



Process-Microstructure-Property Correlation in Vacuum Sintered Al-Al₂O₃ Functionally Graded Pellets

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Article's Information	Abstract
Received: 08.02.2026 Accepted: 29.07.2026 Published: 15.09.2026	Aluminum-alumina (Al-Al ₂ O ₃) functionally graded materials are attractive for components requiring a compliant metallic base and a hard ceramic rich surface. However, their performance is strongly limited by transition layer porosity and incomplete densification during low temperature powder metallurgy. In this study, a five-layer Al-Al ₂ O ₃ graded pellet was fabricated by sequential powder stacking, planetary ball milling of mixed layers, low pressure uniaxial compaction at 20 MPa, and vacuum solid-state sintering at 580 °C. The designed solid phase volume fractions from bottom to top were 100/0, 75/25, 50/50, 25/75, and 0/100 Al/Al ₂ O ₃ , with a nominal pellet diameter of 20 mm and layer thickness of 2 mm. Particle size distribution, X-ray diffraction (XRD), scanning electron microscopy, image-based porosity, Archimedes bulk density, Vickers microhardness, compression, flexural, and sliding wear tests were used to establish the process-microstructure-property relationship. XRD confirmed face centered cubic aluminum and α-Al ₂ O ₃ as the dominant crystalline phases, with no obvious crystalline reaction products after sintering. The pellet retained macroscopic integrity, but porosity varied non-linearly across the gradient, reaching 9.12%, 15.26%, 10.26%, 16.23%, and 15.36% from Layers 1 to 5. The bulk density measured was 2.88 g cm ⁻³ . The microhardness of the Al layer was raised significantly with the Al ₂ O ₃ layer, from 44.6 HV0.1 to 703.0 HV0.1. In addition, the specimen exhibited Compressive strength of 138.24 MPa, Flexural strength of 34.03 MPa and Mean Coefficient of friction of 0.278 ± 0.030. The results indicate that low pressure powder stacking can produce a stable Al-Al ₂ O ₃ hardness gradient but the main limitation, which needs improvement, is the porosity in the transition layer.
Keywords: Aluminum, Alumina, Functionally graded material, Metallurgical characterization.	

<http://doi.org/10.22401/ANJS.29.3.13>

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1. Introduction

Functionally graded materials (FGMs) can be made to have a gradual composition and property variation over a component. This enables to tailor the stiffness, hardness, wear resistance, thermal stability, and damage tolerance in a spatial manner in one single body [1]. The use of this graded architecture is beneficial for metal-ceramic systems because of the reduction in sharp interfaces between metal and ceramic that create a mismatch in properties. The

fact that graded structures have been demonstrated to spread mismatch strains over a finite transition zone and to limit the likelihood of catastrophic failure at the interface [2, 3] are the reasons for the choice of graded structures. In Al-Al₂O₃ systems, Al side is soft and compliant while Al₂O₃ side is abrasion resistance and highly hard. Thus, not only is it necessary to obtain a compositional gradation, but also to maintain the integrity of the interlayer, the stability of the phase and a controlled distribution of defects

in the functional area. The balance between the ability to maintain an oxide film and the ability to reestablish metallic contact is the key to controlling densification in Aluminum based powder metallurgy [4]. The initial Al_2O_3 film on the Al powder hinders direct Al-Al bonding, slows down neck formation and prevents solid state densification [5, 6]. To address this limitation several approaches have been reported, such as liquid bonding with gallium bridges [7, 8] and super solidus or liquid phase sintering to enhance rearrangement and filling of pores [9]. Other approaches have also been explored, such as surface modification by nitrogen to modify interfacial films [10, 11] and consolidation processes based on deformation that compel intimate contact among the particles [12]. These techniques can, however, add new constraints, such as reaction product, liquid related embrittlement, changed interfaces, or loss of the sharpness of the gradient. For layered metal-ceramic FGMs, the problem is more complicated, as layers are densifying at different rates [13–16]. The addition of ceramic increases the hardness by load transfer and plasticity constraint [17] while the remaining porosity reduces the load bearing area and may affect tensile, impact, cyclic, or wear properties [18, 19]. In low temperature Al- Al_2O_3 graded compacts is not simply whether a hardness gradient can be produced, but whether the transition layers can densify without retaining porosity and interfacial defects. Although Al- Al_2O_3 graded materials have been reported previously, many studies emphasized layer formation, hardness variation, or general microstructural characterization. Less attention has been given to the coupled relationship between powder size distribution, phase stability, layer-wise porosity, and hardness gradient. The novelty of this study is therefore the systematic assessment of metallurgical and mechanical characterization Al-

Al_2O_3 pellet fabricated using sub-solidus processing route. This approach allows the densification limitation of compositionally mixed layers to be examined without masking it through aggressive consolidation. The objective is to fabricate an Al- Al_2O_3 pellet using vacuum solid-state route. Evaluate the designed composition gradient phase stability, porosity, density, hardness, compression, flexural, wear variations across the graded thickness. The central hypothesis is that a low-pressure powder stacked Al- Al_2O_3 architecture can preserve phase stability and produce a measurable hardness gradient. However, the mixed transition layers will show non-linear porosity because Al_2O_3 restricts particle rearrangement, interrupts Al-Al contact, and limits pore closure during sub-solidus sintering. Accordingly, the fabricated pellet is characterized using particle size distribution, XRD, SEM, image-based porosity, Archimedes density, and layer-wise microhardness to establish the processing-microstructure-property relationship.

2. Materials and Methods

2.1. Raw materials and layer-wise composition design

Aluminum (Al; CAS No. 7429-90-5; 99.7% purity) and alumina (Al_2O_3 ; CAS No. 1344-28-1; 99.9% purity) powders purchased from Kemphasol, India was used as metal as well as ceramic constituents, respectively. A five layer functionally graded architecture was designed with target solid-phase fractions of $\text{Al}/\text{Al}_2\text{O}_3=100/0$, 75/25, 50/50, 25/75, and 0/100 (bottom to top). The actual powder mass used for each layer and the corresponding Al and Al_2O_3 masses are reported in Table 1. The Al- Al_2O_3 pellet architecture from bottom to top architecture with 2 mm layer thickness and 20 mm diameter is illustrated in figure 1.

Table 1: Ceramic composition for each layer of the specimens

Layer	Mass Al (g)	Mass Al_2O_3 (g)	Total layer mass (g)
1	1.696	0.000	1.696
2	1.272	0.621	1.893
3	0.848	1.241	2.089
4	0.424	1.862	2.286
5	0.000	2.482	2.482

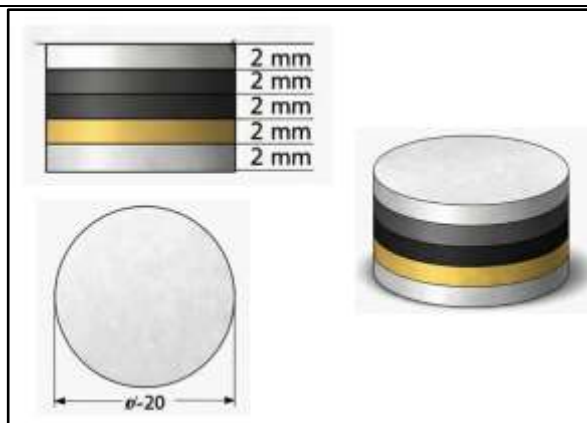


Figure 1. The architecture of Al-Al₂O₃ pellet from bottom to top architecture with 2 mm layer thickness and 20 mm diameter

2.2. Powder size characterization

Particle size distribution (PSD) of Al and Al₂O₃ powders as received was estimated using Mastersizer 2000, Malvern Panalytical laser diffraction with Scirocco 2000 dry dispersion unit. The analysis was performed in volume mode over 0.02-2000 μm. For dry dispersion, the Scirocco compressed air pressure was set within the recommended operating window. Each powder was measured three times, and the reported PSD descriptors (Dv10, Dv50, Dv90 and span) represent the mean of repeated measurements.

2.3. Powder mixing and homogenization

Both powders were dried at 80 °C for 2 h prior to weighing and then cooled in a desiccator. The dried powders were then sieved through a 100-mesh sieve to minimize soft agglomerates and make the product more uniform. In the case of compositionally mixed layers (Layers 2-4) Al and Al₂O₃ powders were weighed as per Table 1 and homogenized in a planetary ball mill (Pulverisette 6, Fritsch). Agglomeration was minimized and dispersive mixing was encouraged by mixing at 200 rpm for 1 h in ethanol. The tungsten carbide (WC) jar and WC balls used were 250 mL and 8 mm respectively with ratio ball:powder equal to 10:1, and continuous milling. The ethanol addition was fixed at 30mL/batch. The slurry was sieved after drying at room temperature until constant mass after milling before compaction.

2.4. Layer stacking and compaction of graded pellets

The green compacts were made in the form of cylindrical pellets (diameter 20 mm) of five layers for functional grading. The powders were introduced into the die (WC-Co, inner diameter 20 mm) in the intended sequence (bottom to top), and the powders were leveled to reduce the thickness variations and then consolidated by uniaxial die compaction to the

desired pressure of 20 MPa. Compaction was done in a 40/PR-6/PM-14/99-2000 uniaxial hydraulic press of Peeco Hydraulic Pvt. Ltd., India, 25 ton capacity. The layers were lightly tamped at about 3 MPa each before adding the next layer of powder to keep the surface stable and minimize disturbance to the powder. All five layers were then stacked and the pellet finally compacted at 20 MPa for 60 s. Only a small amount of zinc-based lubricant was used just on the die wall to prevent ejection. No lubricant was added to the powder mixture, and visible residue was removed by gentle wiping before sintering. The green compact retained a cylindrical geometry of approximately 20 mm diameter and 10 mm total thickness, corresponding to the nominal five-layer design of about 2 mm per layer. The compaction pressure of 20 MPa was selected as a low-pressure condition to preserve the designed layer sequence during powder stacking and final consolidation. Although higher pressures can improve green density, they may also cause layer distortion, lateral powder flow, interlayer smearing, and cracking related to ejection in graded Al-Al₂O₃ compacts with different layer compressibility. Consequently, lower green density and residual porosity were expected, particularly in the mixed and rich ceramic layers. 20 MPa was sufficient to retain a stable graded pellet geometry, but not sufficient to fully densify the Al-Al₂O₃ transition layers.

2.5. Vacuum sintering

Sintering was conducted in a vacuum furnace (KAYKAY, India) evacuated using a rotary vane pump, with temperature monitored by a chamber thermocouple. The heating schedule comprised 5 °C/min to 580 °C, for 1 h, which was cooled to ambient temperature within the furnace. The photograph of sintered Al-Al₂O₃ sample is shown in figure 2. Layer 5 was alumina-rich cold compacted layer, which didn't undergo actual sintering at 580 °C. Its integrity was maintained by mechanical constraint and interfacial anchoring from adjacent layers and by the overall stacked geometry.



Figure 2. The photograph view of Al-Al₂O₃ sample with 20 mm diameter.

2.6. Metallographic preparation - microstructural characterization

Sintered pellets are sectioned to expose a transverse cross section through the full graded thickness. Specimens were ground and surface polished in compliance with metallographic preparation practice (ASTM E3). Microstructural observation and interface assessment were performed using scanning electron microscopy (SEM; Zeiss SmartEDX SIGMA 300) at 30 kV in backscattered electron (BSE) mode to maximize compositional contrast between Al and Al₂O₃ regions.

2.7. Phase analysis (XRD)

Phase constitution of the sintered material was examined using X'Pert PRO, PANalytical X-ray diffraction (XRD) with Cu-K α radiation. The layers were not physically separated for XRD. Instead, the sintered pellet was sectioned and polished to expose the graded cross-section, and surface scans were performed on representative regions corresponding to each designed layer. The incident beam was positioned on the exposed layer surface and diffraction patterns were acquired over $2\theta=20-90^\circ$ at $1^\circ/\text{min}$ scan rate. Phase identification was performed using Match! Software.

2.8. Microhardness profiling across layers

Layer-wise microhardness was measured on polished cross sections using a Vickers microhardness tester under a 0.1 kgf load (HV0.1) with 10 s dwell time. Furthermore, indentations were placed at the mid thickness of each layer, maintaining sufficient spacing between indents and from layer interfaces/edges to avoid interaction of plastic zones. For each layer, three indents were performed and the layer hardness was reported using the mean and dispersion of readings, enabling comparison of hardness gradients.

2.9. Porosity quantification by image analysis

Porosity was assessed using polished cross-sectional micrographs with stereology image analysis consistent with ASTM E1245. For each layer, at least three representative fields of view were analyzed. Pores were segmented by grayscale thresholding to ensure rigor and interlayer comparability. A visually validated threshold was selected and then held constant across all layers while filtering isolated noise pixels. The measured 2D pore area fraction was reported as a proxy for porosity for comparative, layer-wise assessment.

2.10. Bulk density

Bulk density of sintered graded pellet was estimated adopting Archimede's principle as per ASTM B962. The specimens were weighed in both air and fluid

(room temperature distilled water). For samples with porosity, surface connected porosity was sealed after impregnation before the immersion weighing, as done in ASTM B962 for porous powder metallurgy products. The image analysis porosity is a 2D measurement of the area fraction of pores, obtained from polished cross section SEM fields, while the Archimedes method is a bulk volumetric density response to the entire graded pellet. The two methods were thus used in complementary way: image analysis for comparison of layer-wise porosity variation, Archimedes density for evaluation of the overall densification state of the pellet. The sources for the uncertainty of the porosity measurement in images are primarily the field being selected, the thresholding, polishing artefacts, and local microstructural heterogeneity. Therefore, porosity values were reported as mean values from representative fields, and the comparison with Archimedes density was treated as a consistency check rather than a direct one-to-one equivalence.

2.11. Compression, flexural and wear tests

Compression testing was carried out on the samples using a universal testing machine (TESTTEK EM-100, TESTTEK, India) with a 100 kN load cell following ASTM E9 standard. The pellets that have been sintered were put in the middle between the platens and compressed at a controlled displacement loading. This was done at a constant crosshead speed of 1 mm/min. Flexural properties were measured using three point bending specimens conforming to ASTM D790 standard. Friction and sliding wear tests are conducted following ASTM G99 standard using Ducom Pin-on-Disc Tribometer, with an applied load of 10 N and the specifications of the machine are presented in Table A2 in the Appendix.

3. Results and Discussion

3.1. Macroscopic integrity and dimensional outcome after sintering

Figure 2 shows the as-sintered pellet as a geometrically stable cylinder (nominal diameter is 20 mm), with a flat top surface and no obvious macroscopic warpage. The exposed face appears continuously and largely crack-free at the macroscopic scale, indicating that the layered stack survived the thermal cycle as an integrated body rather than separating into discrete laminates. The darker circumferential band observed at the sidewall is attributed to a surface condition change produced during vacuum sintering and subsequent cooling. This change may arise from a combination of altered surface roughness after die wall contact, thickening of local oxide film on exposed rich Al regions from the

minimal die-wall lubricant. The sidewall feature is interpreted as a superficial surface effect rather than evidence of bulk layer separation or internal cracking. Recent studies showed that atmosphere selection can exert a decisive influence on densification and porosity evolution during sintering, with knock-on effects on shrinkage and defect formation [20]. FGM design principles emphasized that a compositional gradient, rather than a sharp bi-material interface, can reduce abrupt mismatch stresses and improve structural integrity under thermally driven mismatch, even when the gradient is implemented as a stepwise architecture [21]. Layer 5 didn't undergo actual sintering during 580 °C. Therefore, the pellet is not interpreted as a fully densified FGM. The Al-rich and mixed layers experience limited solid state bonding, while the terminal alumina-rich layer remains primarily a compacted ceramic layer. Retention after sintering shows mechanical stability at the specimen scale, which is reinforced by compaction, and geometric confinement, and by the anchoring with Layer 4. The adhesion between Layer 4 and Layer 5 is to be considered as secondary to a fully sintered ceramic interface. This limits the direct use of the present structure in high demands for load bearing and/or thermal shock to a certain extent without providing alternative consolidation methods like higher compaction pressure, hot pressing, spark plasma sintering or infiltration after sintering. Thus, the present work is positioned as a feasibility and microstructural study of low-pressure powder stacking and vacuum solid-state treatment, rather than as a fully optimized structural FGM.

3.2. Powder particle size distribution and implications for mixing

The repeatability of the measurements was verified from the overlap of repeated PSD curves and the consistency of Dv10, Dv50, and Dv90 values. Measurement accuracy was taken according to the instrument specification of the Mastersizer 2000 system. Laser diffraction PSD (volume basis) showed that the Al powder (figure 3) is finer and broader, whereas Al₂O₃ (figure 3) is coarser with a narrower distribution. Al exhibits 8.261, 25.427 and 66.964 µm for Dv10, Dv50, and Dv90, respectively, with span=2.309 and D[4,3]=32.698 µm. In contrast, Al₂O₃ showed 17.893, 45.414, 95.018 µm for Dv10, Dv50, Dv90, respectively, with span=1.698 and D[4,3]=51.788 µm. The size contrast (Al being ~0.56× the median size of Al₂O₃) implies that, in mixed layers, Al particles can partially occupy interstitial spaces created by the larger ceramic particles, while

the broader Al distribution supplies an additional fine fraction capable of filling smaller voids that would otherwise persist in a narrow PSD packing. The pairing of PSD has direct implications for the green packing density, pore topology, and further up to sintering shrinkage compatibility. Previous research on powder consolidation demonstrated that using a wide PSD (narrow and broad) can lead to higher packing density and fewer largely connected pores, which results in fewer shrinkage and shape instability issues after sintering [22]. The fine fraction occupies the voids between the coarse particles and reduces the size/coordination of predominant pores [23]. The total powder mass was 10.446 g and the nominal green compact volume was 3.142 cm³ for a 20 mm diameter, 10 mm thickness pellet, the overall green density was estimated to be around 3.33 g.cm⁻³. But this is an average green-density of the total compact and is not of the layers. Because the thickness of each layer of the system after sequential stacking and final consolidation could not be determined, layer-wise green density was not measured. The packing efficiency can be affected by the particle size ratio and distribution width because smaller particles can fill the void space between larger particles and insufficient mixing or cement-rich clusters can result in localized pores [22, 23]. Pore architecture and packing efficiency change with composition due to the change-over from the metal region to the ceramic region that is controlled by the packing network. Even if there is no macroscopic damage to the pellet, the densification kinetics and pathways of the pore closure of adjacent layers with different dominant particle regions (Al-rich vs. Al₂O₃-rich) will differ. This can show up on a micro-scale as interfacial pore strings or as weak transition zones on a localized basis. The PSD measurements were performed on the as-received Al and Al₂O₃ powders, and the PSD of the ball milled mixed powders was not separately measured. Therefore, possible induced milling particle breakage, mild flattening of Al particles, or deagglomeration of Al₂O₃ cannot be fully quantified from the present PSD data. The PSD interpretation is consequently used as an initial powder packing indicator rather than a direct measurement of the post-milling mixed powder state. This limitation is important because any induced milling change in particle size or morphology may influence green packing, interparticle contact, and pore closure during sintering. The implications of this initial PSD mismatch are therefore discussed together with the layer-wise porosity results in Section 3.5.

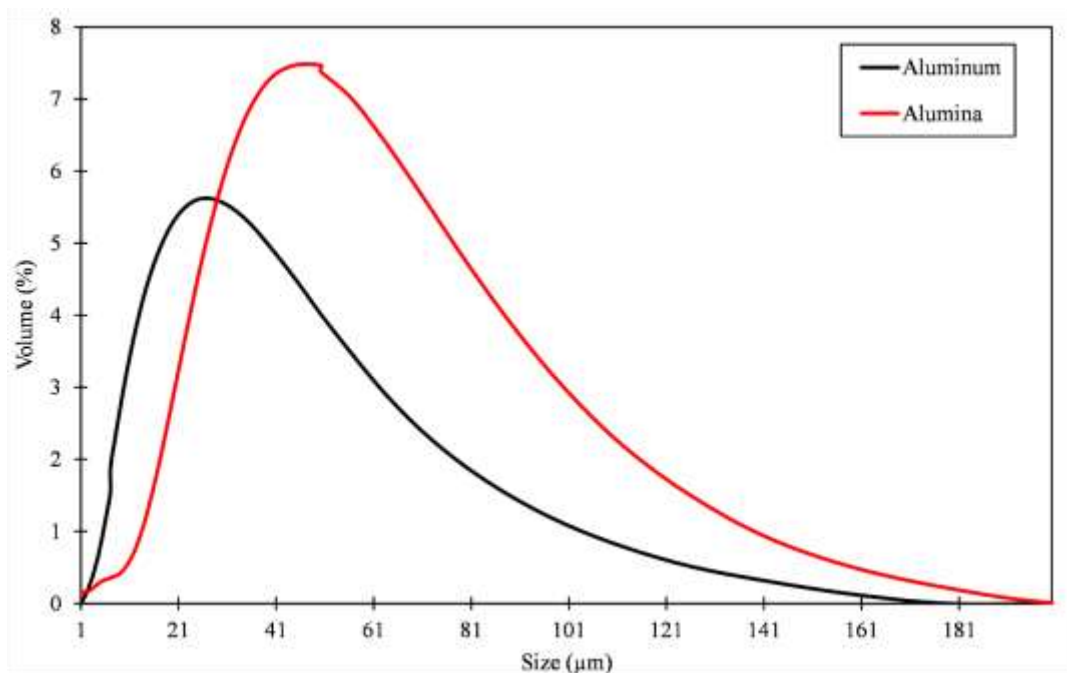


Figure 3. The PSD alumina and aluminum, measured using laser diffraction

3.3. Phase constitution after vacuum sintering (XRD)

Figure 4 presents the combined XRD patterns of all five layers to allow direct comparison of phase evolution across the graded thickness. The layer-wise XRD patterns confirm that the phase constitution remains chemically conservative across the gradient, i.e., dominated by face centered cubic (FCC) Al and α - Al_2O_3 , with no obvious emergence of new reaction products after the 580 °C vacuum sintering cycle. In Al/ Al_2O_3 FGM fabricated by layered compaction, the property gradient (hardness/porosity trends) is driven primarily by constituent fraction and densification/void evolution, rather than by unintended interfacial chemistry [24]. Layer 1 (pure Al) shows the fingerprint of FCC aluminum, with strong reflections at approximately $2\theta=38.3^\circ$, 44.5° , 64.4° , 78.8° , and 82.3° , consistent with the (111), (200), (220), (311), and (222) planes of Al. The absence of extra crystalline peaks is consistent with the fact that vacuum sintering at 580 °C is much lower to Al melting point (660 °C), and therefore the matrix largely undergoes solid state necking/limited densification rather than liquid phase evolution [25]. From Layer 2 to Layer 4, the diffraction patterns progressively develop the characteristic α - Al_2O_3 peak set (reflections in the vicinity of 25 – 26° , 35° , 43° , 52 – 53° , 57 – 58° , 66 – 69° , and 77 – 83°). The Al peaks are still visible and strong with a two-phase physical composite microstructure with compositional gradient. With increasing Al_2O_3 content, the number

and the intensity of the alumina peaks increased, and the Al peaks remain since the alumina content is still sufficiently high to leave a continuous metallic phase in the Al-rich and intermediate layers. This is consistent with the results of the composite processing studies conducted by Zabihi et al. [26] where the two parent materials are the major crystalline phases. In layer 5, the peak positions match the alumina signatures that appear inside Layers 2-4, supporting that alumina remains structurally stable through the cycle. This stability is entirely possible at 580 °C under vacuum. The thermal exposure is sufficient to promote diffusion bonding and stress relaxation at particle contacts, but not a strong driver for forming new crystalline reaction phases in an Al/ Al_2O_3 ex-situ system [27]. This trend is consistent with the designed compositional gradient; however, it should be interpreted semi-quantitatively because peak intensity is also affected by surface condition, local texture, scanned area, sample height, and layer-wise heterogeneity. Nevertheless, amorphous oxide-rich regions, changes in the native surface oxide on Al particles, or very thin interfacial reaction layers may remain below the XRD detection limit. Therefore, the XRD results confirm the dominant crystalline phases and qualitative phase stability. Al powders inherently carry a native Al_2O_3 surface film that can persist and impede interparticle diffusion. The XRD analyses are consistent with a densification pathway that is dominated by oxide limited solid state bonding

and ceramic constraint, rather than extensive metallurgical reaction [25]. The main peak positions (2θ , Cu K α) by layer with phase assignment are given in Table 2. The identified phases were identified using the reference patterns of FCC Al (ICDD/JCPDS PDF No. 04-0787) and α -Al₂O₃/corundum (ICDD/JCPDS PDF No. 46-1212). The main Al and α -Al₂O₃ reflections were near their standard positions over the graded layers, and no systematic peak shift was detected from the peak list measured. Thus, no significant lattice distortion or induced residual stress peak shifting is suggested by the XRD results. However, small peak shifts cannot be fully ruled out because they may also arise from residual microstrain, sample height error, instrumental

broadening, or local thermal mismatch stresses. Since lattice parameter refinement or residual stress analysis was not performed, residual stresses are discussed only qualitatively. Similarly, the absence of additional peaks indicates that no major crystalline reaction products formed during the 580 °C vacuum sintering cycle. Nevertheless, very thin interfacial reaction layers, amorphous rich oxide regions, or minor phases below the XRD detection limit may not be detected by conventional XRD. Quantitative analysis of phases was not performed therefore, the phase interpretation is limited to qualitative phase identification and relative peak intensity comparison across layers.

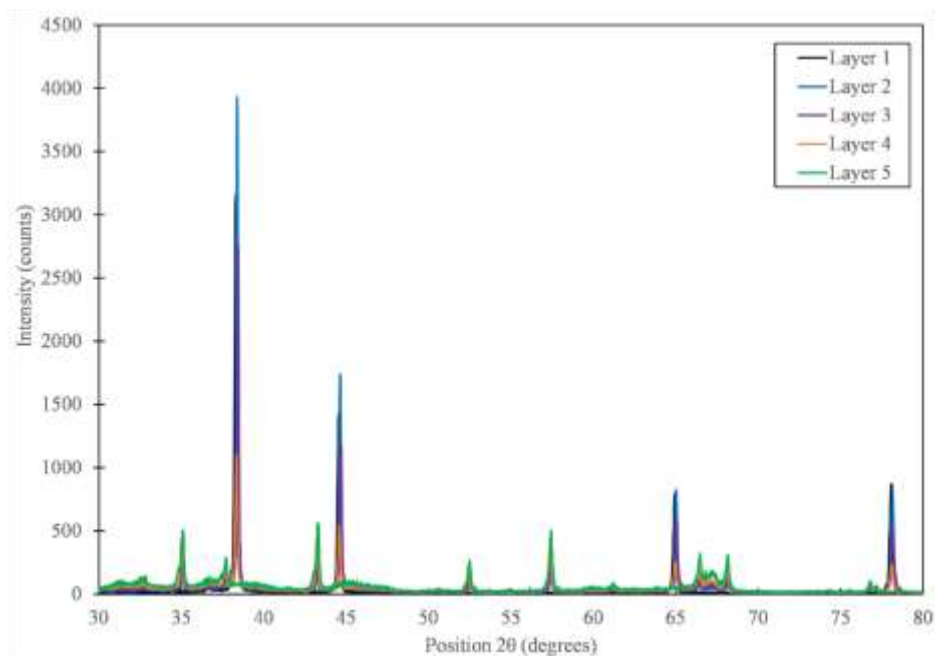


Figure 4. X-ray diffraction pattern of Al-Al₂O₃ for Layers 1-5

3.4. Cross-sectional microstructure across the graded thickness

Cross-sectional SEM images of Layers 1-5 are shown in figure 5(a-e). A systematic change in phase morphology is evident across the graded structure: the Al-rich continuous matrix observed in the initial layer gradually gives way to a microstructure increasingly dominated by Al₂O₃ particles. Layer 5, however, is not interpreted as a conventionally sintered alumina body because the applied vacuum sintering temperature of 580 °C is insufficient for substantial solid-state sintering of Al₂O₃. Its morphology is more appropriately described as that of a compacted, alumina-rich region whose structural stability is maintained through particle interlocking, interfacial attachment and mechanical constraint

imposed by the neighbouring graded layers. No open gaps, extensive delamination or complete separation of the constituent layers can be identified within the examined fields. The multilayer structure therefore appears to have retained its continuity during sintering and subsequent metallographic preparation. This observation agrees with reports on powder-metallurgy-processed FGMs, in which individual compositional layers can remain distinguishable after sintering while developing adequate interlayer cohesion through particle necking and an enlarged contact area [27]. Several isolated voids and pore-like features, together with a few elongated discontinuities, are visible, particularly in figure 5(a). The relatively small and rounded pores within the Al-rich matrix are

characteristic of residual porosity in solid-state-sintered aluminium compacts. During heating, particle-neck formation and progressive pore rounding contribute to densification [28]. Nevertheless, complete pore closure may be hindered by the persistent oxide films surrounding Al powder particles and by the limited diffusion occurring under the selected sintering conditions [29]. A less uniform morphology is observed in the intermediate layers shown in figure 5(b-d). The higher the Al_2O_3 content, the more ceramic particles prevent the rearrangement of metal particles and interrupt diffusion paths which result in the reduction of internal void closure ability of the compact [30]. The resulting microstructural heterogeneity is accordingly consistent with the design of the intended compositional gradient, with the Al rich layers having a comparatively uniform and continuous ceramic rich matrix, while the transition layers have a more complex distribution of ceramic particles, interparticle boundaries and pore bearing regions. The compositional interfaces shown in Layers 2-5 (as depicted in figure 5(b-e)) have a more continuous appearance, which is typical of transition regions rather than interfaces that are even with an opening. Such behaviour is in line with studies on powder metallurgy showing that graded interfaces may remain visually identifiable while still being mechanically and metallurgically integrated when adequate compaction pressure, contact area and sintering conditions are provided. Nevertheless, compositional transitions can become preferential sites for pore accumulation and microcrack initiation because adjacent layers experience unequal

densification and thermal contraction. Jiang et al. [31] associated localized porosity and cracking in graded materials with shrinkage differences and thermal expansion mismatch across neighbouring compositions. Narrow, elongated discontinuities are most apparent in the alumina-rich regions of figure 5(d) and (e). Their formation may be associated with microcracking caused by differential shrinkage and thermally induced stresses during cooling. The likelihood of such damage increases with ceramic content because the material becomes less capable of accommodating local strain through plastic deformation. Some discontinuities, however, may also have arisen during metallographic preparation through particle pull-out, edge chipping or polishing-induced cracking. Features that follow the compositional transition and persist over an appreciable distance aligns with mismatch-induced cracking methodologies previously reported in ceramic-metal graded systems [29][31]. The SEM images were annotated to improve feature identification, including Al-rich regions, Al_2O_3 -rich regions, pores, interfacial/transition zones, and possible discontinuity or pull-out features. These assignments were made based on the designed layer composition, SEM contrast, morphology, and XRD phase constitution. However, since EDS mapping was not performed, the annotations should be interpreted as phase assignments based on morphology rather than direct elemental maps. The elongated discontinuities observed mainly in the rich ceramic layers represent true interfacial microcracking caused by shrinkage mismatch and ceramic brittleness.

Table 2. Key diffraction peaks and phase assignments of the Al- Al_2O_3 graded pellet after vacuum sintering at 580 °C, indexed using FCC Al (ICDD/JCPDS PDF No. 04-0787) and α - Al_2O_3 /corundum (ICDD/JCPDS PDF No. 46-1212)

Layer	Dominant Al (FCC) peaks, 2 θ (°)	Dominant α - Al_2O_3 peaks, 2 θ (°)	Phase call
Layer 1	38.296, 44.547, 64.357, 78.887, 82.265	Not evident at bulk scale	Single-phase Al (FCC) signature
Layer 2	~38.393, 44.633, (Al high intensity retained)	25.482, 35.063, 36.707, 37.678, 43.267, 52.458, 57.407, 65.010, 68.125, 78.142, 82.352	Mixed Al + α - Al_2O_3 , Al peaks dominate; alumina peaks already detectable
Layer 3	~38.441, 44.687, (Al retained)	25.539, 35.118, 37.736, 43.332, 52.516, 57.463, 61.28, 65.060, 66.573, 68.171, 76.84, 78.128, 82.397	Mixed Al + α - Al_2O_3 , stronger alumina contribution than Layer 2
Layer 4	~38.337, 44.583 (Al peaks still visible but less dominant)	25.445, 35.015, 37.673, 43.215, 52.408, 57.359, 61.313, 64.955, ~66–69 cluster, 77.71, 78.090, 80.562, 82.309	Mixed Al + α - Al_2O_3 , alumina dominates; Al persists as residual/ continuous fraction
Layer 5	Not applicable	25.507, (~32.2), (~35.8), 43.275, 52.472, 57.418, 66.85, 68.14, 76.855	Predominantly α - Al_2O_3

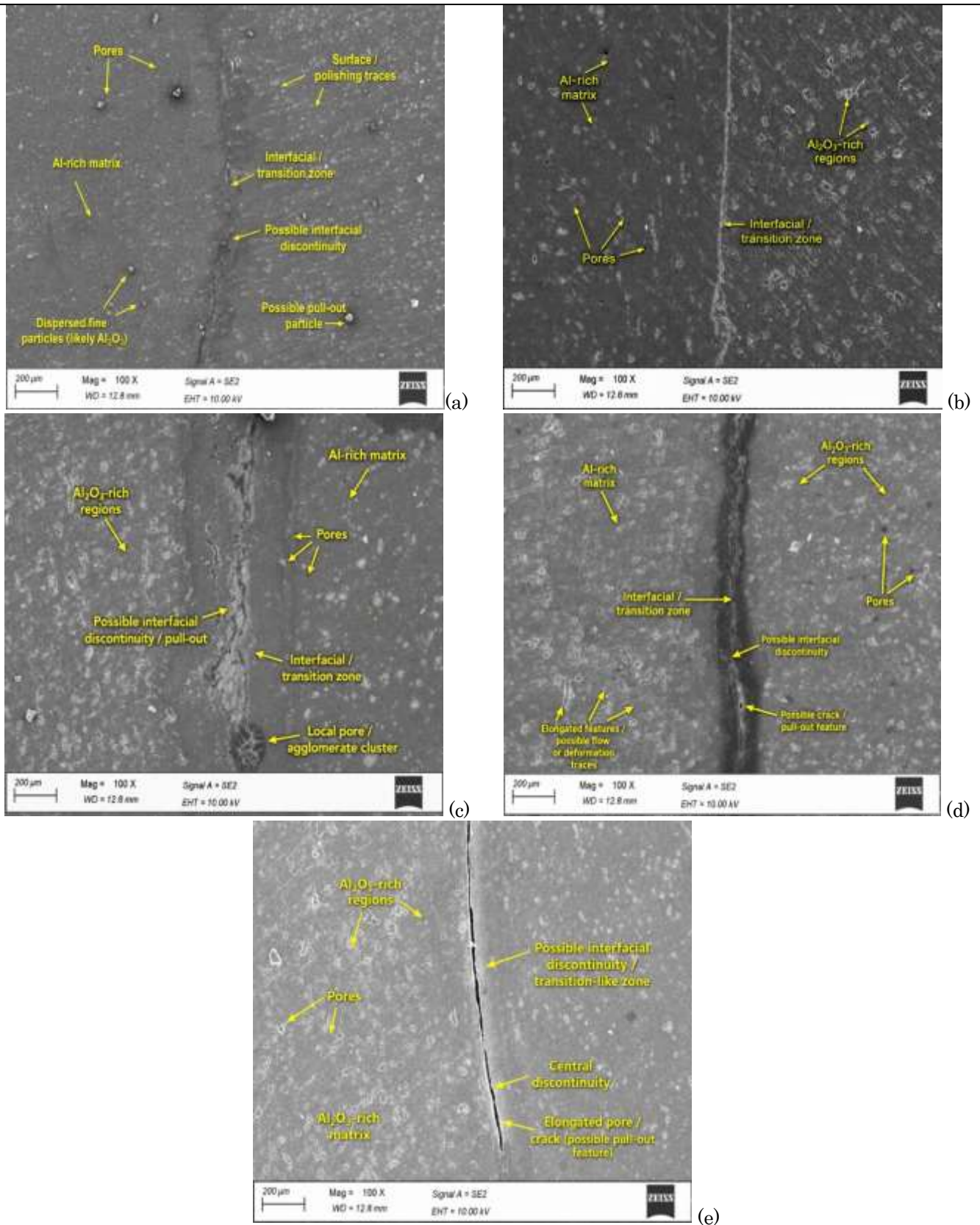


Figure 5. Annotated SEM fractography of Al-Al₂O₃ functionally graded pellet: (a) Layer 1, (b) Layer 2, (c) Layer 3, (d) Layer 4, and (e) Layer 5. The annotations identify Al-rich regions, Al₂O₃-rich regions, pores, interfacial/transition zones, and possible preparation-related discontinuities or pull-out features

3.5. Porosity distribution across layers and its microstructural origin

Porosity varied appreciably across the graded section, with 9.12%, 15.26%, 10.26%, 16.23% and 15.36%, respectively for Layers 1-5. This sequence reveals a non-monotonic distribution rather than a steady increase with Al_2O_3 content. Layer 4 had the highest pore fraction and Layer 3 was significantly lower than the adjacent Layers 2 and 4. Layer 5 was quite porous, but slightly less so than Layer 4. This is because the compaction pressure used was 20 MPa, the comparatively large residual pore fraction could be attributed to this. This is a rather small amount of pressure that would probably not be enough to cause significant rearrangement of particles and plastic deformation, so that the green density was not very high and the interparticle spaces could not be eliminated even before the sintering was begun. Vacuum sintering at 580 °C was characterized by solid-state contact formation, Al particle neck development and limited diffusion. The ability of these mechanisms to function was progressively lowered in the Al- Al_2O_3 transition layers, because the rigid ceramic particles hindered rearrangement of the powder bed and prevented direct Al-Al contact and the continuous metallic pathway available for neck growth. Thus, the resulting pore structure was not only dependent on the nominal composition of each layer, but also on the local packing configuration and compaction response of the layers [32]. The presence of higher levels of Al_2O_3 would be expected to be detrimental to densification as Al_2O_3 does not sinter to a significant extent at 580 °C and is a physical barrier to joining of neighboring Al particles. As a result, the continuity of the metallic phase reduces and the ability to close the pores decreases [30]. Tian et al. [33] also reported that the ceramic reinforcements can slow down the densification of Al matrix composites by blocking diffusion. Layers 2-4 were mixtures of Al and Al_2O_3 while Layer 5 was the final alumina layer. Thus, the maximum porosity in Layer 4 can be correlated with its high ceramic fraction, reduced deformable Al phase continuity and increased rigid ceramic-ceramic contacts. Due to the chosen thermal cycle, significant sintering of Al_2O_3 is not anticipated in this, which mostly preserved the properties of a compacted ceramic powder layer instead of a fully sintered alumina. The integrity was therefore primarily obtained through the process of green compaction and interlocking of the particles and mechanical constraint from the adjacent layers of grading. If dissimilar particle sizes, shapes and mechanical responses are mixed, their packing efficiency can either increase or decrease depending

on the size ratio, spatial distribution and degree of mixing in Layer 3. In the case of a multilayer assembly, this effect can be more complicated due to the inter-layer resistance as well as the variation compliance of deposited powder in between each layer, which is also affected by the die wall friction. The alumina particles were essentially rigid, and compaction was primarily due to particle rearrangement and some amount of plastic deformation of the Al constituent at 20 MPa. There might be residual pores in Layer 2 as the continuous Al particle network has been interrupted by introduced ceramic particles. In the case of Layer 4, ceramic-ceramic interaction was further enhanced by the greater alumina fraction, which further inhibited void closure by deformation. It is believed that the lower measured pore fraction was obtained in Layer 3 due to a relatively good balance between particle filling and metallic phase deformability. The same deviation from the simple rule-of-mixtures trend is also observed for multilayer FGMs and metal matrix composites where the packing and distribution of the reinforcements in the local region control the pore structure [32]. For Al, the relatively high sintering temperature, 580 °C, may still limit the ability for effective densification due to the presence of a stable oxide film on the original Al particles. This surface layer prevents direct contact with the metals, restricts neck formation and prevents the removal of any remaining voids, unless it is disturbed during compaction or processing [34]. Similarly, Liu et al. [35] found the oxide film to be another limitation on the sintering of Al powders and demonstrated that reinforcement can also hinder particle rearrangement and diffusion. It is thought that this is one reason for the 9.12% porosity that Layer 1 maintained after being mixed with the highest proportion of Al, and the larger pore fractions that the ceramic bearing layers retained at the end of the sintering cycle. The quantitative results support the SEM observations as all pores are distributed throughout the graded area and discontinuities are present in certain compositional transitions.

3.6. Density and densification efficiency

The bulk density of complete Al- Al_2O_3 graded pellet, measured by the Archimedes method, was 2.88 g.cm⁻³. Since the layers were not physically separated after sintering, the density of each individual layer was not directly measured. Instead, calculated layer-wise apparent density values were derived from the measured 2D image porosity and theoretical density of mixture for each layer, as shown in equation (2).

$$P(\%) = [1 - (\rho_{exp}/\rho_{th})] \times 100 \quad \dots (1)$$

where $P(\%)$ denoted the % of porosity, ρ_{exp} , and ρ_{th} denotes the experimental and theoretical densities. The nominal Al and Al_2O_3 weight fraction of each layer was used to calculate theoretical density with constituent densities of 2.70 and 3.95 g cm^{-3} , respectively. As the Al_2O_3 content of the mixture was increased, the calculated mixture density was increased monotonically. The higher density ceramic phase would improve the theoretical density of the Al- Al_2O_3 mixture, but the rigidity of the ceramic phase may limit the ability to rearrange the powder

and form a metallic neck and thus cause a lower level of densification during sintering [36]. The porosity of each layer was then measured, and the bulk density of each layer was estimated from the measured porosity by applying the equation (3) and the relative density of each layer was estimated by applying the equation (4).

$$\rho_{bulk,layer} = (1 - P/100)\rho_{th,layer} \quad \dots (2)$$

$$\rho_{rel} = \rho_{bulk,layer}/\rho_{th,layer} \quad \dots (3)$$

Table 3 illustrates the theoretical density, bulk density and densification efficiency of Al- Al_2O_3 graded pellet layer wise.

Table 3. Theoretical density, calculated apparent density, and relative density indicators of the Al- Al_2O_3 graded pellet after vacuum sintering.

Layer	Layer composition (Al/ Al_2O_3) (wt.%)	Porosity, $P(\%)$	Theoretical density, ρ_{th} (g cm^{-3})	Calculated apparent density, ρ_{bulk} (g cm^{-3})	Relative density, ρ_{rel} (%)
1	100/0	9.12	2.7	2.454	90.88
2	67.22/32.78	15.26	3.012	2.553	84.74
3	40.526/59.474	10.26	3.326	2.985	89.74
4	18.557/81.443	16.23	3.637	3.047	83.77
5	0/100	15.36	3.95	3.343	84.64

The theoretical density was found to increase with the addition of Al_2O_3 . However, the relative density decreased in the mixed and ceramic dominated regions. Layers 2, 4 and 5 thus had lower relative densities than the Al-rich layer, with Layer 4 having the largest deficit in density. This is consistent with the known response of powder metallurgy Al-Ceramic Composites: the rigid reinforcement particles do not allow rearrangement, prevent direct Al-Al contacts and limit available transport mechanisms to effectively eliminate pores [36]. The retained porosity also was due to the sintering temperature. Since the temperature of 580 °C was lower than the melting temperature of Al, the compact was held at a temperature well below the melting point, and the densification was chiefly due to interparticle contact, diffusion and neck development. The vacuum environment did not allow further oxidation to occur but was not expected to easily break up the stable oxide films already on the Al powder surface. The films and the discontinuous transport paths formed by Al_2O_3 particles inhibited metallurgical bonding and pore closure. Higher-temperature processing has been reported to yield liquid phase that yields a more effective densification [36, 37]. For example, Kusunuki et al. [37] achieved relative densities of about 95-97% by using liquid phase formation to promote the bonding of particles and to reduce the voids. The latter cycled solid state

at 580 °C did not offer an equivalent mechanism involving the liquid. So, the residual porosity in the Al- Al_2O_3 mixed layers is consistent with the processing conditions selected. The apparent bulk density of the whole graded specimen was calculated as the volume-weighted average of the calculated density for each layer (equation (5)). The nominal volumes were assumed to be equal, because all five layers were given the same nominal thickness of 2 mm, and the diameter of the pellets was kept constant, so that the result is also a volume weighted result and is the same as the arithmetic mean of the five-layer densities. This is a first-order estimate since the specimen was cylindrical and there was no macroscopic separation of the layers after sintering. However, it is assumed all the layers have the same final thickness in the calculation. It thus does not take into consideration the potential for shrinkage in each layer due to variation in Al content, ceramic fraction, green density or sintering response.

$$\rho_{bulk,overall(pred)} = \sum_{i=1}^5 f_i \rho_{bulk,i} \quad , \quad f_i = 1/5 \quad \dots (4)$$

The calculated value of 2.876 g cm^{-3} is close to the internal consistency between porosity and measured bulk density of 2.881 g cm^{-3} by Archimedes, indicating that the value is reasonable. This agreement, however, is not confirmation of direct layer-wise density since the actual thicknesses of the layers were not measured after the sintering. The

volume-weighted calculated density value is in very good agreement with the experimental density measured by the Archimedes method, which helps to verify the internal consistency of the porosity-based calculation, but is not a direct measurement of the layer density. Some difference between the two methods should be expected, however. SEM image analysis quantifies a cross section and is subject to field of view, pore morphology, thresholding and induced polishing pull-out. By comparison, Archimedes measurement will be the average density of the pellet based on the volume of its bulk, and may be affected by surface connected pores, sealing quality and immersion stability. Thus, the porosity values found from the SEM should be considered comparative layer-wise porosity indicators and Archimedes density should be taken as the densification response that occurs throughout the bulk.

3.7. Hardness gradient across layers

A significant through-thickness Vickers microhardness gradient existed. The mean values increased from 44.6 ± 4.03 HV0.1 in Layer 1 to 83.2 ± 3.90 HV0.1 in Layer 2, followed by 210.2 ± 5.13 , 512.8 ± 6.81 and 703.0 ± 2.34 HV0.1 in Layers 3, 4 and 5, respectively. The hardness increased monotonically,

but not linearly, with the number of layers and the Al_2O_3 content of the layers as shown in figure 6 and summarized in Appendix Table A1. The designed variation in composition resulted in a large gradient in local indentation resistance as shown by the hardness of Layer 5, which was about 15.8 times harder than Layer 1. Residual pores, variation in Al-particle neck development and lack of bonding of the particles due to the presence of persistent surface oxides, may affect the measured response in the Al-rich regions. The hardness of the intermediate layers is more sensitive to which type of indentation they encounter, whether it is the metallic matrix or an Al_2O_3 particle, an agglomerate or a region containing pores. These differences can thus be observed between measurements in the same nominal composition depending on the variations in reinforcement spacing and in the local particle concentration. The behavior of the progressive increase of hardness is in accordance with the behavior of metal-ceramic systems. A similar increase in indentation resistance due to the high intrinsic hardness of ceramic particles, transfer of applied load from the softer matrix to ceramic particles and plastic deformation restriction in metallic phase was also observed by Ehsae et al. [38].

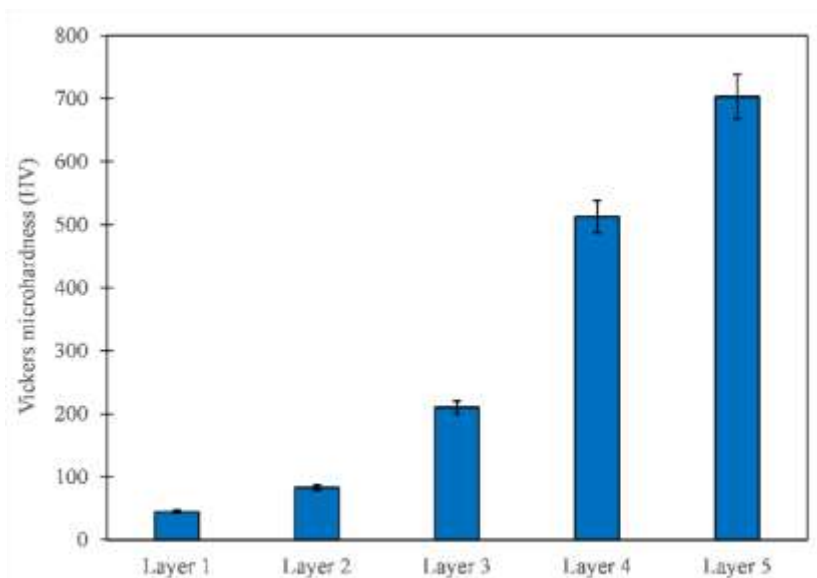


Figure 6. The layer-wise hardness gradient of the sintered Al-Al₂O₃ functionally graded pellet

The microhardness values in the graded section were found to be ascending in the section despite the non-monotonic porosity distribution of the section, which shows local maximum porosities in Layers 2 and 4,

while Layer 3 exhibited lower pores. This separation highlights the fact that there was more of an increase in the indentation resistance with the addition of Al_2O_3 than a decrease due to the residual porosity.

This influence was particularly strong in the outer layer, where the microstructure evolved from an Al based composite with intermittent ceramic particle reinforcement to a more ceramic dominated structure. Yadav et al. [39] also described that, in general, the lower the porosity the lower the hardness, due to the decrease in the effective load bearing area and the increase in the local compliance. However, an increase in the Al_2O_3 fraction can result in an overall increase in hardness due to the increased hardness of the indenter and the increased constraint of the surrounding metallic matrix. The significant increase in hardness values between Layers 2 and 3 and between Layers 3 and 4 suggests that there may have been a change in the local load bearing mechanism. In relatively low reinforcement content, the Al_2O_3 particles still do not coalesce and form a continuous and plastically deformable Al matrix. The size of the indented ceramic fraction decreases as the ceramic fraction increases, and the hard phase increases in importance to bearing the load of the indenter. With this changing morphology, the matrix constraint increases and the plastic flow under the indenter decreases, which results in an intermediate layer of the material exhibiting a nonlinear increase in hardness. This is confirmed by the layer-wise SEM observations. The microstructures of layers 1 and 2 are mostly Al controlled, and the hardness is comparatively low. Layer 3 is the main transition layer that the ceramic phase starts to have a stronger influence on the local indentation response. The resistance to penetration in Layers 4 and 5 depends primarily on the increased amount and consistency of the harder Al_2O_3 phase. The effect of this strengthening is moderated by the presence of porosity, but no overall reversal of the trend towards hardness. Adequate interfacial integrity in Al- Al_2O_3 graded composites also allows for the compositional grading to be transferred into a continuous change in the composition of the Al- Al_2O_3 composite for a graded thickness, as reported by Ramezani et al. [40]. Therefore, present hardness values are consistent with the expected response of Al- Al_2O_3 graded systems, while the local dispersion reflects the combined influence of porosity, ceramic distribution, and phase heterogeneity. One-way ANOVA confirmed that the hardness variation

across the five layers was statistically significant ($F=11211.63$, $p < 0.05$), indicating that the increase in hardness from the Al-rich layer to the Al_2O_3 -rich layer represents a statistically meaningful functional gradient rather than random measurement variation.

3.8. Compression test

The compression test was carried out to give a direct load bearing indication of the sintered Al- Al_2O_3 graded pellet that was beyond the local hardness response indicated by the previous test. The stress-strain relationship of the pellet is shown in Figure 7. The specimen experienced a peak compressive load of 13.6 kN, and a peak compressive stress of 138.24 MPa. This is an indication that the pellet had some structural integrity even after vacuum sintering even though there was some porosity remaining in mixed and rich ceramic layers. The compressive response is consistent with the effect of ceramic reinforcement on load resistance, and the absolute strength is still controlled by the porosity, interparticle bonding, and the continuity of the Al- Al_2O_3 interfaces. Similar recent studies on Al/ Al_2O_3 and ceramic reinforced aluminium composites by Kurzawa et al. [41] and Abdellah et al. [42] have shown that compressive performance is strongly controlled by reinforcement fraction, particle distribution, porosity level, and matrix reinforcement interfacial bonding.

3.9. Flexural test response

The flexural response of the vacuum sintered Al- Al_2O_3 functionally graded pellet was evaluated using three-point bending. The flexural stress-strain response is presented in Figure 8. The specimen recorded a maximum bending load of 179 N, flexural strength of 34.03 MPa, flexural modulus of 570 MPa, and maximum deflection of 11 mm. These results indicate that the graded pellet retained measurable bending resistance after sintering, despite the residual porosity present in the mixed and ceramic-rich layers. The flexural response is mainly governed by the combined effects of ceramic fraction, porosity, inter-particle bonding, and the integrity of the Al- Al_2O_3 transition regions, Yadav et al. [39].

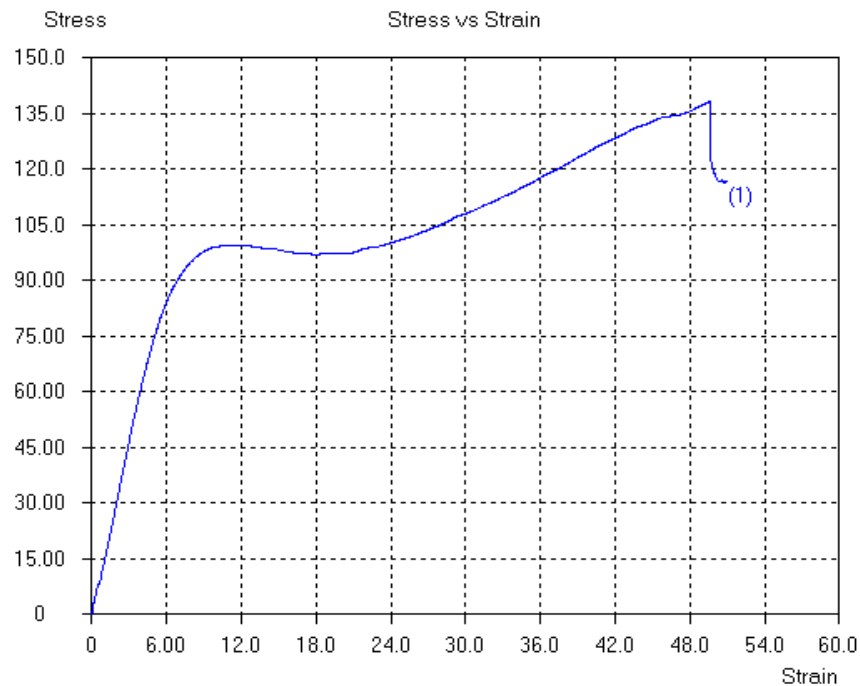


Figure 7. Compressive stress-strain response of the sintered Al-Al₂O₃ functionally graded pellet.

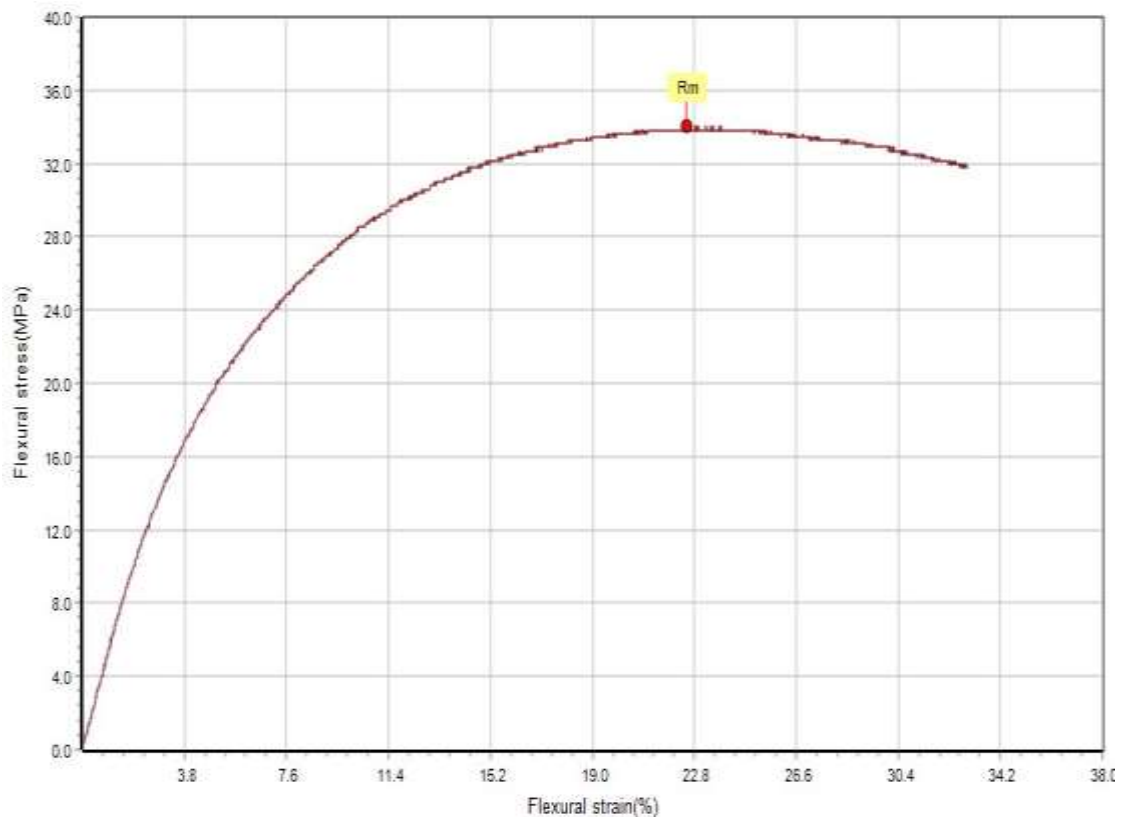


Figure 8. Flexural stress-strain response of the vacuum-sintered Al-Al₂O₃ functionally graded pellet.

3.10. Wear test

The wear depth response (Figure 9) reveals that there was limited material removal in the test with a final wear depth of about 1.71 μm and the maximum absolute wear depth signal obtained was about 2.56 μm after 300 s of the wear test. The coefficient of friction remained constant from the initial running-in period with a mean of 0.278 ± 0.030 throughout the test. This moderate friction response and low wear

depth are consistent with the high hardness of the rich Al_2O_3 surface layer, which can restrict plastic deformation and reduce direct metallic contact during sliding. Recent studies on ceramic reinforced aluminum composites have similarly shown that wear and friction response are governed by reinforcement content, hardness, normal load, sliding conditions, and reinforcement distribution [19, 40, 41].

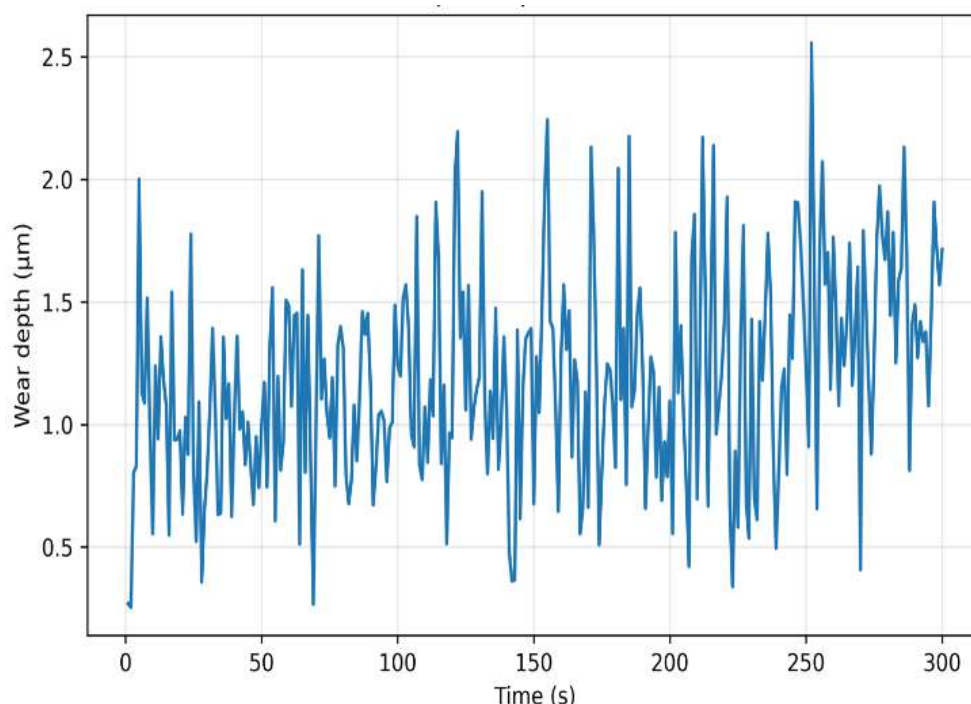


Figure 9. The wear depth response of the vacuum sintered Al- Al_2O_3 functionally graded pellet under a 10 N sliding load

4. Limitations of the Study

The following are the limitations of the study:

- Only one compaction pressure, 20 MPa, was used in this study; therefore, the reported porosity, densification efficiency, and hardness gradient should be interpreted as the outcome of the selected low pressure powder stacking route rather than optimized processing conditions.
- The vacuum sintering temperature was limited to 580 °C. This temperature was sufficient to preserve the pellet geometry and phase stability, but it was not sufficient to fully densify the Al- Al_2O_3 transition layers or to truly sinter the terminal pure Al_2O_3 layer.
- Layer-wise green density was not measured because the individual compacted thickness of each layer could not be determined after sequential stacking and final consolidation.
- Therefore, the relationship between initial packing state and final porosity remains interpretative.
- There was no measurement of actual post sintering thickness of each layer. Thus, a nominal equal thickness was used for the volume weighted density calculation, and it may not accurately reflect variations in shrinkability among Al_2O_3 -rich, mixed, and Al-rich layers.
- The density of the entire graded pellet was determined using the Archimedes method and the individual densities were not measured. The layer-wise density values have thus to be regarded as calculated apparent density values instead of experimentally measured layer densities.
- The morphology interpretations of the regions assigned as Al-rich regions, Al_2O_3 -rich regions and

transition zones are not interpreted as elemental confirmation and are not used for EDS mapping.

5. Conclusions

The study demonstrates the degree of applicability of a low pressure, sub-solidus powder stacking route to the fabrication of a stable phase Al-Al₂O₃ functionally graded pellet and presents the limits in densification imposed by the rich ceramic transition layers. The major results are summarized below:

- The designed five-layer Al/Al₂O₃ architecture was retained after vacuum sintering at 580 °C, and the pellet remained macroscopically stable without visible layer separation. This confirms that sequential powder stacking and controlled low pressure compaction can preserve the intended graded configuration, although not as a fully densified structural FGM.
- SEM analysis confirmed a gradual transition from an Al-rich matrix to an Al₂O₃-rich ceramic region, indicating retention of the designed graded architecture. However, localized pores and transition zone discontinuities show that incomplete pore closure and induced mismatch defects remain the key microstructural limitations.
- XRD confirmed that the sintered layers were dominated by FCC Al and α -Al₂O₃, with no obvious crystalline reaction products. Thus, the observed property gradient is mainly governed by constituent fraction, particle packing, limited oxide bonding, and porosity evolution rather than by new phase formation.
- Porosity evolved non-linearly across the graded thickness, with 9.12%, 15.26%, 10.26%, 16.23%, and 15.36% from Layers 1 to 5, respectively. The maximum porosity of the transition layers suggests that the Al-Al₂O₃ mixed regions are the most significant microstructural factor, because they impede Al-Al contact, hinder the rearrangement and hinder the closing of pores.
- The bulk density measured was 2.88 g·cm⁻³, which agreed with the bulk density calculated from the individual porosity and the theoretical bulk density of the mixture. This agreement helps to confirm the reliability of the image-based porosity measurement methodology with Archimedes density as a viable technique for the evaluation of graded powder metal parts.
- Microhardness increased strongly from 44.6 HV0.1 in the Al-rich layer to 703.0 HV0.1 in the Al₂O₃-rich layer, demonstrating that the compositional gradient was effectively translated into a functional hardness gradient. The hardness increase remained dominant despite residual

porosity, indicating that ceramic fraction and plasticity constraint controlled local indentation resistance more strongly than porosity reduction effects.

- The pellet exhibited good mechanical and tribological properties under the test conditions, such as compressive strength of 138.24 MPa, flexural strength of 34.03 MPa, mean coefficient of friction of 0.278 ± 0.030 , and final wear depth of $\sim 1.71 \mu\text{m}$. These answers suggest possible applications in localized surface reinforcement, and for cases where a high surface hardness is needed, if further densification and interfacial integrity are obtained.

Low pressure powder stacking can create a stable Al-Al₂O₃ hardness gradient without any detectable reaction phases, and the porosity of the transition layer is identified as the main obstacle to the structural performance. The porosity in the transition layers should be minimized in future work, by optimizing compaction pressure, tailoring of the particle size, hot pressing, spark plasma sintering, transient liquid phase assistance, or post-sintering infiltration. Additional interfacial characterization replicated fabrication trials, and service-oriented testing under wear, thermal cycling, and impact loading are also required before extending this route toward load bearing or thermally demanding FGM applications.

Conflicts of Interest: The authors declare no conflict of interest.

Funding: The authors didn't receive any funding

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Appendix

Table A1. Layer-wise hardness gradient with standard deviation

Layer	Al/Al ₂ O ₃ vol. %	Hardness readings, HV0.1	Mean $\pm$ SD, HV0.1
Layer 1	100/0	40.2, 45.5, 48.1	44.6 $\pm$ 4.03
Layer 2	75/25	78.9, 84.2, 86.5	83.2 $\pm$ 3.90
Layer 3	50/50	205.4, 209.6, 215.6	210.2 $\pm$ 5.13
Layer 4	25/75	505.4, 514.2, 518.8	512.8 $\pm$ 6.81
Layer 5	0/100	701.6, 701.7, 705.7	703.0 $\pm$ 2.34

Table A2. The specifications of the Ducom pin-on-disc tribometer

Parameter	Value
Disc rotational speed	100-2000 rpm
Sliding speed range	0.26 to 10 m/sec
Disc size	160 mm diameter x 8 mm thick
Wear track diameter	20 to 140 mm
Pin size	8-12 mm diagonal/ diameter
Max normal load	200 N
Max Temperature	400 °C