Enhancing the Strength and Toughness of 3D-Printed Alumina Reinforced with Boron Nitride Nanotubes

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BNNT-alumina interface, BNNT-induced microstructural refinements, and the critical role of silica additives as high-temperature sintering aids. Our findings provide valuable insights into the process-structure-property relationships and the reinforcement mechanisms of BNNT-reinforced ceramics, paving the way for the optimal design and manufacturing of lightweight, strong, tough, and durable ceramic materials.

KEYWORDS: boron nitride nanotubes, ceramics, additive manufacturing, mechanical enhancement, microstructures

1. INTRODUCTION

Additive manufacturing (AM) is revolutionizing the manufacturing of ceramics, which are renowned for their lightweight and high-strength properties. However, the inherent porous microstructure of AM-produced ceramics makes them susceptible to fracture, a long-standing shortcoming for ceramics. Reinforcement using nanofillers offers a promising solution. Boron nitride nanotubes (BNNTs) are exceptional candidates for ceramics reinforcement¹⁻³ because of their outstanding mechanical, thermal, and chemical properties. BNNTs exhibit an ultrahigh Young's modulus (up to ~1.3 TPa)⁴⁻¹² and tensile strength (up to ~60 TPa),¹³ comparable to carbon nanotubes (CNTs) and positioning them among the strongest known materials. Despite their longitudinal stiffness, BNNTs are remarkably flexible in the transverse direction,^{14–17} enabling them to conform effectively to the ceramic particle surface, enhancing load transfer and reinforcement. Unlike CNTs, BNNTs demonstrate exceptional thermal stability, maintaining their pristine structure at temperatures reaching 900 °C in oxidative environments¹⁸ or over 1800 °C inside inert gas.¹⁹ Additionally, BNNTs possess a nearly zero or even negative coefficient of thermal expansion (CTE),^{20,21} which helps preserve or even reduce the already low CTE of ceramics. Our recent studies reveal superior load transfer characteristics at the BNNT-ceramic interface, with nanotube pullout as the primary interfacial failure mechanism.^{22,23} The

robust BNNT-ceramic interaction arises from the partially ionic B-N bonding characteristics and the resulting anisotropic energy landscapes, enabling BNNTs to act as crack arresters within ceramics. The energy dissipation through frictional sliding during nanotube pullout enhances the toughness of the ceramic composite.

Those distinctive mechanical properties allow BNNTs to significantly enhance the bulk mechanical performance of ceramics even at low concentrations, as documented in the literature. For instance, incorporating 0.1 wt % of BNNTs into silica (SiO_2) improves the flexural modulus by ~72%,³ flexural strength by ~51–55%,^{2,3} and fracture toughness by ~46%.² Similarly, adding 1.0 to 5.0 wt % BNNTs to alumina (Al_2O_3) increases flexural strength by 59–67%,^{24,25} fracture toughness by 17–51%,^{24–26} and hardness by 117%.²⁶ Notably, these property enhancements compare favorably to those of conventional ceramic matrix composites that often require much higher filler contents, reaching 20–30% or higher.^{24,27,28} While most of those extraordinary mechanical enhancements

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Figure 1. Manufacturing and mechanical properties of DLP-printed BNNT-alumina nanocomposites: (a) schematic representation of the key manufacturing processes; (b) optical images depicting the green parts, sintered parts, and notch-machined parts (0.6% BNNT); (c) representative flexural stress–strain curves; (d) flexural strength and fracture toughness; (e) Vickers hardness (inset: SEM image showing a representative Vickers indentation, scale bar 500 μ m); (f) coefficient of thermal expansion.

are observed in composites manufactured using traditional molding methods, studies on AM-produced BNNT-ceramics remain scarce.³ This gap in the literature underscores the need for further investigations into the potential of BNNTs in AM ceramic composites.

AM offers distinct advantages over conventional ceramic manufacturing methods. It enables the fabrication of complex geometries with exceptional precision and design flexibility, often challenging or unattainable using traditional techniques. A variety of AM techniques have been used for ceramic fabrications, including fused filament fabrication (FFF),²⁹ selective laser melting (SLM),³⁰ direct ink writing (DIW),³¹ and vat photopolymerization (VP).^{32,33} Digital light processing (DLP), a subset of VP technologies, has emerged as the most widely adopted AM technology for ceramics because of its superior printing resolution and rapid printing speed.³⁴ In DLP-based 3D printing, a photosensitive slurry vat is exposed

to UV light through a glass panel. An integrated projector beneath the slurry vat projects 3D image slices onto the resin layers. Once a layer is cured, the build platform moves vertically upward, allowing a fresh slurry to refill the gap between the release film and the previously cured layer. The layer-by-layer process repeats until the complete 3D structure is fully fabricated.

However, DLP-manufactured ceramics encounter several challenges, including formation of pore defects, cracks, and delamination, primarily caused by polymer removal during high-temperature debinding. Those microstructural defects significantly compromise the mechanical properties of DLP-produced ceramics compared to those created using traditional methods.³⁵ Consequently, enhancing the mechanical properties of ceramics produced via the DLP technique is essential for boosting their durability and reliability and broadening the scope of applications of AM-built ceramics.

con	BNNT centration (wt %)	flexural strength (MPa)	flexural (elastic) modulus (GPa)	fracture toughness (MPa·m ^{1/2})	hardness (GPa)	CTE at room temperature (ppm/°C)	density (g/cm ³)	porosity (%)
	0	47.2 ± 4.1	18.1 ± 1.4	1.01 ± 0.07	3.6 ± 0.6	5.91 ± 0.22	2.77 ± 0.13	29.0 ± 3.4
	0.2	58.7 ± 3.7	22.2 ± 1.4	1.30 ± 0.11	3.7 ± 0.5	5.70 ± 0.15	2.70 ± 0.17	30.9 ± 4.4
	0.4	68.6 ± 3.1	25.3 ± 1.8	1.54 ± 0.07	4.3 ± 0.5	5.52 ± 0.24	2.61 ± 0.13	33.1 ± 3.5
	0.6	80.6 ± 6.3	30.1 ± 2.2	1.76 ± 0.13	4.6 ± 0.4	5.30 ± 0.29	2.59 ± 0.14	33.6 ± 3.6

Table 1. Mechanical and Structural Properties of 3D-Printed BNNT-Alumina Nanocomposites

This study explores the mechanical property enhancements of DLP-produced BNNT-reinforced alumina nanocomposites. The bulk mechanical properties, local interfacial load transfer between BNNTs and the alumina matrix, and microstructure of the BNNT-alumina nanocomposite are comprehensively analyzed using in situ Raman micromechanical measurements in conjunction with microstructural analysis. The addition of tiny amounts of BNNTs substantially enhances the mechanical properties of DLP-produced alumina, highlighting the critical roles of interfacial load transfer and BNNT-induced microstructure alterations. The findings provide valuable insights into the process–structure–property relationship and the reinforcement mechanisms of BNNT-reinforced ceramics, guiding the optimal design and manufacturing of lightweight, strong, tough, and durable ceramic materials.

2. EXPERIMENTAL SECTION

2.1. Materials and Manufacturing. Figure 1a illustrates the essential steps to fabricate DLP-produced BNNT-alumina nanocomposites. Those processes are described in detail in the Materials and Experimental Methods section of the Supporting Information. Briefly, the as-received, puffball-like, and high-purity BNNTs (BNNT Materials, Inc.), produced using high-temperature pressure (HTP) methods,³⁶ are first dispersed in acetone through ultrasonication. Next, α -alumina powders with an average particle size of ~500 nm and a specific surface area of $\sim 20 \text{ m}^2/\text{g}$ (US Research Nanomaterials, Inc.) are blended into the BNNT dispersion. To aid the sintering of alumina, amorphous silica powders with an average particle size of ~400 nm (US Research Nanomaterials, Inc.) are added at a mass ratio of 5% relative to alumina. The acetone suspension is then uniformly mixed using a tip sonicator. The homogenized suspension is dried on a hot plate at 90 °C for 12 h to remove acetone. The dried composite material is ground with a mortar and sieved through a 50mesh sieve to produce fine ceramic composite powders. The powders are mixed with UV-sensitive resins and a photoinitiator to formulate the 3D printing paste.

The green bodies of BNNT-alumina nanocomposites are manufactured using a bottom-up DLP 3D printer (Hunter, Flashforge Inc.) equipped with a 405 nm light source, followed by a three-stage thermal debinding at 429 °C, 535 °C, and 700 °C. The first two debinding temperatures correlate with two significant mass-loss peaks in the derivative thermogravimetric (DTG) profile (Figure S1 in the Supporting Information), which shows stable mass above 650 °C, indicating complete removal of polymer binders. After thermal debinding, the BNNT-alumina samples are sintered at 1450 °C for 1 h in a pressureless tube furnace under argon flow, followed by natural cooling. Alumina nanocomposites with BNNT loadings of up to 0.6 wt % are fabricated using the same approach. Unless stated otherwise, BNNT concentrations are expressed as weight percentages throughout this study. Figure 1b shows the optical images of the typical asprinted green bodies, sintered components, and notch-machined samples of the BNNT-alumina. The average volume shrinkage for alumina samples containing BNNT concentrations of 0.0%, 0.2%, 0.4%, and 0.6% is determined to be approximately 42.2%, 40.6%, 38.7%, and 38.2%, respectively.

2.2. Characterization. The rheological properties of BNNT-ceramic slurries are characterized using a rotational rheometer, while the thermal debinding behavior of the green body is analyzed via

thermogravimetric analysis (TGA) in air. The microstructure and morphology of the printed nanocomposites are examined using microcomputed tomography (micro-CT) and scanning electron microscopy (SEM). Phase composition is identified by X-ray diffraction (XRD), and thermal expansion is evaluated using thermomechanical analysis (TMA). Raman spectroscopy, including in situ micromechanical Raman testing, is employed to assess BNNT dispersion and interfacial load transfer characteristics.

Mechanical properties, including flexural strength and fracture toughness, are characterized through quasi-static three-point bending and single-edge notched beam (SENB) tests. Flexural stress and strain are calculated based on classical beam theory, and fracture toughness is calculated by accounting for notch tip stress concentration and Weibull statistical analysis. Vickers microhardness testing is performed in accordance with ASTM E384.

Comprehensive experimental parameters, analytical equations, and data processing procedures are provided in the Supporting Information.

3. RESULTS AND DISCUSSION

3.1. Bulk Mechanical Properties. 3.1.1. Flexural Strength. Figure 1c presents the representative flexural stress-strain curves of alumina nanocomposites subjected to three-point bending along the width direction (i.e., perpendicular to the layer stacking or building direction, as illustrated in the inset drawings of Figure 1d). That orientation exhibits superior properties compared to the thickness (or building) direction.³ The flexural strength of the alumina nanocomposites consistently increases with increasing BNNT concentrations. However, the fracture strain remains relatively constant, ranging from approximately 0.26% to 0.28%. Those observations indicate that the failure mechanism in BNNTalumina is dominated by matrix failure. The addition of amorphous silica noticeably enhances the failure strain of pure alumina from $\sim 0.15\%$ (without silica, as shown in Figure S2) to ~0.26% (with silica), while its flexural strength remains unchanged. Figure 1d shows the flexural strength and fracture toughness of BNNT-alumina nanocomposites, which are summarized in Table 1. The measured average flexural strength of pristine alumina is 47.2 ± 4.1 MPa.

Previous studies report that the flexural strength of alumina ceramics produced using VP-based 3D printing typically ranges from 3.3 to 50 MPa when sintered at temperatures from 1200 to 1600 $^{\circ}$ C.³⁷ That wide variation is attributed to the well-known relationship between the sintering temperature and alumina density, where higher temperatures and pressure improve densification. In this study, which employs pressure-less sintering, the measured flexural strength of 47.2 MPa is near the upper limit of the previously reported range. The superior performance is attributed to the use of submicrometer-sized alumina powders. Unlike the micrometer-sized powders commonly used in previous studies, the finer particles promote enhanced densification, resulting in improved mechanical properties. Incorporating BNNTs leads to a progressive increase in the flexural strength of alumina,



Figure 2. Failure analysis of BNNT-alumina nanocomposites: (a) Two-dimensional microcomputed tomography analysis highlighting structural defects (*z* axis aligned with the building direction); (b) SEM micrograph revealing interlayer defects; (c,d) SEM images of the fractured surface morphology (0.6% BNNT). The red arrows indicate protruding BNNTs, while the blue arrows indicate bridging nanotubes across pores and particles. The insert atomic force microscopy image in (c): a typical BNNT of ~600 nm in length and ~3 nm in diameter (scale bar: 100 nm).

reaching 58.7 ± 3.7 MPa (0.2%), 68.6 ± 3.1 MPa (0.4%), and 80.6 ± 6.3 MPa (0.6%), corresponding to increases of 24%, 45%, and 71%, respectively.

3.1.2. Fracture Toughness. DLP-printed alumina ceramics demonstrate a mean fracture toughness of 1.01 MPa·m^{1/2}, considerably lower than the reported values of 4.0-6.0 MPa $m^{1/2}$ for conventionally manufactured alumina. 38 The substantial reduction is primarily attributed to the microstructural characteristics of the DLP-fabricated alumina ceramics. In those ceramics, the internal defects form during thermal debinding and elevated residual porosity remains after sintering.³ Incorporating small amounts of BNNTs significantly improves the fracture toughness of alumina, achieving $1.30 \pm 0.11 \text{ MPa} \cdot \text{m}^{1/2}$ (0.2%), $1.54 \pm 0.10 \text{ MPa} \cdot \text{m}^{1/2}$ (0.4%), and 1.76 \pm 0.13 MPa·m^{1/2} (0.6%), respectively. Figure 1e shows that the introduction of BNNTs substantially enhances the hardness of alumina, reaching ~4.6 GPa with 0.6% BNNT, which corresponds to a 27.8% increase compared to that of pure alumina (~3.6 GPa).

3.1.3. Coefficient of Thermal Expansion. Figure 1f shows the evolution of the CTE with temperature for BNNT-alumina nanocomposites, based on the measured thermal expansion strain curves along the specimen's length (Figure S3). The CTE is calculated as $\alpha = d(\ln L)/dT$, where L = L(T)represents the measured specimen length and T is the temperature. The CTE of the alumina nanocomposite increases linearly with the temperature and decreases with an increase in BNNT concentration. Specifically, the CTE of BNNT-alumina at room temperature (~25 °C) and 900 °C is 5.91 (8.79), 5.70 (8.30), 5.52 (7.90), and 5.30 (7.41) ppm/°C for 0%, 0.2%, 0.4%, and 0.6% BNNT concentrations, respectively. The significant reduction in CTE (up to 10.3% at room temperature and 15.7% at 900 °C) is primarily because (i) BNNTs exhibit a considerably lower CTE than the DLP-produced alumina matrix (~0.57 ppm/°C for BNNT compared to 6.9 ppm/°C for alumina at room temperature) and (ii) the effective load transfer at the BNNT-alumina

interface restricts the thermal expansion of alumina caused by the higher stiffness of BNNTs.²¹ Notably, adding silica significantly lowers the CTE of the alumina matrix (\sim 14.4%) because of its CTE being substantially lower than that of alumina.

The remarkable enhancement of mechanical properties achieved with minimal BNNT additions highlights their exceptional potential as ceramic fillers for advanced ceramics technology. In this study, the BNNT concentration is limited to 0.6% to avoid excessive increases in slurry viscosity (see Figure S4), which could hinder effective layer recoating—a critical step in DLP-based 3D printing. For instance, adding 0.8% BNNTs results in an alumina suspension viscosity of ~3.2 Pa·s, which exceeds the threshold viscosity of 3 Pa·s at a shear rate of 30 s⁻¹.^{34,39} Alternative processing methods, such as conventional powder pressing—sintering, may be necessary to explore whether the observed mechanical enhancements continue or plateau at higher BNNT loadings.

The silica additive plays a crucial role in realizing the superior reinforcement of BNNTs in alumina. As shown in Figure S2, the flexural strength of BNNT-alumina fabricated without silica sintering additives consistently decreases with higher BNNT concentrations. The decrease is caused by the pressure-free sintering employed in this study. During pressureless sintering, densification is primarily driven by a reduction in surface energy, which promotes atomic diffusion and the formation of sintering necks between adjacent alumina particles. However, the incorporation of BNNTs can negatively affect the process. Because of their significantly lower CTE as compared to alumina particles and their poor interfacial wettability, BNNTs tend to accumulate at particle boundaries, disrupting particle-to-particle contact and inhibiting neck formation. The interference with local mass transport limits the densification of the alumina matrix and can lead to an increased residual porosity, potentially compromising the bulk mechanical properties of the nanocomposite.

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Figure 3. In situ Raman micromechanical measurements: (a) Representative Raman spectra; (b) comparative Raman mapping analysis of pristine BNNTs and 0.6% BNNT-alumina nanocomposites; (c) schematics of the in situ three-point bending Raman spectroscopy (building direction perpendicular to loading-pin displacement): 3D configuration (left) and planar positions of selected measurement points A, B, and C (right); (d) evolution of Raman spectra at point A in a 0.6% BNNT-alumina nanocomposite under varying matrix strains during three-point bending tests; (e) in situ monitoring of the BNNT characteristic peak shifts at points A, B, and C as a function of maximum matrix strain along the *x* direction.

The incorporation of amorphous silica serves as a sintering aid and enhances the densification of alumina.⁴⁰ The primary mechanism lies in the formation of a low-melting glassy phase during sintering. That liquid glassy phase facilitates densification through the wetting of alumina particle surfaces, which promotes interparticle connections, and the acceleration of ionic diffusion and recrystallization

3.2. Failure Modes. *3.2.1. Interlayer Defects.* Figure 2a displays the two-dimensional (2D) micro-CT images of alumina, captured with the X-ray tube oriented perpendicular (top) and parallel (bottom) to the building direction. The top image reveals distinct interlayer cracks that are perpendicular to the building direction, which are commonly observed defects in DLP-printed ceramics.⁴¹ Those interlayer cracks originate from weak interlayer bonding forces encountered during DLP printing combined with gas escaping preferentially along the layer interfaces during thermal debinding. That

combination of factors ultimately leads to the formation of interlayer cracks. In contrast, the bottom image does not show interlayer crack defects but reveals spherical voids and pores of varying sizes. Those structural defects contribute to the degradation of mechanical properties in 3D-printed alumina ceramics, explaining the significant reduction in mechanical performance compared with conventionally manufactured counterparts. SEM imaging of DLP-printed alumina along the building direction, as shown in Figure 2b, clearly depicts individual layers that are distinctly visible and oriented perpendicular to the building direction. The SEM analysis reveals the presence of delamination cracks, which are consistent with micro-CT findings.

3.2.2. Protruding Nanotubes. Figures 2c and 2d display the cross-sectional morphologies of a fractured 0.6% BNNT-alumina specimen. The nanotubes are uniformly dispersed within the alumina matrix without any noticeable agglomer-



Figure 4. Interfacial load transfer characteristics: Schematics of (a) the alumina nanocomposite reinforced by random nanotubes and (b) the equivalent single-nanotube nanocomposite configuration; theoretical predictions of (c) the point-wise IFSS distribution and (d) the normal stress in the nanotube in a 0.6% BNNT-alumina at failure strain.

ation, which is crucial for optimal load transfer between the nanotubes and the matrix. Effective load transfer is evidenced by the individual BNNTs protruding from the fractured surface, which occurs as a result of nanotube pullout. As highlighted by the blue arrows, BNNTs bridge pores and ceramic particle boundaries.

Stress concentrations typically develop around pores when the matrix is subjected to external loads. The pore-bridging behavior of BNNTs alleviates stress concentrations and inhibits local crack formation, thereby enhancing the material's overall strength. Cracks tend to propagate rapidly through the pores and particle boundaries in ceramics. The nanotube pullout and bridging behavior observed within the ceramic matrix are believed to enhance resistance to crack propagation through two primary mechanisms: increased energy dissipation via frictional sliding during nanotube pullout as cracks propagate and crack path deflection.⁴² Those microscopically observed BNNT features within the alumina matrix provide valuable insights into the macroscopic enhancements of strength and fracture toughness in ceramic nanocomposite.

3.3. Interfacial Load Transfer Characteristics. 3.3.1. In Situ Raman Micromechanical Measurements. Figure 3a displays the typical Raman spectra for pristine BNNTs and 0.6% BNNT-alumina. The pristine BNNTs exhibit a prominent G band at ~1369 cm^{-1.43} In contrast, the 0.6% BNNT-alumina shows a considerably weaker, yet discernible, G band slightly shifted to ~1371 cm⁻¹. Lower BNNT concentrations result in G bands that are too weak for an accurate measurement. Consequently, Raman measurements were performed exclusively on the 0.6% BNNT nanocomposite to ensure a sufficient peak intensity for reliable analysis. Figure 3b presents the Raman mapping of $18 \times 36 \,\mu\text{m}$ areas on pristine BNNTs and 0.6% BNNT-alumina with a step interval of 1.2 μ m (i.e., the laser spot size). The average Raman shift is calculated to be 1368.6 \pm 0.2 cm⁻¹ for BNNTs and 1370.2 \pm 0.6 cm⁻¹ for BNNT-alumina. The observed blue shift indicates that the BNNTs are compressed within the alumina matrix. The compression is attributed to the compressive stress and strain associated with the volumetric contraction of the nanocomposite green body during sintering and the CTE mismatch between BNNTs and alumina during cooling from high-temperature sintering. The Raman mapping results confirm the uniform distribution of BNNTs within the ceramic nanocomposite.

The left schematic in Figure 3c illustrates the in situ Raman micromechanical characterization of BNNT-alumina used to investigate the interfacial load transfer behavior. In this experimental setup, an unnotched BNNT-alumina specimen is subjected to a three-point bending test, while Raman spectra are simultaneously recorded at three selected points (A, B, and C) as indicated in Figure 3c (right).

Figure 3d displays the evolution of the G-band peak frequency of BNNTs as a function of the maximum strain in the ceramic matrix (i.e., the strain at point A). The G-band peak frequency shows a consistent decreasing trend as the maximum strain increases. The observation indicates that within the strength range of the nanocomposite, the applied load is effectively transferred from the alumina matrix to the nanotubes as the external load increases. After the failure of the nanocomposite specimen, the G-band peak frequency returns to its original, preloading position, which is attributed to the release of strain in the matrix and nanotubes following fracture failure. The reversible behavior indicates that the BNNT–



Figure 5. Comparative analysis of the experimentally measured and ROM-predicted mechanical properties of BNNT-alumina nanocomposites: (a) elastic modulus and (b) flexural strength.

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alumina interface maintains its integrity throughout the loading with no noticeable interfacial sliding or debonding.

The finding aligns well with previous studies that reported strong interfacial binding interactions between BNNTs and alumina.¹³ The failure mechanism of BNNT-alumina is dominated by matrix failure, which is fundamentally different from the failure mechanisms observed in BNNT-polymer nanocomposites,^{44,45} BNNT-metal nanocomposites,¹³ or conventionally manufactured BNNT-silica nanocomposites,² where interfacial failure typically occurs before matrix failure.

Figure 3e illustrates the strain-induced Raman shifts at points A-C during the deformation of the alumina nanocomposite. A consistent decrease in the G-band peak frequency is observed at points A and B before macroscopic failure. The slope of the Raman shift per unit percent of maximum matrix strain is calculated to be approximately -3.0 \pm 0.3 cm⁻¹/% at point A, while point B shows about half that value at -1.6 ± 0.2 cm⁻¹/%. The difference is attributed to the matrix strain at point B being half that at point A, as predicted by classical beam theory. That observation demonstrates that throughout the loading, the applied load on the matrix is effectively transferred to the nanotubes at points A and B through interfacial load transfer. At point C, which is located at the neutral axis of the beam, the Raman shift curve remains nearly unchanged, indicating that the nanotubes in that region undergo minimal deformation. Following the macroscopic fracture of the nanocomposite specimen, the Raman peak frequencies at all points return to their initial preloading values. The reversible behavior confirms that the deformations in the matrix and nanotubes are elastic.

3.3.2. Local Interfacial Load Transfer Analysis. The straining of BNNTs within the ceramic matrix arises from the interfacial shear stress transfer. For a sufficiently long and straight nanotube, the strain accumulates from both ends of the nanotube and remains constant along its middle region. In the DLP-manufactured alumina nanocomposite, BNNTs exhibit a random distribution rather than a unidirectional alignment, as revealed by polarized Raman spectroscopy measurements from our recent study.²¹ Figure 4a depicts a simplified two-dimensional model of the nanocomposite system reinforced with uniformly distributed but randomly oriented nanotubes. The model is based on two fundamental assumptions: BNNTs are sparsely distributed within the alumina matrix, maintaining identical nanocomposite cell dimensions, and the nanotube–ceramic matrix interface is

uniform and fully bonded. In this model, the equivalent strain $(\epsilon_{\rm nt}^{\rm eq})$ in randomly distributed BNNTs can be calculated from the matrix strain (ϵ_m) , which is given by $\epsilon_{\rm nt}^{\rm eq} = \frac{1-v_{\rm m}}{2}\epsilon_{\rm m}$, where $v_{\rm m}$ is Poisson's ratio of the matrix. The relationship indicates that the equivalent nanotube strain corresponds to the strain of nanotubes oriented at 45° relative to the tensile direction of the matrix. For the BNNT-alumina nanocomposite with a maximum matrix failure strain of 0.25%, the equivalent strain in BNNTs is calculated to be ~0.1%, assuming $v_{\rm m} = 0.22$.⁴⁶ The corresponding normal tensile stress in the nanotubes is estimated to be ~1.07 GPa, based on the Young's modulus $E_{\rm nt} = 1.07$ TPa for HTP-BNNTs.⁴⁷ The calculation demonstrates the effective stress transfer from the alumina matrix to the BNNTs within the nanocomposite structure.

Figure 4b illustrates the simplified nanocomposite unit cell configuration containing a single nanotube. The interfacial shear stress (IFSS) distribution along the nanotube-matrix interface is analyzed using shear-lag theory and is expressed as

$$\tau = \frac{E_{\rm nt}\epsilon_{\rm nt}^{\rm eq}n}{2} \frac{\cosh(2nz/D_{\rm nt})}{\sinh(nl/D_{\rm nt})}$$
(1)

where $n = \sqrt{\frac{E_m}{E_m(1 + v_m)\log(\frac{D_m}{D_{nt}})}}$, E_m is the Young's modulus of the

matrix, $D_{\rm m}$ represents the equivalent diameter of the matrix in the nanocomposite cell, and $D_{\rm nt}$ and l are the diameter and length of the nanotube, respectively. Figure 4c presents the pointwise interfacial shear stress (IFSS) distribution along the nanotube in 0.6% BNNT-alumina. The calculation is based on the following parameters: $E_{\rm m} = 300$ GPa; $D_{\rm nt} = 2.9$ nm (the median diameter of BNNTs^{48,49}); $D_{\rm m} = 31.3$ nm, calculated based on the densities of alumina (3.9 g/cm³) and BNNTs (1.35 g/cm³). The maximum IFSS that occurs at the nanotube ends (i.e., $z = \pm l/2$) is calculated to be independent of the nanotube length (*l*) when l > 44 nm, assuming a shear stress threshold of 1 MPa. For the calculation, *l* is set to 400 nm. The IFSS before nanocomposite failure is calculated to be ~180 MPa.

The average IFSS in the effective interfacial transfer region (i.e., $\tau \ge 1$ MPa) is calculated to be ~26 MPa, substantially lower than the previously reported value of ~46.0 MPa obtained using single-nanotube pullout measurements.¹³ The discrepancy further supports that the primary failure mode in 3D-printed BNNT-alumina originates from the failure of the



Figure 6. Microstructural characterization of BNNT-alumina nanocomposites: (a) Density and porosity; (b) XRD spectra; (c) representative Williamson–Hall plots of the pure alumina and 0.6% BNNT-alumina; (d) assessment of crystalline size and lattice strain via the Williamson–Hall analysis approach.

alumina matrix rather than from the BNNT-matrix interface. The findings indicate that there is significant potential for improving interfacial load transfer through optimization of the microstructure of 3D-printed BNNT-ceramics. Figure 4d shows the normal stress distribution along the BNNT. The maximum normal stress under the nanocomposite failure load reaches 1.07 GPa, substantially lower than the tensile strength of HTP-BNNTs (up to 60 GPa).¹³

The micromechanical analysis confirms the effective interfacial load transfer characteristics as one of the primary reinforcement mechanisms in BNNT-alumina. The exceptionally high stiffness and strength of BNNTs enable them to carry a substantial portion of the applied load when they are wellbound to the surrounding matrix. The efficient load transfer facilitates stress redistribution, mitigates local stress concentrations, and inhibits crack initiation and propagation, thereby significantly enhancing the overall strength of the nanocomposite.

3.4. Theoretical Prediction of the Bulk Mechanical Properties. Predicting how the macroscopic mechanical properties of ceramic materials vary with the BNNT concentration is crucial for guiding the optimal design and manufacturing of advanced ceramic nanocomposites and pursuing their applications. In this study, we use the Rule of Mixtures (ROM) model to theoretically predict the bulk mechanical properties of BNNT-reinforced alumina, specifi-

cally, the elastic modulus and flexural strength. The elastic modulus of the nanocomposite (E_c) using the ROM model is expressed as⁴³

$$E_{\rm c} = \eta V_{\rm nt} E_{\rm nt} + (1 - V_{\rm nt}) E_{\rm m} \tag{2}$$

where $V_{\rm nt}$ is the volume fraction of BNNTs and η is the orientation factor of the nanotube inside the matrix (for randomly distributed nanotubes $\eta = 3/8$).⁵⁰ Figure 5a compares the normalized elastic modulus between the experimentally measured values and the ROM predictions. The measured elastic modulus consistently exceeds the values predicted by the ROM model. Furthermore, the discrepancy increases with the BNNT concentration. The tensile strength (σ_c) of the nanocomposite using the ROM model is given as

$$\sigma_{\rm c} = \eta V_{\rm nt} E_{\rm nt} \epsilon_{\rm cr} + (1 - V_{\rm nt}) f_{\rm t} \tag{3}$$

where $\epsilon_{\rm cr}$ is the strain at which interfacial failure occurs. In situ Raman micromechanical measurements indicate that failure in BNNT-alumina is governed by the matrix failure. Therefore, $\epsilon_{\rm cr}$ can be approximated using the flexural failure strain shown in Figure 1c. The matrix's tensile strength is estimated as $f_{\rm t} \approx 0.7 f_{\rm b}$ (see Section 1.5 in the Supporting Information).

Figure 5b shows that the experimentally measured flexural strength consistently surpasses the ROM predictions. Moreover, the discrepancy widens with an increasing BNNT concentration, mirroring the trend observed in the elastic modulus comparison. The ROM model only accounts for the property improvement resulting from the interfacial load transfer between the reinforcement filler and the matrix. The observed discrepancy between experiments and predictions can be attributed to the BNNT-induced microstructure changes in the 3D-printed alumina. The findings highlight the need for a systematic microstructural analysis of BNNTalumina nanocomposites to better understand the underlying mechanisms contributing to the enhanced mechanical properties.

3.5. Microstructural Analysis. Figure 6a shows the density and porosity of BNNT-alumina, which are summarized in Table 1. The porosity of the nanocomposite consistently increases with the BNNT concentration. The increased porosity and reduced ceramic density are consistent with prior findings^{2,51} and can be attributed to two primary factors: the inherently lower density of BNNTs compared to the alumina matrix and the presence of nanotubes hindering intimate contact between ceramic particles, thus impairing the localized densification.

The XRD spectra in Figure 6b reveal that α alumina remains the only detectable phase across all BNNT concentrations, confirming the absence of reaction phases or matrix phase transformations. The results demonstrate that incorporating BNNTs at relatively low concentrations maintains the superior characteristics of the α -alumina phase. To further investigate the effect of BNNTs on the microstructure of the α -alumina phase, the Williamson–Hall approach is employed to calculate the crystallite size and lattice strain in the alumina nanocomposite, which is given as⁵²

$$\beta \cos \theta_{hkl} = \frac{k\lambda}{D} + 4\epsilon^{W-H} \sin \theta_{hkl}$$
(4)

where β is the full width at half-maximum (fwhm), θ_{hkl} is the diffraction angle, k is the shape factor (0.9 for spherical particles), λ is the X-ray wavelength (1.54 Å for Cu K α radiation), D is the crystallite size, and e^{W-H} is the lattice strain. According to the Williamson-Hall model, the crystallite size and the lattice strain can be calculated from the y-axis intercept and the slope of the plot of $\beta \cos \theta_{hkl}$ versus 4 sin θ_{hkb} respectively. Figure 6c presents a comparative analysis of Williamson-Hall plots for pure alumina and 0.6% BNNTalumina. The fitted straight lines for 0.6% BNNT-alumina exhibit an increased y intercept and a decreased slope compared with pristine alumina. Correspondingly, the calculated crystallite size and lattice strain, as shown in Figure 6d, decrease almost linearly with increasing BNNT concentration. The trend indicates that BNNTs function as a grainrefining agent during the sintering of the alumina matrix, similar to their behavior in metal matrices.⁵³ The Hall–Petch relationship suggests that a decrease in crystallite size increases the density of grain boundaries, which hinders dislocation motion and enhances strength and toughness.⁵⁴ Simultaneously, the reduction in lattice strain may further enhance mechanical properties by softening internal stress concentrations and promoting a more uniform crystalline microstructure.55

Microstructural analysis of the alumina nanocomposite highlights the reinforcing role of BNNT-induced microstructural modifications. The measured strength enhancement reflects the combined effects of load transfer and BNNTinduced changes in the microstructure, whereas the ROM- predicted strength accounts solely for the load transfer mechanism. The discrepancy between experimental results and ROM predictions is therefore attributed to additional reinforcement provided by microstructural modifications. Those changes may include beneficial effects, such as particle refinement and reduced lattice strain, as well as adverse effects, such as increased porosity. Based on this classification of reinforcement mechanisms, the respective contributions of load transfer and microstructural modification to the overall strength improvement are estimated to be 17.9%/6.4% for 0.2% BNNT, 36.6%/8.8% for 0.4% BNNT, and 54.0%/16.6% for 0.6% BNNT.

4. CONCLUSIONS

In this study, we experimentally investigate the bulk and local mechanical properties of AM-produced BNNT-reinforced alumina nanocomposites. The results demonstrate that even small additions of BNNTs significantly enhance the mechanical properties of the nanocomposite. The macroscopic failure of AM BNNT-alumina is governed by matrix failure. Microstructural analysis reveals that the introduction of BNNTs preserves the crystallinity of α -alumina while inducing beneficial microstructural changes, including a refined grain size and reduced lattice strain. The findings suggest that effective load transfer, nanotube pullout and bridging effects, and favorable microstructural changes induced by BNNTs collectively contribute to the significant strengthening and toughening of the ceramic matrix. The findings provide fundamental insights into the reinforcement mechanisms of BNNTs within ceramic nanocomposites, thereby advancing the development of lightweight, durable, and reliable ceramic materials.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsanm.5c01183.

Manufacturing of the tested nanocomposite specimens, mechanical testing and analysis methods, thermal expansion measurements, and rheological property measurements (PDF)

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Notes

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REFERENCES

(1) Du, M.; Bi, J.-Q.; Wang, W.-L.; Sun, X.-L.; Long, N.-N. Microstructure and Properties of SiO2Matrix Reinforced by BN Nanotubes and Nanoparticles. *J. Alloys Compd.* **2011**, 509 (41), 9996–10002.

(2) Anjum, N.; Wang, D.; Gou, F.; Ke, C. Boron Nitride Nanotubes Toughen Silica Ceramics. ACS Appl. Eng. Mater. 2024, 2 (3), 735– 746.

(3) Tank, M.; Leon, A. D.; Huang, W.; Patadia, M.; Degraff, J.; Sweat, R. Manufacturing of Stereolithographic 3D Printed Boron Nitride Nanotube-Reinforced Ceramic Composites with Improved Thermal and Mechanical Performance. *Funct. Compos. Struct.* **2023**, 5 (1), 015001.

(4) Treacy, M. M. J.; Ebbesen, T. W.; Gibson, J. M. Exceptionally High Young's Modulus Observed for Individual Carbon Nanotubes. *Nature* **1996**, *381* (6584), 678–680.

(5) Chopra, N. G.; Zettl, A. Measurement of the Elastic Modulus of a Multi-Wall Boron Nitride Nanotube. *Solid State Commun.* **1998**, *105* (5), 297–300.

(6) Arenal, R.; Wang, M.-S.; Xu, Z.; Loiseau, A.; Golberg, D. Young Modulus, Mechanical and Electrical Properties of Isolated Individual and Bundled Single-Walled Boron Nitride Nanotubes. *Nanotechnology* **2011**, 22 (26), 265704.

(7) Hernandez, E.; Goze, C.; Bernier, P.; Rubio, A. Elastic Properties of C and BxCyNz Composite Nanotubes. *Phys. Rev. Lett.* **1998**, 80 (20), 4502–4505.

(8) Choyal, V.; Choyal, V. K.; Kundalwal, S. I. Effect of Atom Vacancies on Elastic and Electronic Properties of Transversely Isotropic Boron Nitride Nanotubes: A Comprehensive Computational Study. *Comput. Mater. Sci.* **2019**, *156*, 332–345.

(9) Suryavanshi, A. P.; Yu, M.-F.; Wen, J.; Tang, C.; Bando, Y. Elastic Modulus and Resonance Behavior of Boron Nitride Nanotubes. *Appl. Phys. Lett.* **2004**, 84 (14), 2527–2529.

(10) Zhao, Y.; Chen, X.; Park, C.; Fay, C. C.; Stupkiewicz, S.; Ke, C. Mechanical Deformations of Boron Nitride Nanotubes in Crossed Junctions. J. Appl. Phys. **2014**, 115 (16), 164305.

(11) Zheng, M.; Chen, X.; Park, C.; Fay, C. C.; Pugno, N. M.; Ke, C. Nanomechanical Cutting of Boron Nitride Nanotubes by Atomic Force Microscopy. *Nanotechnology* **2013**, *24* (50), 505719.

(12) Wei, X.; Wang, M.-S.; Bando, Y.; Golberg, D. Tensile Tests on Individual Multi-Walled Boron Nitride Nanotubes. *Adv. Mater.* **2010**, 22 (43), 4895–4899.

(13) Jiang, Y.; Li, N.; Liu, Z.; Yi, C.; Zhou, H.; Park, C.; Fay, C. C.; Deng, J.; Chew, H. B.; Ke, C. Exceptionally Strong Boron Nitride Nanotube Aluminum Composite Interfaces. *Extreme Mech. Lett.* **2023**, *59*, 101952.

(14) Zheng, M.; Ke, C.; Bae, I.-T.; Park, C.; Smith, M. W.; Jordan, K. Radial Elasticity of Multi-Walled Boron Nitride Nanotubes. *Nanotechnology* **2012**, *23* (9), 095703.

(15) Zheng, M.; Zou, L.; Wang, H.; Park, C.; Ke, C. Engineering Radial Deformations in Single-Walled Carbon and Boron Nitride Nanotubes Using Ultrathin Nanomembranes. *ACS Nano* **2012**, *6* (2), 1814–1822.

(16) Zheng, M.; Chen, X.; Bae, I.-T.; Ke, C.; Park, C.; Smith, M. W.; Jordan, K. Radial Mechanical Properties of Single-Walled Boron Nitride Nanotubes. *Small* **2012**, *8* (1), 116–121.

(17) Zheng, M.; Zou, L.; Wang, H.; Park, C.; Ke, C. Quantifying the Transverse Deformability of Double-Walled Carbon and Boron Nitride Nanotubes Using an Ultrathin Nanomembrane Covering Scheme. J. Appl. Phys. 2012, 112, 104318.

(18) Chen, X.; Dmuchowski, C. M.; Park, C.; Fay, C. C.; Ke, C. Quantitative Characterization of Structural and Mechanical Properties of Boron Nitride Nanotubes in High Temperature Environments. *Sci. Rep.* **2017**, *7* (1), 11388.

(19) Tank, M. J.; Reyes, A. N.; Park, J. G.; Scammell, L. R.; Smith, M. W.; De Leon, A.; Sweat, R. D. Extreme Thermal Stability and Dissociation Mechanisms of Purified Boron Nitride Nanotubes: Implications for High-Temperature Nanocomposites. *ACS Appl. Nano Mater.* **2022**, 5 (9), 12444–12453.

(20) Mashreghi, A. Thermal Expansion/Contraction of Boron Nitride Nanotubes in Axial, Radial and Circumferential Directions. *Comput. Mater. Sci.* **2012**, *65*, 356–364.

(21) Wang, D.; Chen, R.; Anjum, N.; Ke, C. Thermal Expansion of Boron Nitride Nanotubes and Additively Manufactured Ceramic Nanocomposites. *Nanotechnology* **2025**, *36* (6), 065703.

(22) Li, N.; Dmuchowski, C. M.; Jiang, Y.; Yi, C.; Gou, F.; Deng, J.; Ke, C.; Chew, H. B. Sliding Energy Landscape Governs Interfacial Failure of Nanotube-Reinforced Ceramic Nanocomposites. *Scr. Mater.* **2022**, *210*, 114413.

(23) Yi, C.; Bagchi, S.; Gou, F.; Dmuchowski, C. M.; Park, C.; Fay, C. C.; Chew, H. B.; Ke, C. Direct Nanomechanical Measurements of Boron Nitride Nanotube—Ceramic Interfaces. *Nanotechnology* **2019**, *30* (2), 025706.

(24) Wang, W.-L.; Bi, J.-Q.; Sun, K.-N.; Du, M.; Long, N.-N.; Bai, Y.-J. Fabrication of Alumina Ceramic Reinforced with Boron Nitride Nanotubes with Improved Mechanical Properties. *J. Am. Ceram. Soc.* **2011**, *94* (11), 3636–3640.

(25) Wang, W.-L.; Bi, J.-Q.; Wang, S.-R.; Sun, K.-N.; Du, M.; Long, N.-N.; Bai, Y.-J. Microstructure and Mechanical Properties of Alumina Ceramics Reinforced by Boron Nitride Nanotubes. *J. Eur. Ceram. Soc.* **2011**, *31* (13), 2277–2284.

(26) Pandey, K. K.; Singh, S.; Choudhary, S.; Zhang, C.; Agarwal, A.; Li, L. H.; Chen, Y.; Keshri, A. K. Microstructural and Mechanical Properties of Plasma Sprayed Boron Nitride Nanotubes Reinforced Alumina Coating. *Ceram. Int.* **2021**, 47 (7), 9194–9202.

(27) Li, H.; Liu, Y.; Liu, Y.; Zeng, Q.; Liang, J. Silica Strengthened Alumina Ceramic Cores Prepared by 3D Printing. *J. Eur. Ceram. Soc.* **2021**, 41 (4), 2938–2947.

(28) Becher, P. F.; Wei, G. C. Toughening Behavior in Sic-Whisker-Reinforced Alumina. J. Am. Ceram. Soc. 1984, 67 (12), C 267–C 269.
(29) Sarraf, F.; Abbatinali, E.; Gorjan, L.; Sebastian, T.; Colombo,

P.; Churakov, S. V.; Clemens, F. Effect of MgO Sintering Additive on Mullite Structures Manufactured by Fused Deposition Modeling (FDM) Technology. *J. Eur. Ceram. Soc.* **2021**, *41* (13), 6677–6686. (30) Qiu, Y.-D.; Wu, J.-M.; Chen, A.-N.; Chen, P.; Yang, Y.; Liu, R.-Z.; Chen, G.; Chen, S.; Shi, Y.-S.; Li, C.-H. Balling Phenomenon and Cracks in Alumina Ceramics Prepared by Direct Selective Laser Melting Assisted with Pressure Treatment. Ceram. Int. 2020, 46 (9), 13854–13861.

(31) Ye, Z.; Chu, C.; Zhang, D.; Ma, S.; Guo, J.; Cheng, Y.; Xu, G.; Li, Z.; Sun, A. Study on 3D-Direct Ink Writing Based on Adding Silica Submicron-Particles to Improve the Rheological Properties of Alumina Ceramic Ink. *Mater. Today Commun.* **2021**, *28*, 102534.

(32) Xing, Z.; Liu, W.; Chen, Y.; Li, W. Effect of Plasticizer on the Fabrication and Properties of Alumina Ceramic by Stereolithography-Based Additive Manufacturing. *Ceram. Int.* **2018**, *44* (16), 19939–19944.

(33) Chen, T.; Wang, D.; Chen, X.; Qiu, M.; Fan, Y. Three-Dimensional Printing of High-Flux Ceramic Membranes with an Asymmetric Structure via Digital Light Processing. *Ceram. Int.* **2022**, 48 (1), 304–312.

(34) Hsiang, H.-I.; Lee, C.-Y.; Chen, C.-C.; Wang, J.; Tang, D.; Dong, Z. Polycrystalline Alumina Ceramic Fabrication Using Digital Stereolithographic Light Process. *Ceram. Int.* **2021**, *47* (23), 33815– 33826.

(35) Zhang, K.; Meng, Q.; Qu, Z.; He, R. A Review of Defects in Vat Photopolymerization Additive-Manufactured Ceramics: Characterization, Control, and Challenges. *J. Eur. Ceram. Soc.* **2024**, *44* (3), 1361–1384.

(36) Smith, M. W.; Jordan, K. C.; Park, C.; Kim, J.-W.; Lillehei, P. T.; Crooks, R.; Harrison, J. S. Very Long Single- and Few-Walled Boron Nitride Nanotubes via the Pressurized Vapor/Condenser Method. *Nanotechnology* **2009**, *20* (50), 505604.

(37) Huang, W.; Wu, Y.; Chen, W.; Chen, H.; He, J.; Yan, J.; Chen, C.; Song, Y.; Ji, H.; Xu, H. Reinforcement Effect in Printing Precision and Sintering Performance for Liquid Crystal Display Stereolithography Additive Manufacturing of Alumina Ceramics. *Ceram. Int.* **2022**, *48*, 33809.

(38) Mussler, B.; Swain, M. V.; Claussen, N. Dependence of Fracture Toughness of Alumina on Grain Size and Test Technique. *J. Am. Ceram. Soc.* **1982**, *65* (11), 566–572.

(39) Xu, X.; Zhou, S.; Wu, J.; Zhang, C.; Liu, X. Inter-Particle Interactions of Alumina Powders in UV-Curable Suspensions for DLP Stereolithography and Its Effect on Rheology, Solid Loading, and Self-Leveling Behavior. J. Eur. Ceram. Soc. **2021**, *41* (4), 2763–2774.

(40) Liu, J.; Li, Q.; Huo, M.; Zhang, X.; Yue, X.; Liang, J.; Li, J. Microstructure and Mechanical Properties of 3D-Printed Nano-Silica Reinforced Alumina Cores. *Ceram. Int.* **2022**, *48* (20), 30282–30293.

(41) Zhou, S.; Liu, G.; Wang, C.; Zhang, Y.; Yan, C.; Shi, Y. Thermal Debinding for Stereolithography Additive Manufacturing of Advanced Ceramic Parts: A Comprehensive Review. *Mater. Des.* **2024**, *238*, 112632.

(42) Xia, Z.; Riester, L.; Curtin, W. A.; Li, H.; Sheldon, B. W.; Liang, J.; Chang, B.; Xu, J. M. Direct Observation of Toughening Mechanisms in Carbon Nanotube Ceramic Matrix Composites. *Acta Mater.* **2004**, *52* (4), 931–944.

(43) Wang, W.; Li, Z.; Prestat, E.; Hashimoto, T.; Guan, J.; Kim, K. S.; Kingston, C. T.; Simard, B.; Young, R. J. Reinforcement of Polymer-Based Nanocomposites by Thermally Conductive and Electrically Insulating Boron Nitride Nanotubes. *ACS Appl. Nano Mater.* **2020**, 3 (1), 364–374.

(44) Anjum, N.; Alsmairat, O. Q.; Liu, Z.; Park, C.; Fay, C. C.; Ke, C. Mechanical Characterization of Electrospun Boron Nitride Nanotube-Reinforced Polymer Nanocomposite Microfibers. *J. Mater. Res.* **2022**, 37 (24), 4594–4604.

(45) Chang, H.; Lu, M.; Arias-Monje, P. J.; Luo, J.; Park, C.; Kumar, S. Determining the Orientation and Interfacial Stress Transfer of Boron Nitride Nanotube Composite Fibers for Reinforced Polymeric Materials. *ACS Appl. Nano Mater.* **2019**, *2* (10), 6670–6676.

(46) Gudlur, P.; Boczek, A.; Radovic, M.; Muliana, A. On Characterizing the Mechanical Properties of Aluminum–Alumina Composites. *Mater. Sci. Eng., A* **2014**, *590*, 352–359.

(47) Zhao, Y.; Chen, X.; Park, C.; Fay, C. C.; Stupkiewicz, S.; Ke, C. Mechanical Deformations of Boron Nitride Nanotubes in Crossed Junctions. J. Appl. Phys. **2014**, 115 (16), 164305.

(48) Chen, X.; Zhang, L.; Park, C.; Fay, C. C.; Wang, X.; Ke, C. Mechanical Strength of Boron Nitride Nanotube-Polymer Interfaces. *Appl. Phys. Lett.* **2015**, *107* (25), 253105.

(49) Yamakov, V.; Park, C.; Kang, J. H.; Chen, X.; Ke, C.; Fay, C. Piezoelectric and Elastic Properties of Multiwall Boron-Nitride Nanotubes and Their Fibers: A Molecular Dynamics Study. *Comput. Mater. Sci.* **201**7, *135*, 29–42.

(50) Deng, L.; Eichhorn, S. J.; Kao, C.-C.; Young, R. J. The Effective Young's Modulus of Carbon Nanotubes in Composites. *ACS Appl. Mater. Interfaces* **2011**, 3 (2), 433–440.

(51) Ghobadi, H.; Nemati, A.; Ebadzadeh, T.; Sadeghian, Z.; Barzegar-Bafrooei, H. Improving CNT Distribution and Mechanical Properties of MWCNT Reinforced Alumina Matrix. *Mater. Sci. Eng., A* **2014**, *617*, 110–114.

(52) Ghasemi Hajiabadi, M.; Zamanian, M.; Souri, D. Williamson-Hall Analysis in Evaluation of Lattice Strain and the Density of Lattice Dislocation for Nanometer Scaled ZnSe and ZnSe:Cu Particles. *Ceram. Int.* **2019**, *45* (11), 14084–14089.

(53) Mohammed, S. M. A. K.; Paul, T.; John, D.; Zhang, C.; Agarwal, A. Understanding the Role of Ultrasonic Cavitation Assisted Casting of Boron Nitride Nanotube-Reinforced Aluminum Matrix Composite. J. Mater. Res. Technol. **2023**, 25, 2405–2418.

(54) Pande, C. S.; Cooper, K. P. Nanomechanics of Hall–Petch Relationship in Nanocrystalline Materials. *Prog. Mater. Sci.* 2009, 54 (6), 689–706.

(55) Li, Z.; Xiong, M.; Lei, J.; Li, J.; Chen, Y.; Zhang, S. Effect of Mo Doping on the Microstructures and Mechanical Properties of ZnO and AZO Ceramics. *Ceram. Int.* **2021**, *47* (21), 30563–30571.