

Composition-Dependent Phase Evolution of Reactive-Sintered AlON

Shuixian Yang^{1, 2}, Xiaojian Mao^{*3}, Run Tian³, Jian Zhang³, Shiwei Wang³

¹National Demonstration Center For Experimental Physics
Education, Nanjing University, Nanjing 210093, China.

²School of Physics, Nanjing University, Nanjing 210093, China

³State Key Laboratory of High Performance Ceramics, Shanghai Institute
of Ceramics, Chinese Academy of Sciences, Shanghai 201899, China

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Abstract

Based on an investigation of the reactive sintering behavior of Al₂O₃-AlN up to 1950 °C, a comprehensive framework is established for understanding the multi-stage reactive sintering process of AlON ceramics. The process comprises four distinct stages: (1) initial densification dominated by Al₂O₃ sintering, during which preferential Al₂O₃ grain growth delays AlON formation; (2) formation of a nitrogen-rich AlON solid solution, accompanied by lattice and volumetric expansion; (3) dissolution of residual Al₂O₃ into the AlON lattice, together with lattice contraction and volumetric shrinkage; (4) final densification of phase-pure AlON. This mechanism is consistently verified across a broad composition range of 22–40 mol% AlN, offering critical insights for process optimization. Furthermore, blackening of the sintered sample is identified as an inevitable intermediate stage during the densification of AlON ceramics, which is unrelated to phase impurities or carbon contamination.

Keywords: Aluminum oxynitride (AlON), reactive sintering behavior, blackening

I. Introduction

Transparent aluminum oxynitride (AlON) ceramic is one of the most promising advanced ceramic materials thanks to its high strength and hardness, excellent chemical resistance, and broad optical transmission from the ultraviolet to mid-infrared region. These properties enable its widespread use in infrared windows, missile domes, transparent armor, and other applications requiring extremely durable optical components^{1–4}.

AlON, defined as a nitrogen-stabilized cubic aluminum oxide⁵, is a single-phase solid solution in the Al₂O₃-AlN binary system. The stability region of single-phase AlON has been reported to range from approximately 27 to 40 mol% AlN at temperatures between 1700–2170 °C^{4,6}. Two major fabrication routes have been extensively used to produce transparent AlON ceramics: sintering of pre-synthesized AlON powders, and reactive sintering of Al₂O₃-AlN powder mixtures^{7–10}. For the first method, achieving high-purity and compositionally uniform AlON powder is crucial. The preparation of AlON powder based on the controlled carbon reduction of Al₂O₃ in N₂ was first reported in 1959 by Yamaguchi and Yanagida⁵. This method, now known as the carbothermal reduction nitridation (CRN) method, is currently the most common approach for synthesizing AlON powder on account of the low cost

of raw materials and the high quality of the final product. However, obtaining phase-pure AlON powder with stable stoichiometry by means of CRN is challenging and time-consuming because this process is highly sensitive to both raw materials and synthesis conditions^{7, 11, 12}.

By contrast, direct reactive sintering of Al₂O₃ and AlN offers a much simpler and more economical route. The first translucent AlON ceramic disk prepared by means of reactive sintering was reported in 1979⁹. However, it soon became apparent that achieving dense AlON ceramics with high optical quality with this method was difficult¹³. Therefore, subsequent efforts have focused on the role of sintering additives in improving the densification and transparency of AlON ceramics. For example, Cheng *et al.*¹⁴ fabricated a 0.6-mm-thick AlON ceramic structure with a total transmittance of 60 % using Y₂O₃ as a sintering additive. Lu *et al.*¹⁵ reported an improved transmittance of 73.2 % at 1600 nm for 1-mm-thick samples doped with MgO and Y₂O₃, while Qin *et al.*¹⁶ further achieved a transmittance of 82.5 % at 600 nm for 2-mm-thick ceramics via gas-pressure reactive sintering using Y₂O₃ and La₂O₃ as co-additives. Although these studies demonstrated improved transmittance based on additive optimization, the achieved transmittance remains below expectations. This limitation likely stems from an incomplete fundamental understanding of the reactive sintering behavior between Al₂O₃ and AlN under realistic processing conditions.

* Corresponding author: maoxiaojian@mail.sic.ac.cn

Bandyopadhyay *et al.*¹⁷ examined the effect of reaction parameters on the formation of γ -AlON from Al_2O_3 and AlN under isothermal conditions, revealing that an oxygen-rich γ -AlON forms initially and subsequently reacts with AlN. However, the long isothermal holds (2.5 h at each temperature) used in this study do not reflect the continuous heating process typically employed in practical sintering of AlON transparent ceramics, where samples are heated to high temperatures at a fixed rate, followed by a prolonged dwell for pore elimination and densification. Similarly, another study using Y_2O_3 as an additive demonstrated that density and phase composition are highly sensitive to the AlN/ Al_2O_3 ratio and temperature¹⁸. Nevertheless, a comprehensive analysis of the reaction pathways and densification mechanisms during the reactive sintering of AlN- Al_2O_3 is still lacking. For example, the phase transformation sequence of Al_2O_3 and AlN during sintering remains unclear even for a given composition. A key question is whether Al_2O_3 undergoes preferential self-sintering prior to or concurrently during reaction with AlN, especially since Al_2O_3 densifies below 1640 °C, whereas AlON formation is reported only above this temperature. Moreover, the densification behavior of AlON itself requires further clarification. Consequently, it is essential to gain a comprehensive understanding of phase transformations and microstructural evolution during reactive sintering as it underpins the development of fully dense AlON ceramics with superior optical properties.

In this study, Al_2O_3 -AlN powder mixtures spanning a broad composition range (22–45 mol% AlN) were sintered at various temperatures and dwell times. The phase transformation and microstructural evolution of the Al_2O_3 -AlN sample were systematically examined. By elucidating these mechanisms, this work aims to provide critical insights that can guide the fabrication of high-quality AlON ceramics via reactive sintering.

II. Experimental

Commercial high-purity Al_2O_3 powder (purity > 99.99 %, $D_{50} = 0.2 \mu\text{m}$, Baikowski) and high-purity AlN powder (purity > 99.99 %, $D_{50} = 2 \mu\text{m}$, H.C. Starck) were used as the starting materials. Appropriate mixtures of these powders were dispersed in ethanol and ball-milled in a planetary mill at 250 rpm for 20 h using alumina grinding balls. After drying and sieving, the mixed powders were uniaxially dry-pressed into discs (20 mm in diameter, 6 mm in thickness) and subsequently cold-isostatically pressed at 200 MPa. The compacted pellets were placed in BN crucibles, covered with BN lids, and sintered in a graphite resistance furnace under nitrogen flow.

To investigate the sintering behavior, green bodies containing 30 mol% AlN and 70 mol% Al_2O_3 were heated at a rate of 5 K/min to temperatures ranging from 1400 to 1950 °C, followed by holding times of 0–30 h. Dilatometric curves were obtained by measuring the radial dimensions of the sample before and after sintering at each temperature. The phase composition of the sintered samples was explored by means of X-ray diffraction (XRD; Bruker D8, Germany) using Cu K_α radiation

($\lambda = 0.154 \text{ nm}$) in the 2θ range of 0–120 °C. The lattice parameters of the sintered samples were determined by plotting the lattice constants calculated from reflections in the range of 50–120 °C against the Nelson-Riley function $f(\theta)$. The fracture microstructures of the sintered ceramics were examined using scanning electron microscopy (SEM; TM-3000, Hitachi, Japan) equipped with a backscattered electron detector after the samples had been sputter-coated with gold.

III. Results and Discussion

Fig. 1 shows the XRD patterns of the samples containing 30 mol% AlN and 70 mol% Al_2O_3 after sintering at various temperatures. When the temperature is below 1650 °C, only Al_2O_3 and AlN phases are detected, with no indication of AlON formation. At 1700 °C, diffraction peaks corresponding to AlON phase appear, though residual Al_2O_3 and AlN are still present. When the temperature reaches 1800 °C, the AlN phase disappears completely, leaving only AlON together with unreacted Al_2O_3 . At temperatures above 1850 °C, only AlON is observed.

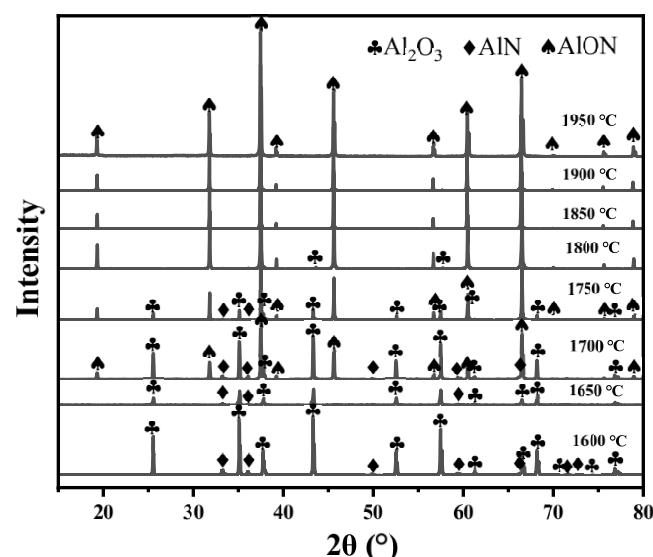


Fig. 1: XRD patterns of 30 mol% AlN – 70 mol% Al_2O_3 samples sintered at different temperatures without holding.

The absence of AlON below 1650 °C, despite being close to the frequently reported formation temperature of ~1640 °C¹⁹, is likely attributable to the insufficient reaction kinetics in this study, such as the relatively low reactivity of the raw powders and the lack of an isothermal hold at 1650 °C. The preferential consumption of AlN above 1800 °C, leading to a two-phase mixture of AlON and Al_2O_3 , suggests the initial formation of a nitrogen-rich AlON solid solution. At temperatures exceeding 1850 °C, the remaining Al_2O_3 gradually dissolves into the AlON lattice, indicating that the inter-diffusion process is strongly temperature-dependent. This ultimately results in the formation of a homogeneous, single-phase AlON.

The corresponding variation in the lattice parameter of AlON (nominal composition: 30 mol% AlN – 70 mol% Al_2O_3) sintered between 1750 °C and 1950 °C is shown in Fig. 2. It can be seen that the lattice parameter gradually increases with increasing temperature, reaches

a maximum at 1850 °C, and then decreases upon further heating. The initial lattice expansion is ascribed to the increased nitrogen content in the AlON solid solution, which is consistent with the well-established trend that the AlON lattice parameter increases with higher AlN content^{9,20–21}. This behavior aligns with the reaction mechanism revealed by XRD analysis, wherein a nitrogen-rich AlON intermediate forms prior to the development of stoichiometric AlON.

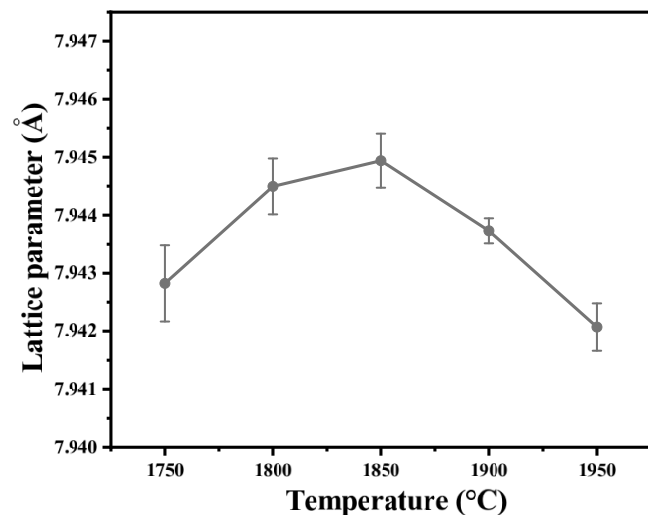


Fig. 2: Temperature dependence of the lattice parameter for the 30 mol% AlN – 70 mol% Al₂O₃ sample.

However, a key observation is that the maximum lattice parameter appears at 1850 °C, which is significantly higher than the temperature (~1800 °C) at which AlN is fully consumed, as indicated by XRD analysis. This discrepancy suggests that although the chemical reactions required to form the most nitrogen-rich phase are completed by 1800 °C, the subsequent processes of homogeneous nitrogen distribution and full lattice relaxation require additional thermal energy and time. These diffusion-controlled processes reach completion only at 1850 °C. This phenomenon reflects a well-established principle in solid-state chemistry: diffusion-driven compositional homogenization typically lags behind the initial phase transformation²². The subsequent lattice contraction observed above 1850 °C results from the diffusion of Al₂O₃ into the AlON lattice, which drives the composition toward stoichiometric equilibrium. This process increases the oxygen-to-nitrogen ratio and, according to the established defect chemistry model of AlON ($\text{Al}_{(64+x)/3}\text{V}_{(8-4x)/3}\text{O}_{32-x}\text{N}_x$)²³, reduces the concentration of cation vacancies to maintain charge neutrality. As the material approaches stoichiometric composition, the reduction in vacancy concentration inevitably leads to denser atomic packing and thus a decrease in the lattice parameter.

However, this result significantly differs from a previous report¹⁷, which suggested that an oxygen-rich AlON solid solution is formed prior to the nominal composition. This discrepancy is likely attributed to the 2.5 h dwell time used in that study at temperatures between 1560 °C and 1850 °C – an approach method not employed in our continuous-heating sintering process.

To further validate our findings, compositions ranging from 22 to 45 mol% AlN (with corresponding Al₂O₃) were sintered at 1750–1950 °C without holding. The detailed phase evolution behavior, as determined by means of a systematic analysis of the corresponding XRD patterns, is summarized in Fig. 3. For the samples containing 22 mol% and 25 mol% AlN, a mixture of AlON and unreacted Al₂O₃ is observed after sintering at 1750 °C and 1800 °C. For the 22 mol% AlN sample sintered above 1850 °C, the XRD pattern closely resembles that of cubic AlON but exhibits distinct shoulder peaks at 2θ of approximately 33° and 34°. Comparison with existing literature indicates that these peaks may correspond to the Φ-AlON phase^{24–25}, although definitive identification requires further analysis.

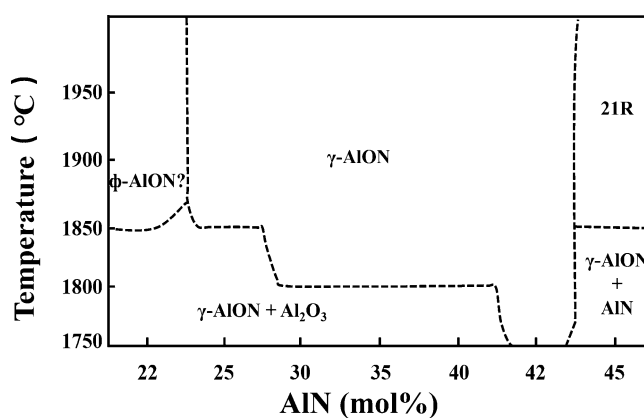


Fig. 3: Phase evolution of samples containing 22–45 mol% AlN (with corresponding Al₂O₃) sintered between 1750 and 1950 °C.

For the 25 mol% AlN sample, all residual Al₂O₃ was consumed when the temperature exceeded 1850 °C, yielding a single-phase AlON ceramic. For the sample containing 30 mol% AlN, a mixture of AlON and Al₂O₃ is observed at 1750 °C. At 1800 °C, the AlON phase becomes dominant, with only a trace amount of residual Al₂O₃ (as shown in Fig. 1). Finally, a fully single-phase AlON structure is obtained above 1850 °C. A similar evolution pathway is observed for the 35 and 40 mol% AlN samples: both exhibit coexistence of AlON and Al₂O₃ at 1750 °C and transform to pure AlON at temperatures above 1800 °C. Thus, the phase evolution behavior is consistent across the composition range of 30–40 mol% AlN.

For the sample containing 42 mol% AlN, sintering at 1750 °C was sufficient to complete the reaction between Al₂O₃ and AlN, yielding AlON as the sole crystalline phase. By contrast, for the 45 mol% AlN sample, AlON coexists with unreacted AlN at 1750 °C, and the 21R(Al₇O₃N₅)^{24,26} phase is detected when the sintering temperature exceeds 1850 °C.

Overall, these results demonstrate that for nominal compositions containing 25–40 mol% AlN, the phase evolution follows a consistent sequential pathway: (1) formation of a nitrogen-rich AlON solid solution via reaction between AlN and a portion of the available Al₂O₃; (2) dissolution of the remaining Al₂O₃ into the nitrogen-rich AlON solid solution as temperature

increases; (3) convergence toward the nominal composition with further increase in the temperature, ultimately yielding single-phase ALON at sufficiently high temperatures.

Fig. 4 shows the backscattered electron (BSE) micrographs of the fracture structure for the 30 mol% AlN – 70 mol% Al₂O₃ samples sintered at various temperatures. As shown in Fig. 4(a)–(c), clear grain coarsening of both Al₂O₃ and AlN occurs as the sintering temperature increases from 1 400 to 1 650 °C. The larger and brighter grains observed in the samples sintered at 1 600 and 1 650 °C (indicated by arrows in Figs. 4(b) and 4(c)) can be attributed to Al₂O₃, based on the atomic number contrast in BSE imaging together with XRD confirmation and sintering kinetics. This indicates that Al₂O₃ undergoes more rapid grain growth than AlN in this temperature range, which is reasonable because Al₂O₃ usually begins to sinter below 1 600 °C, while AlN typically requires temperatures near 1 800 °C to initiate significant sintering²⁷.

The pronounced growth of Al₂O₃ grains reduces their specific surface area and decreases the interfacial contact with AlN adjacent particles. This reduced interfacial area

slows the solid-state reaction kinetics between Al₂O₃ and AlN required for ALON formation. Thus, the onset temperature of ALON phase formation (~1 700 °C) observed in this study is higher than the theoretical value, consistent with the XRD results shown in Fig. 1.

At 1 700 °C (Fig. 4(d)), the microstructure becomes more homogeneous, exhibiting improved grain-size uniformity and more regular morphology. According to the corresponding XRD results, this change can be ascribed to the initial formation of the ALON phase, which promotes grain coarsening and rearrangement. This is likely because the formation of the new ALON phase introduces a transient interfacial energy and enhances atomic diffusion at the reaction fronts, providing a driving force for grain boundary migration and particle rearrangement. As the sintering temperature continues to rise, the progressive formation of ALON stimulates grain growth, resulting in more regular morphology and stronger intergranular bonding.

Once the ALON phase is fully formed at 1 800 °C, rapid grain coarsening and a substantial reduction in porosity occur at temperatures above 1 850 °C (Fig. 4(g–i)). These

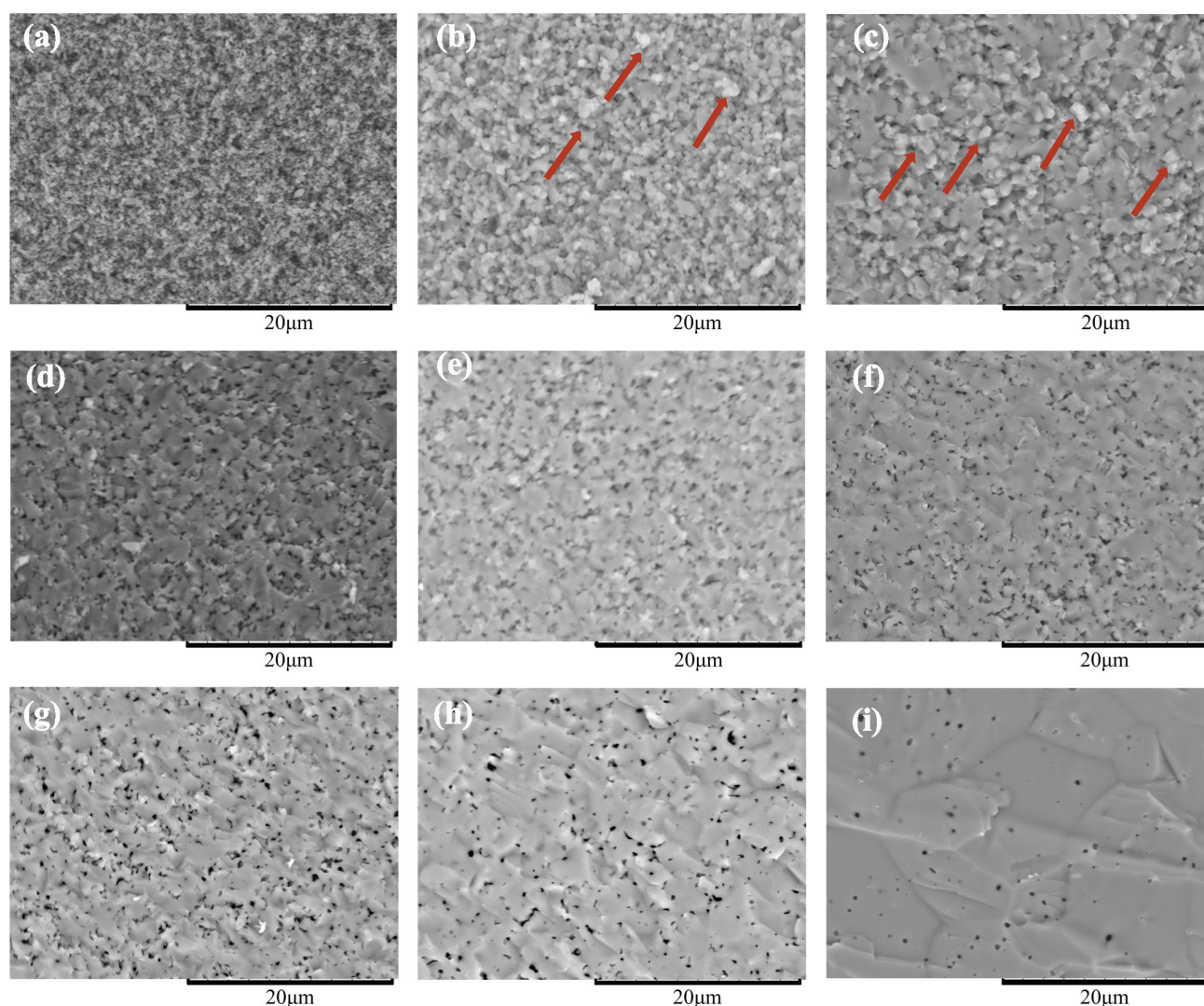


Fig. 4: Microstructures of the samples with 30 mol% AlN – 70 mol% Al₂O₃ heated at 5 K/min to different temperatures without holding: (a) 1 400 °C, (b) 1 600 °C, (c) 1 650 °C, (d) 1 700 °C, (e) 1 750 °C, (f) 1 800 °C, (g) 1 850 °C, (h) 1 900 °C, (i) 1 950 °C.

observations indicate that the densification of sintered ALON ceramics accelerates significantly after complete phase formation. However, numerous entrapped pores, with an average diameter of around $1\ \mu\text{m}$, remain within grains or along grain boundaries in the sample sintered at $1950\ ^\circ\text{C}$ (Fig. 4 (i)). These residual pores critically limit optical transmittance. Therefore, optimizing the sintering scheme and incorporating appropriate sintering additives are essential for eliminating pores and achieving high-transparency ALON ceramics synthesized via reactive sintering of Al_2O_3 and AlN .

To elucidate the sintering trajectory of the 30 mol% AlN – 70 mol% Al_2O_3 sample, the shrinkage curve was constructed from the radial length measurements taken before and after sintering at various temperatures, along with the corresponding bulk density trend. Both are shown in Figs. 5(a) and (b), respectively. As the temperature rises to $1650\ ^\circ\text{C}$, the sample exhibits continuous shrinkage, accompanied by a steady increase in density. However, a distinct expansion regime (labeled as Region I) appears between 1650 and $1750\ ^\circ\text{C}$, during which the bulk density decreases significantly. Correspondingly, the density reaches a minimum at $1750\ ^\circ\text{C}$ coinciding with the peak of expansion. Beyond $1800\ ^\circ\text{C}$, linear shrinkage resumes and is accompanied by a monotonic increase in density until the highest sintering temperature is reached. The initial shrinkage is attributed to the densification of Al_2O_3 , as confirmed by XRD results (Fig. 1), which indicate that no reaction between Al_2O_3 and AlN occurs within this temperature range. This interpretation is further supported by microstructural observations (Fig. 4(a-c)), which reveal faster grain growth of Al_2O_3 compared to AlN .

The subsequent expansion observed in Region I coincides with the onset of the ALON phase formation (Fig. 1). This strong correlation is consistent with a previous report¹⁸, which attributed similar dilatometric expansion to the formation of ALON. More importantly, this reaction-induced volumetric expansion is the

primary reason for the concomitant decrease in bulk density observed in Fig. 5(b). The formation of ALON from Al_2O_3 and AlN typically involves a positive molar volume change, wherein the product phase exhibits a larger volume than the reactant assemblage. This intrinsic expansion effect directly leads to both macroscopic expansion (Fig. 5(a)) and a reduction in bulk density (Fig. 5(b)), under the condition of constant mass. A comparable expansion phenomenon observed during the reactive sintering of magnesium aluminate spinel (MgAl_2O_4)²⁸ further supports the interpretation that such volumetric swelling is characteristic of transformation-induced reactions in mixed oxide-nitride systems.

The expansion reaches its maximum (and corresponding density minimum) at $1750\ ^\circ\text{C}$, which corresponds to the stage where AlN is nearly consumed and a nitrogen-rich ALON solid solution forms, as indicated by the XRD patterns (Fig. 1). Although the phase evolution progresses toward stoichiometric ALON with the continued incorporation of residual Al_2O_3 up to approximately $1800\ ^\circ\text{C}$, the macroscopic dimensional change transitions from expansion back to shrinkage, and the density begins to increase. This critical reversal suggests that, within the 1750 – $1800\ ^\circ\text{C}$ range, the driving force for sintering and pore elimination surpasses the internal expansion force associated with the ongoing phase transformation. The increased thermal energy available above $1750\ ^\circ\text{C}$ accelerates pore removal, enabling densification and producing the final shrinkage observed in the dilatometric curve.

Generally, high temperatures (e.g. $1950\ ^\circ\text{C}$) and long holding times (typically exceeding 20 h) are required to fabricate ALON transparent ceramics due to their strong covalent bonding and high melting point. To investigate the effect of holding time on the optical appearance and microstructure, samples containing 30 mol% AlN –70 mol% Al_2O_3 were reactively sintered at $1950\ ^\circ\text{C}$ for varying holding times. The resulting optical images and fracture microstructures are presented in Fig. 6 and Fig. 7, respectively.

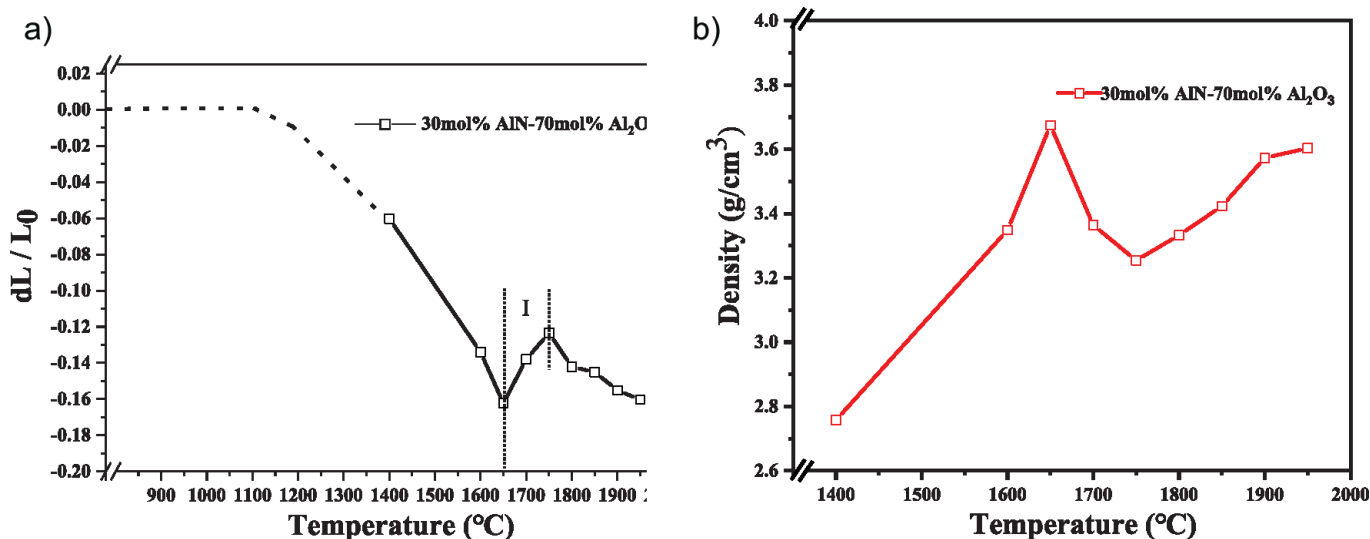


Fig. 5: (a) Dilatometric curve and (b) density of the samples containing 30 mol% AlN – 70 mol% Al_2O_3 after sintering at 1400 – $1950\ ^\circ\text{C}$.



Fig. 6: Optical images of samples containing 30 mol% AlN – 70 mol% Al₂O₃ after sintering at 1 950 °C for different holding times.

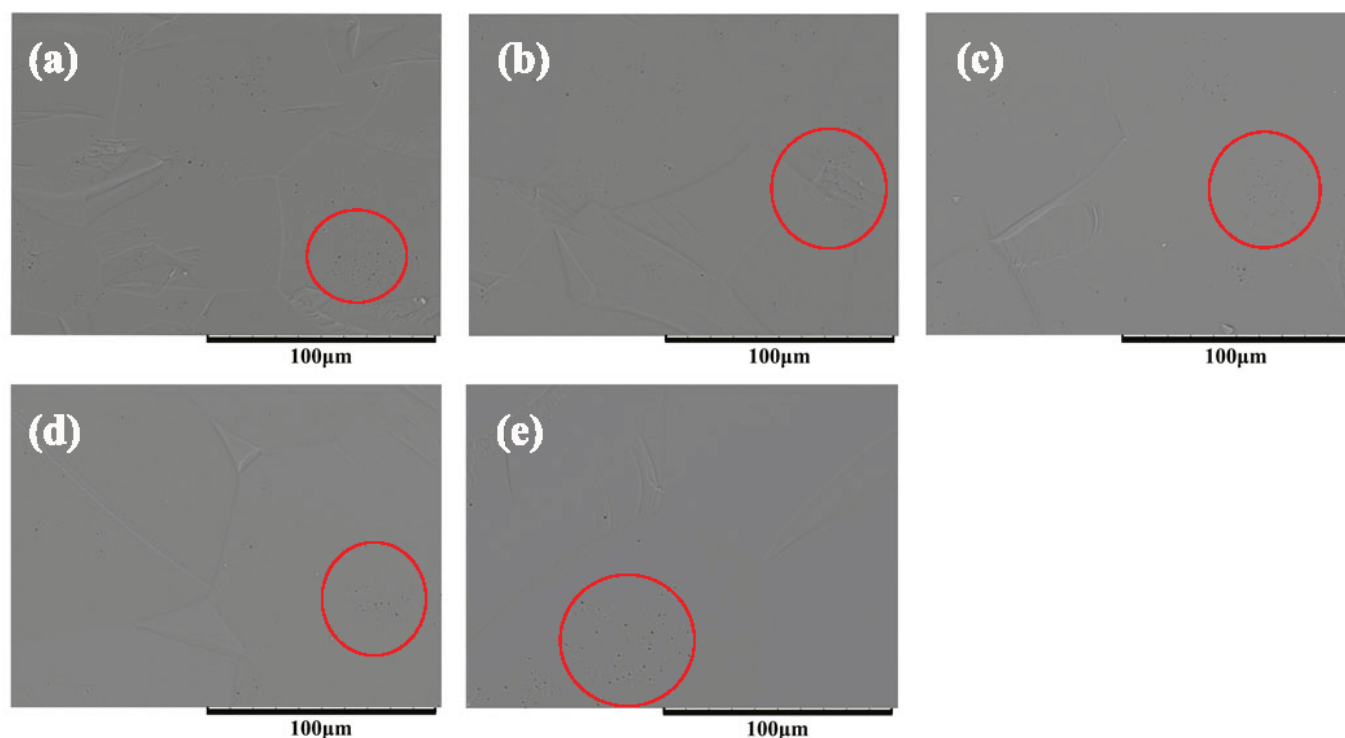


Fig. 7: Backscattered electron (BSE) micrographs of 30 mol% AlN – 70 mol% Al₂O₃ samples sintered at 1 950 °C for different holding times: (a) 5 h, (b) 10 h, (c) 15 h, (d) 20 h, (e) 30 h.

As shown in Fig. 6, the sintered samples underwent a distinct color evolution: initially colorless, then gradually turning black, followed by grayish, and eventually becoming white with the increase in holding time. XRD analysis confirmed that all these samples, regardless of color, consisted solely of the AlON phase with no detectable impurities. Moreover, this same color evolution sequence was consistently observed across compositions from 25 to 42 mol% AlN sintered under identical conditions, indicating that the blackening is an inherent and unavoidable intermediate stage during the reactive sintering of AlON. Notably, previous studies on reactively sintered AlON ceramics have also reported gray or black coloration^{9,29–30}, although little or no attention has been paid to its origin.

To explore the cause of this phenomenon, an attempt was first made to eliminate common sources of discoloration in ceramics. Fig. 7 shows the corresponding microstructural evolution of the 30 mol% AlN – 70 mol% Al₂O₃ samples sintered at 1 950 °C for different holding times. Significant grain growth and pore reduction are observed with the

increase in holding time. However, these microstructural changes do not explain the blackening phenomenon as no remarkable structural differences are observed between samples of different colors; small and aggregated pores primarily remain inside the grains even after long holding times. Moreover, the eventual disappearance of the gray coloration argues against carbon contamination from the graphite furnace.

With phase composition, microstructure, and carbon contamination excluded, point defects were considered as a potential cause. In many oxide ceramics, such as Al₂O₃, ZrO₂, and MgAl₂O₄, point defects like oxygen vacancies generated under vacuum, inert, or reducing atmospheres are known to induce coloration^{31–34}. However, attributing the blackening phenomenon in AlON to oxygen vacancies appears unlikely for two reasons. First, in this work, coloration diminished with prolonged sintering. This behavior contradicts an oxygen-vacancy-based mechanism because such vacancies typically approach thermodynamic equilibrium under extended sintering, resulting in persistent or inten-

sified coloration^{35–36}. Second, the formation of oxygen vacancies is inconsistent with the prevailing defect-chemistry model for AlON, described by the McCauley's formula $\text{Al}_{(64+x)/3}\text{V}_{[8-x]/3}\text{O}_{32-x}\text{N}_x$ (V refers to cation vacancies, $2 < x < 5$), where charge compensation occurs through cation vacancies rather than oxygen vacancies²³. Therefore, blackening is more likely related to the diffusion of aluminum cation vacancies during sintering. Although the existing characterization techniques cannot verify this hypothesis, the available evidence strongly suggests a defect-related origin. Further studies – potentially involving advanced spectroscopic or computational methods, as well as experiments under controlled sintering atmospheres (e.g., N_2 partial pressure) – are needed to elucidate the detailed mechanism underlying this blackening phenomenon.

IV. Conclusions

This work systematically elucidated the phase transformation and microstructural evolution during the reactive sintering of AlON from Al_2O_3 and AlN, providing a clear understanding of the underlying sintering mechanism. In the wide composition range of 25–40 mol% AlN (with corresponding Al_2O_3), the initial shrinkage of the sintered sample was attributed to the densification of Al_2O_3 prior to the formation of AlON phase. As the sintering temperature increased, a nitrogen-rich AlON solid solution gradually formed, accompanied by volume expansion until the complete consumption of AlN. Subsequently, the remaining Al_2O_3 diffused into the nitrogen-rich AlON lattice, reaching the nominal composition. The final stage involved densification of AlON ceramics, accompanied by shrinkage, grain growth, and pore elimination. For the sample containing 42 mol% AlN, only AlON phase was formed with the increase in temperature from 1750 °C to 1950 °C. By contrast, for the sample containing 45 mol% AlN, AlON coexisted with residual AlN at 1750 °C and 1950 °C. At higher temperatures, AlN disappeared, while the 21R-AlON phase emerged. Furthermore, a transient blackening phenomenon, likely related to the diffusion of aluminum cation vacancies, appeared as an inevitable intermediate stage during the densification of AlON ceramics. A complete mechanistic understanding of this blackening phenomenon remains an important objective for future research. Overall, the findings of this study can serve as a useful reference for optimizing the reactive sintering process to achieve dense, single-phase AlON ceramics with controlled microstructure and optical properties.

Acknowledgements

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