover, the oxide layer (Al_2O_3) on AlN powder surface formed by the thermal oxidation treatment is also able to hinder the hydrolysis[17]. But it can lead to increased oxygen content and is detrimental to the improvement of thermal conductivity of AlN ceramic.

Besides the above methods to prevent hydrolysis reaction, there is an attempt to modify AlN powder surface via a protective layer of Y_2O_3 formed by the adsorption of $Y(NO_3)_3 \cdot 6H_2O$ on AlN surface[18]. The Y_2O_3 coating can not only act as a barrier layer, but also is conductive to a homogeneous distribution of sintering additive for AlN ceramic[19]. Unfortunately, the adsorption of rare-earth salt is not expected to be a completely effective approach to form a continuous and homogeneous coating on ceramic powder surface, which would deteriorate the hydrolysis resistance of AlN powder. As one of the promising powder coating routes, a chemical precipitation process has recently been widely employed to homogeneously coat Al_2O_3 and SiC powder[20,21]. Considering the hydration of AlN powder in an aqueous media, a non-aqueous chemical precipitation method has been used to coat AlN powder[22], which can provide a technical viewpoint to coat AlN powder by a rare-earth oxide layer for improving its anti-hydrolysis property. In the present work, we attempted to prepare the Y_2O_3 coated AlN composite powder by a non-aqueous chemical precipitation process to prevent hydrolysis of AlN powder. The as-received and as-treated AlN powder were suspended in water for 48 h to evaluate their hydrolysis resistance by monitoring the pH-time profiles of the suspensions. The coating effectiveness and integrity were investigated in detail by TEM, XPS and Zeta potential measurements. Further, to illustrate the superiority of the coating treatment in the fabrication of AlN ceramic, the thermal conductivity and microstructure comparison between the chemical precipitation and ball-milling processed AlN ceramics were studied.

1 Experiment

1.1 Surface coating of AlN powder

Y_2O_3 coating was deposited on AlN powder by a chemical precipitation process under an anhydrous condition. The commercial AlN powder (D_{50}=1.15 μm, Xiamen Juci Technology Co., Ltd, China) was used as starting materials. The $Y(NO_3)_3 \cdot 6H_2O$ (99.99% purity, Shanghai Aladdin Bio-Chem Tech-nology Co., Ltd, China) was calcined at 260 °C for 30 min to remove the crystal water. The anhydrous $Y(NO_3)_3$ was then dissolved in ethanol to prepare precursor solution, where the Y^{3+} concentration was accurately controlled to 0.05 mol/L. Afterwards, 19 g AlN powder was slowly added in 176 mL precursor solution, and mixed with 0.3 g polyethylene glycol (PEG 2000). According to the calculation based on the transformation of $Y(NO_3)_3$ to Y_2O_3, the mass ratio of the Y_2O_3 to AlN was 5: 95. The above suspension was dispersed in a bath under ultrasound for 60 min, and then vigorously stirred for 120 min to prevent the sedimentation of ceramic particles. The mixed solution (pH=12.66) of ethylenediamine (EDA, Shanghai Macklin Biochemical Co., Ltd, China) and ethanol with the mass ratio of 1: 2 was used as the non-aqueous precipitant solution, and slowly dripped into the ceramic suspension under strong mechanical stirring to tailor the pH value of the suspension in the range of 9.3~9.5. During the dripping process of the non-aqueous precipitant solution, the precipitations of yttrium-amine coordination compound were formed and simultaneously deposited on the surface of the AlN powder. Finally, the as-prepared composite ceramic powder (AlN coated with the yttrium-amine coordination compounds) was washed with ethanol twice and air-dried at 60 °C for 10 h. The dried composite powder was then calcined at 500 °C for 2 h in air to decompose yttrium-amine coordination compounds into Y_2O_3.

1.2 Fabrication of sintered AlN ceramic

The above coated powder was further mixed with 4.5wt% paraffin and uniaxially pressed in a steel die under 100 MPa for 60 s, followed by cold isostatic pressing under 200 MPa for 5 min. After the molding and debonding process, the debond samples were placed into a BN crucible and sintered at 1850 °C for 4 h in a controlled atmosphere furnace (ZT-90-22, Shanghai Chenhua Technology Co., Ltd, China) with a nitrogen-gas flow rate of 0.5 L/min. The sintered AlN samples via the chemical precipitation process were referred to as sample CP.

For comparison, 95wt% AlN and 5wt% Y_2O_3 powders (99.99% purity, D_{50}=500 nm, Shanghai Macklin Biochemical Co., Ltd, China) were ball milled in ethanol for 4 h employing a planetary ball mill (QM-QX4, Nanjing NanDa Instrument Plant, China), with the milling speed of 250 r/min. The ball-milling processed AlN ceramic was fabricated via the same molding, debonding and sintering procedures as sample CP. The sintered AlN samples via the ball-milling process were referred to as sample BM.

1.3 Characterization

The hydrolysis behavior of as-received and as-treated AlN powders was studied by dispersing the powders in water to obtain aqueous suspensions with a solid loading of 20wt% and registering the pH evolution at room temperature for 48 h by a pH meter (PHS-3G, INESA Scientific Instrument Co., Ltd, China). The microstructure of the as-treated AlN powder was investigated by a transmission electron microscopy (TEM, Talos F200S, Thermo Fisher Scientific Inc., America) coupled with an energy dispersive spectroscope (EDS). The samples for TEM analysis were prepared by dispersing the as-treated AlN powder in ethanol and applying the prepared suspension onto a copper grid. An X-ray photoelectron spectroscopy (XPS, Escalab 250Xi, Thermo Fisher Scientific Inc., America) was used to study the surface chemistry of the as-treated AlN powder.

To testify the coating effectiveness, the as-received and as-treated AlN powders were separately suspended in ethanol to avoid the effect of any further hydrolysis on the electrophoretic behavior of AlN powder. The Zeta potentials of the AlN suspensions were measured by a Zeta potential analyzer (Zetasizer Nano ZS, Malvern Panalytical Co., Ltd, England). The pH of the slurries was adjusted with HCl or NaOH solutions with a concentration of 0.1 mol/L and the solid loading of the slurries was kept as 0.06vol%. It should be pointed out that in the ethanol medium, the operational pH value (O.pH) differs from the actual pH and their relation can be expressed by the following equation[23,24]:

$$\text{O.pH}=\text{pH}-2.91 \tag{1}$$

The density of the sintered AlN ceramic samples was measured by Archimedes method using an analytical balance with an accuracy of 0.0001 g. The thermal conductivity at room temperature was measured by the laser flash method using a laser thermal conductivity instrument (LFA 447, Netzsch Instruments Co., Ltd, Germany). The microstructures of the fracture surface of the sintered ceramics were characterized by a scanning electron microscopy (SEM, LYRA 3 XMU, Tescan, Czech).

2 Results and Discussion

2.1 Surface characterization of AlN powder

A HAADF (High-Angle Annular Dark Field)-TEM image and EDS map for Y of as-treated AlN particles are shown in Fig. 1a and 1b, respectively. It can be noted that Y is mainly encapsulated in the shell of the as-treated particle, indicating the integrality of Y-containing shell. A high magnification TEM image of the as-treated AlN particle is shown in Fig. 1c, which displays that the particle is coated by a layer of amorphous deposition with an average thickness of ~2.63 nm. To identify the composition of the amorphous layer, the EDS analysis was conducted on both amorphous layer (shell) and the bulk of the AlN particle (core) for comparison, as shown in Fig. 1d and 1e, respectively. The impurity peaks for Cu are introduced by the copper mesh used in the TEM test. Obvious Y and O peaks can be detected in the amorphous shell, indicating the existence of Y-O compound in the shell. Meanwhile, only some weak Y and O peaks can be detected in the core. The TEM and EDS results suggest that the AlN particles are encapsulated by the amorphous layer of Y-O compound.

To examine the elements and present their chemical states on the surface of the as-treated AlN powder, the XPS measurements were carried out, and the XPS survey scan is shown in Fig. 2. The value corresponding to C1s peak (284.78 eV) was used as a reference for XPS spectra analysis. The as-received AlN surface shows three elements (Al, N and O), and the as-treated AlN surface shows four elements (Al, N, O and Y). It should be noted that the O on the AlN surface mainly arises from the unavoidable oxidation of the raw AlN powder at room temperature[25,26]. The detection depth (5~10 nm) of XPS for inorganic materials is higher than the thickness (2.63 nm) of Y_2O_3 shell, so N can be detected in the as-treated powder by XPS. The binding energy of 157.49 eV for $Y3d_{5/2}$ is presented, and this binding energy is in accordance with the documented values (158.1, 158.2 and 157.4 eV) of $Y3d_{5/2}$ of Y_2O_3[27-29]. Thus, the $Y3d_{5/2}$ peak can be assigned to Y^{3+} in Y_2O_3. The quantitative calculations from the narrow scan results of Al2p, O1s, N1s, C1s and Y3d were performed to

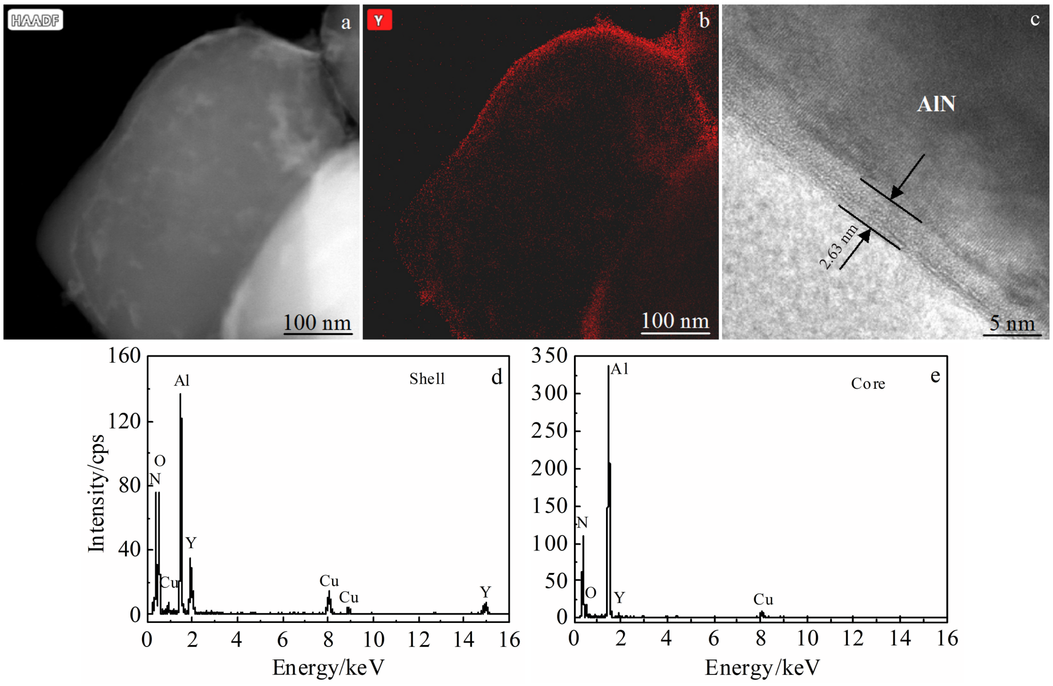

Fig.1 HAADF-TEM image (a) and EDS map (b) for Y element of an as-treated AlN particle; high magnification TEM image of the as-treated AlN powder (c); EDS spectra recorded from the shell (d) and core (e) of the as-treated AlN powder

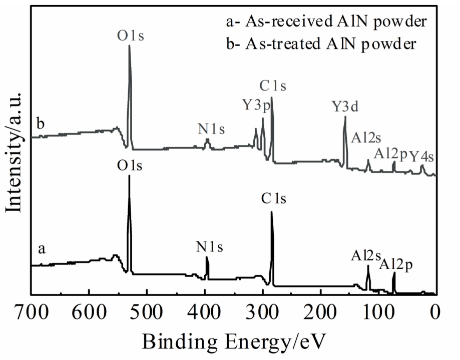

Fig.2 XPS survey scan of as-received and as-treated AlN powders

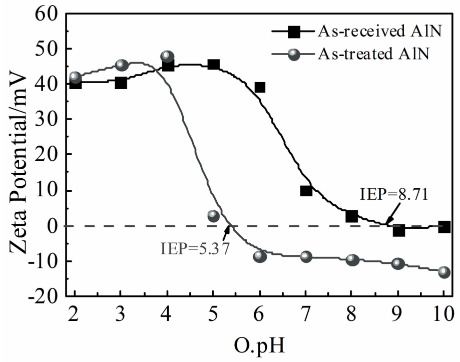

Fig.3 Zeta potentials of the as-received and as-treated AlN powders as a function of O.pH

determine the chemical compositions of the as-received and as-treated AlN powder surface, as shown in Table 1. The surface of the as-received AlN powder contains 26.99at% O, indicating that the oxidized layer on the AlN particle surface forms due to the unavoidable partial oxidization of the raw AlN powder at room temperature. In addition, the Y/N atomic ratio of the as-treated (Y_2O_3-coated) AlN powder is calculated to be 0.77, which is much greater than the Y/N atomic ratio (0.019) of the stoichiometric mixture (95wt% AlN+5wt% Y_2O_3). The much higher values of the Y/N atomic ratio values in the as-treated AlN powder can evidence that the surface of the as-treated AlN powder is enriched with Y, i. e., Y is localized in the surface layer of the as-treated AlN powder. It can be concluded from the TEM and XPS results that the surface of the AlN ceramic powder is thoroughly covered with an amorphous Y_2O_3 layer prepared by the chemical precipitation process.

Zeta potential can show the surface electron charge of the powder surface. The Zeta potentials of the as-received AlN and the as-treated AlN powders were measured as a function of O.pH, as shown in Fig.3. It can be seen that the as-received AlN powder exhibits a Zeta potential value of 45.5 mV in the ethanol solution with the O.pH of 4.0. With the increase of the O.pH in the non-aqueous system, the absolute value of Zeta potential decreases, and becomes negative at the O.pH of 9.0. The Zeta potential of the as-received AlN powder is measured to be just -0.1 mV at an O.pH of 10.0. This low absolute value is consistent with the poor dispersive stability of the system, indicating that the obvious sedimentation of the AlN particles occurs just after being left to stand for 10 min. Meanwhile, the Zeta potential of the as-treated AlN powder is measured to be 48.0 mV in the ethanol solution with the O. pH of 4.0. Similarly, the absolute value of Zeta potential decreases with the increased O.pH, and becomes negative at the O.pH of 6.0. The as-received and as-treated AlN powders show the isoelectric point (IEP) at 8.71 and 5.37, respectively. The significant difference in the IEP is attributed to the alteration of the surface chemical state of the ceramic particles. The surface of the as-received AlN powder is terminated with Al-OH, which is generated from the surface oxidation layer of Al_2O_3, due to the fact that the AlN is partially oxidized at room temperature[25,26]. While the surface of the as-treated AlN powder is terminated with Y-OH, which is generated from the surface hydroxylation reaction of Y_2O_3 shell. The distinct change in the Zeta potentials between the as-received and as-treated AlN powders also clearly testify that the AlN powder is successfully coated with Y_2O_3 by the chemical precipitation process. The Y_2O_3 coating on AlN powder can act as a barrier layer to prevent water from coming into contact with the AlN surface, which can protect AlN powder against hydrolysis. In addition, Y_2O_3 is intrinsically a hydrophilic material[30], so it can favor the preparation of hydrophilic AlN powder to facilitate its dispersion in water[10], which is suitable for aqueous powder shaping processing of AlN powder.

Table 1 Chemical compositions of as-received and as-treated AlN powders (at%)

AlN powder	C1s	O1s	N1s	Al2p	Y3d
As-received	45.12	26.99	8.44	19.45	0
As-treated	42.85	32.44	8.20	10.18	6.33

2.2 Hydrolysis resistance of the as-treated AlN powder

To evaluate the effects of AlN powder surface coating on hydrolysis reactions of AlN, the hydrolysis behavior of the as-received and as-treated AlN powders at room temperature was studied. Changes in the pH values for aqueous AlN suspensions as a function of time are shown in Fig. 4. The hydrolysis process of AlN powder in aqueous solution will result in the increase of the suspension's pH due to the following thermodynamically favorable reactions [5, 14, 31]:

$$AlN + 2H_2O \rightarrow AlOOH_{(amorph)} + NH_3 \tag{2}$$

$$AlOOH_{(amorph)} + H_2O \rightarrow Al(OH)_{3(XSTAL)} \tag{3}$$

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \tag{4}$$

The suspension's pH of the as-received AlN powder drastically increases from 8.74 to 12.25 just within 8 h, indica-ting a significant hydrolysis of the as-received AlN powder; and after 8 h, the pH slowly increases due to the formation of aluminum trihydroxide phase ($Al(OH)_{3(XSTAL)}$)[32,33]. In contrast, the as-treated AlN powder is stable in water as there is no significant change in the pH of the aqueous suspension of the

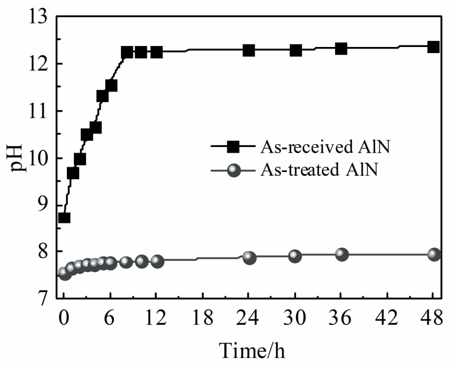

Fig.4 pH vs. time for the aqueous suspensions of as-received and as-treated AlN powders

as-treated AlN powder even after a period of 48 h. The nonreactivity of the as-treated AlN powder confirms the effectiveness of surface coating treatment in protecting AlN powder against hydrolysis.

2.3 Thermal conductivity and microstructure of sintered AlN ceramics

Table 2 lists the relative density and thermal conductivity of sample BM and sample CP. The relative density of sample CP is a little higher than that of sample BM. Compared with conventional ball-milling process, the chemical precipitation process can show better homogeneous mixing for sintering additives (Y_2O_3)[22], which is favorable to form homogeneous liquid-phase yttrium aluminates[34] to promote the sintering densification of AlN ceramic at high temperatures. The thermal conductivity of sample CP is 8.17% higher than that of sample BM. Fig.5 presents the SEM images of the fracture surfaces of sample BM and sample CP. Samples BM (Fig.5a) has a larger number of pores than sample CP (Fig.5b), which is consistent with the higher relative density of the sample CP compared to that of sample BM. The pores can cause phonon scattering[35] and thus lower the thermal conductivity of sample BM. The test results in Table 2 and Fig.5 indicate that the chemical precipitation process is an effective way to fabricate AlN ceramics with enhanced thermal conductivity.

Table 2 Properties of the sintered AlN ceramics fabricated by the ball-milled and coated AlN powders

Sample	Relative density/%	Thermal diffusivity/mm^2·s	Thermal conductivity/ W·m^{-1}·K^{-1}
BM	99.7	61.72	149.86
CP	100	66.50	162.10

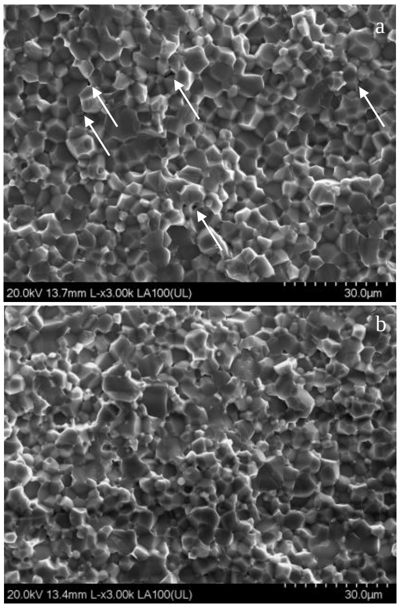

Fig.5 SEM images of fracture surface of sample BM (a) and sample CP (b) (pores are marked with arrows)

3 Conclusions

1) A chemical precipitation process was used to fabricate Y_2O_3-coated AlN powder with a core-shell structure under an anhydrous condition, in order to improve the hydrolysis resistance of AlN powder. The Zeta potential, TEM and XPS experimental results demonstrate that a continuous layer of amorphous Y_2O_3 can be fully wrapped on the surface of AlN powder.

2) The Y_2O_3-coated AlN powder is stable in water within 48 h, confirming that the Y_2O_3 coating is able to act as a H_2O diffusion barrier layer from the media to the surface of AlN powder, protecting AlN powder against hydrolysis.

3) The thermal conductivity of sample CP is 8.17% higher than that of sample BM. The improved thermal conductivity of the chemical precipitation processed AlN ceramic is attributed to the denser microstructure compared with the ball-milling processed sample.

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AlN粉体表面抗水解涂层的制备及其对AlN陶瓷热导率的影响

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摘　要：提升AlN陶瓷粉体的抗水解性能对于其储存和成型加工至关重要。使用一种抗水解涂层作为阻止水分与AlN表面接触的屏障，以提升AlN粉体的抗水解性能。采用化学沉淀工艺在AlN粉体表面制备均匀、全包覆的非晶Y_2O_3涂层。利用TEM、XPS和Zeta电位测试详细研究了包覆层的有效性和完整性。通过测试室温下水基AlN悬浮液的pH-时间曲线以研究AlN粉体的水解性能。结果表明，经包覆处理的AlN粉体能够在水中保持稳定至48 h，这说明Y_2O_3表面包覆处理可以有效钝化AlN粉体，从而避免了其水解反应的发生。此外，与球磨工艺引入烧结助剂相比，化学沉淀工艺有利于提升AlN陶瓷的热导率。

关键词：AlN陶瓷；化学沉淀工艺；Y_2O_3涂层；水解；热导率

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