

Thermal treatment effects on the microstructure and flexural performance of handmade α - Al_2O_3 crucibles

Efectos del tratamiento térmico en la microestructura y el comportamiento a flexión de crisoles de α - Al_2O_3 elaborados artesanalmente

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Abstract

Alumina crucibles are widely used ceramic components in materials science and engineering owing to their high thermal stability and suitability for processes such as melting and solid-state synthesis. This study reports the structural, morphological, and mechanical characterization of locally handmade alumina crucibles sourced from a commercial supplier in Bogotá, Colombia, evaluated after three distinct thermal histories: as-received at room temperature (AT), high-temperature exposure at 1350 °C for 12 hours (HT), and cold treatment by liquid nitrogen immersion at -196 °C (CT). X-ray diffraction identified α - Al_2O_3 (corundum) as the dominant crystalline phase in all conditions, with minor SiO_2 and Al_2SiO_5 -related secondary signals. No phase transformations were detected after any treatment, confirming the material's high structural and chemical stability. Fracture surface analysis revealed that HT samples exhibited the largest projected pore area ($8.12 \pm 1.67 \mu\text{m}^2$) compared with AT ($6.51 \pm 1.60 \mu\text{m}^2$) and CT ($5.38 \pm 1.22 \mu\text{m}^2$), accompanied by a distributed network of grain-scale microcracks. Mechanical testing indicated that HT crucibles reached the highest apparent bending modulus (331.78 GPa) and load-bearing capacity, surpassing AT (238.87 GPa) and CT (106.17 GPa), which was attributed to a microcrack toughening mechanism, whereas CT displayed a more brittle response. The mechanical behavior was governed primarily by defect architecture rather than

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compositional changes, and the crucibles demonstrated performance levels compatible with the moderate temperature melting of non-ferrous metals.

Keywords: Alumina, refractory crucibles, thermal treatment, mechanical properties, microstructure, locally manufactured materials.

Resumen

Los crisoles de alúmina son componentes cerámicos ampliamente utilizados en ciencia e ingeniería de materiales debido a su alta estabilidad térmica y su idoneidad para procesos como la fusión y la síntesis en estado sólido. Este estudio presenta la caracterización estructural, morfológica y mecánica de crisoles de alúmina elaborados artesanalmente y obtenidos de un proveedor comercial en Bogotá, Colombia, evaluados después de tres historias térmicas diferentes: recibidos a temperatura ambiente (AT), exposición a alta temperatura a $1350\text{ }^{\circ}\text{C}$ durante 12 horas (HT) y tratamiento en frío mediante inmersión en nitrógeno líquido a $-196\text{ }^{\circ}\text{C}$ (CT). La difracción de rayos X identificó la $\text{-Al}_2\text{O}_3$ (corindón) como la fase cristalina predominante en todas las condiciones, junto con señales secundarias menores asociadas a SiO_2 y Al_2SiO_2 . No se detectaron transformaciones de fase después de ninguno de los tratamientos, lo que confirma la elevada estabilidad estructural y química del material. El análisis de las superficies de fractura reveló que las muestras HT presentaron la mayor área proyectada de poros ($8,12 \pm 1,67\text{ }\mu\text{m}^2$) en comparación con AT ($6,51 \pm 1,60\text{ }\mu\text{m}^2$) y CT ($5,38 \pm 1,22\text{ }\mu\text{m}^2$), acompañada por una red distribuida de microgrietas a escala de grano. Los ensayos mecánicos indicaron que los crisoles HT alcanzaron el mayor módulo aparente de flexión ($331,78\text{ GPa}$) y la mayor capacidad de carga, superando a AT ($238,87\text{ GPa}$) y CT ($106,17\text{ GPa}$). Este comportamiento se atribuyó a un mecanismo de tenacificación por microgrietas, mientras que las muestras CT mostraron una respuesta más frágil. El comportamiento mecánico estuvo gobernado principalmente por la arquitectura de defectos más que por cambios composicionales, y los crisoles demostraron niveles de desempeño compatibles con la fusión de metales no ferrosos a temperaturas moderadas.

Palabras clave: alúmina, crisoles refractarios, tratamiento térmico, propiedades mecánicas, microestructura, materiales de fabricación local.

Introduction

Crucibles are essential refractory vessels for laboratory and thermochemical operations, enabling the containment and processing of materials at very high temperatures during melting, heat treatment, calcination, and solid-state synthesis (Alimdjanova *et al.*, 2025; Khan *et al.*, 2024). Their selection and performance depend on thermo-mechanical stability and chemical inertness under aggressive environments, which is why application-oriented characterization and process control remain critical for safe and reproducible high-temperature work (Fan *et al.*, 2021).

Commercial and custom refractory crucibles are commonly based on oxides such as alumina (Al_2O_3), magnesia (MgO), and zirconia (ZrO_2), selected to withstand temperatures above approximately $\sim 1200^\circ\text{C}$ and intense thermal gradients. Small additions of specific elements or phases (e.g., Ni, Zr, or mullite formers) may be used to tune sintering, microstructure, and corrosion resistance, thereby improving thermo-mechanical robustness and service lifetime in service (Fan *et al.*, 2023a). The ability to resist thermal shock without permanent deformation or catastrophic fracture is particularly important for minimizing downtime and extending component life; likewise, chemical stability against molten metals and slags reduces interfacial reactions, limits refractory dissolution, and prevents contamination of melts and treated samples (Nwankwo *et al.*, n.d.).

Together with microstructural optimization and proper raw-material selection (purity, additives, and coatings), these attributes translate into durable and reliable performance in metallurgical and materials research settings (Nwankwo *et al.*, n.d.).

Among oxide refractories, $\alpha\text{-Al}_2\text{O}_3$ (corundum) is a workhorse material because of its high hardness, elastic stiffness, and thermal/chemical stability (Ikesue and Aung, 2023; Fan *et al.*, 2023a). Corundum's trigonal structure offers a well-understood crystallography, enabling tailoring of the processing–property relationship. When alumina is sintered to high density, hardness typically reaches the upper teens of GPa, and flexural strength can exceed several hundred MPa under appropriate processing and surface finish (Ikesue and Aung, 2023). In practice, the processing route (e.g., powder quality from Bayer-derived alumina, slip casting, and sintering at $1500\text{--}1600^\circ\text{C}$) governs defect populations and grain growth, ultimately controlling mechanical performance and reliability (Fan *et al.*, 2023a). Crystallographic fingerprints of $\alpha\text{-Al}_2\text{O}_3$ and its strongest reflections are extensively tabulated, facilitating phase identification and preferred-orientation assessment during quality control (Ikesue and Aung, 2023).

The process–microstructure–performance linkage in alumina crucibles is especially sensitive to porosity architecture (volume fraction, size distribution, and connectivity), grain size, and secondary phases introduced intentionally (e.g., ZrO_2 , MgO , and silica) or carried as residuals.

As porosity increases and pores coarsen, strength decreases sharply and data variability increases; conversely, densification, defect refinement, and controlled grain growth improve flexural strength and thermal-shock tolerance. These trends have been quantified in both conventionally processed and additively manufactured alumina, where microstructural control raises the Weibull modulus and shifts characteristic strength upward (Makireddi *et al.*, 2020). For porous or slip-cast alumina crucibles with moderate porosity, reported room-temperature flexural strength can fall to a few tens of MPa; however, strength retention after thermal-shock cycling may remain acceptable for certain melting operations (Fan *et al.*, 2021, p. 2).

Benchmark properties of high-purity sintered alumina include a Young's modulus on the order of 380–400 GPa and a Poisson's ratio near 0.25–0.27, reflecting a stiff, low-compliance solid (Munro, 1997). Under optimized processing and surface preparation, monolithic alumina can exhibit flexural strengths typically in the ~300–550 MPa range and a fracture toughness of ~3–5 MPa·m^{1/2}, values consistent with a hard yet intrinsically brittle ceramic (Shakirzyanov *et al.*, 2025). Recent advances in advanced forming (e.g., stereolithography) reaffirm that dense alumina parts approach the higher end of these ranges when porosity is minimized, and flaw populations are carefully controlled.

Despite these well-established principles, handmade α -Al₂O₃ crucibles, which remain attractive in cost-constrained contexts, often experience variable thermal histories and uncontrolled microstructures, and systematic studies connecting

thermal treatment, microstructure, and service-relevant mechanical response are scarce. This knowledge gap is consequential: without data-driven guidance, users face risks of sample contamination, premature failure, and avoidable costs. Studies on handmade crucibles produced from local clays in West Africa highlight economic and availability advantages relative to imports, underscoring the value of fit-for-purpose characterization to inform local manufacturing (Barry *et al.*, 2024; Nwankwo *et al.*, n.d.).

At the same time, reports of low strength and poor thermal-shock stability in locally produced crucibles often trace back to compositional and microstructural shortcomings, including insufficient alumina-rich or mullite phases, high residual porosity, and non-optimized sintering cycles (Barry *et al.*, 2024).

This work addresses that gap by examining how thermal treatments modulate the phase constitution (XRD), microstructure, and porosity (SEM/stereology) and flexural performance of handmade α -Al₂O₃ crucibles. Specifically, we evaluate the effects of thermal history on (i) lattice-level indicators and secondary-phase signatures, (ii) porosity content and architecture, and (iii) bending strength and its scatter, discussed in the context of defect populations relevant to thermal-shock scenarios. By explicitly mapping processing to microstructure and mechanical response, our study provides application-oriented guidance to improve local manufacturing practices, establish a technical baseline for performance benchmarking, and reduce variability and risk in high-temperature operations that rely on alumina crucibles.

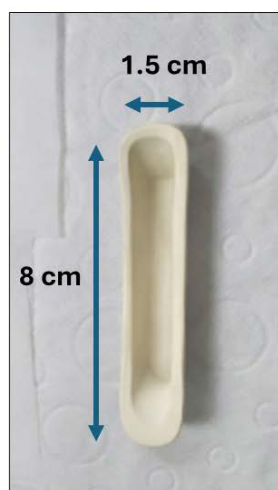
Materials and methods

Materials and specimen description

Handmade alumina (Al_2O_3) crucibles were sourced from local artisanal manufacturers in Ricaurte, Bogotá, Colombia. According to the supplier, the crucibles were produced manually from high-purity alumina powders; however, no certified technical datasheet,

standardized manufacturing protocol, or detailed compositional/mechanical specifications were available. The nominal external dimensions were ~80 mm in length, ~15 mm in width, and ~10 mm in depth, with a boat-shaped geometry (Figure 1). Minor dimensional variability across pieces was expected given the artisanal process; therefore, all test-relevant dimensions (span, width (b), and thickness (d)) were individually measured with a digital caliper (± 0.01 mm) immediately prior to testing to minimize geometric bias.

Figure 1. Handmade crucibles used in the study.



Source. Own elaboration.

Thermal treatments

Three thermal-history conditions were defined: (i) as-received (AT), with no thermal treatment; (ii) high-temperature treatment (HT), consisting of furnace exposure at 1350°C in ambient air for 12h, followed by controlled cooling to room temperature; and (iii) cold treatment (CT), consisting of a single immersion cycle at -195°C

(liquid nitrogen) for 12h. The HT condition was selected to be representative of a consolidation/densification heat-treatment window for alumina-based bodies, whereas CT was explored as a contrasting history to probe microstructural and mechanical sensitivity to severe temperature excursions. After each treatment, crucibles were allowed to equilibrate to laboratory conditions for at least 24h prior to characterization.

Phase analysis by X ray diffraction (XRD)

Powder XRD patterns were collected on a PANalytical X'Pert PRO MPD diffractometer (Cu K α , λ = 1.5406 Å) over 10–90° 2 θ . Diffraction data were acquired using a step size of 0.02° with an equivalent counting time sufficient to resolve minor peaks. Phase identification was performed by pattern matching against reference databases (ICDD/NIST) with particular attention to the α -Al₂O₃ (corundum) fingerprints and potential SiO₂/aluminosilicate signatures. Peak positions and relative intensities of corundum were cross checked against well curated datasets (e.g., NIST SRM676a/1976c summaries and recent IUCr/NIST analyses of α -Al₂O₃ intensity sequences) to avoid misassignment of quartz family reflections near 26.6° 2 θ (Cu K α).

Microstructure and composition (SEM/EDS) and pore stereology

Fracture surfaces were examined by scanning electron microscopy (SEM, TESCAN VEGA3). Representative regions were imaged to assess grain morphology, pore distribution and size, and the presence of defects associated with the manufacturing route and thermal history. Qualitative energy dispersive X ray spectroscopy (EDS) was used to screen for elemental signatures of possible secondary phases (e.g., Si, Mg, or Zr bearing species). To contextualize porosity, image analysis and stereology were performed on SEM micrographs using ImageJ. The images were calibrated with scale bars. A reproducible thresholding

workflow was applied to segment pores, and the area fraction and equivalent circular diameters were extracted.

Flexural testing

Bending tests were performed on a universal testing machine (Shimadzu AG IS, 5 kN load cell) in a three point bending configuration to obtain comparative flexural strength across thermal histories. Because the available material consisted of complete crucibles (i.e., non standard bars) and only one specimen per condition was available, tests were executed as an exploratory comparison and do not constitute an ASTM compliant strength characterization. Where geometry permitted, a constant outer span and mid span loading were used; the span (L), width (b), and thickness (d) of the loaded section were measured immediately prior to testing. The cross head speed was chosen to keep the time to failure within the typical windows used for advanced ceramics.

Although the setup was inspired by ASTM C1161 practice (flexural strength of advanced ceramics at ambient temperature), we explicitly acknowledge two departures from the standard: (1) specimen geometry was not machined to a prescribed bar, and (2) $n = 1$ per condition prevents statistical treatment or size scaling. Accordingly, results are interpreted as comparative indicators suitable for discussing thermal history effects within this study but not as design allowables. For future work, we plan to test ≥ 10 replicate bars per condition, report strength distributions, and estimate Weibull parameters in full compliance with ASTM C1161.

The maximum outer fiber stress (reported here as “flexural strength” for brevity) under three point bending was calculated from simple beam theory:

$$\sigma_f = \frac{3 F_{\max} L}{2 b d^2} \quad (1)$$

where $F_{\max}$ is the peak load (N), L is the support span (mm), b is the specimen width (mm), and d is the thickness (mm). Results are reported in MPa. The Young’s modulus in bending, when estimated, was obtained from the initial force-stroke slope $S = \Delta F / \Delta \delta$ according to ASTM C1161 data reduction guidance for three point bending of rectangular beams:

$$E = \frac{L^3 S}{4 b d^3} \quad (2)$$

This formulation avoids non physical ratios of “stress/strain at failure” and reflects small deflection beam theory.

Data handling, limitations, and quality assurance

Because each thermal history condition was represented by a single crucible, no statistical analysis (e.g., Weibull plotting) was attempted. All dimensional measurements and raw load-displacement data were archived; SEM fields of view and image analysis steps were documented to ensure reproducibility. The exploratory nature of the test matrix is clearly disclosed alongside the departures from ASTM C1161 so that flexural results are interpreted as comparative metrics rather than standardized material properties.

Results and discussion

Structural characterization (XRD)

Figure 2 shows the X-ray diffraction (XRD) patterns collected for the as-received (AT), high-temperature (HT, 1350 °C/12 h), and cold-treated (CT, −195 °C/12 h) crucibles. All diffractograms are dominated by α - Al_2O_3 (corundum), as evidenced by the set of reflections near 35.1° (104), 43.3° (113), 52.5° (024), 57.5° (116), and 66–68° 2 θ (300/1.10.4) for Cu K α radiation, peak families that are canonical for corundum and widely tabulated in ICDD/NIST resources and corundum intensity studies.

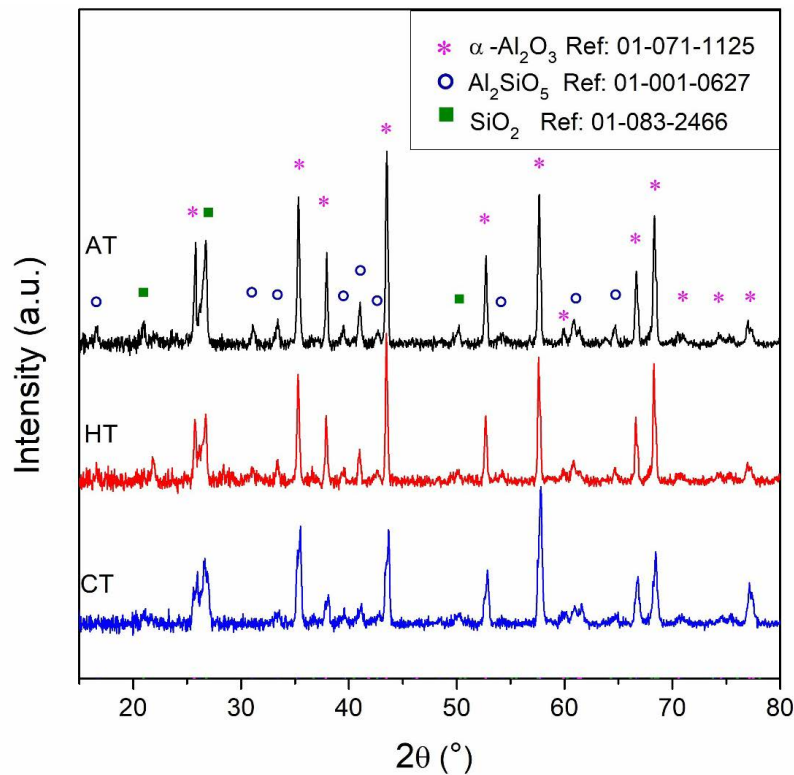
Low angle features around 26.6° 2 θ and, depending on the specimen, near 22.8° 2 θ are not characteristic of α - Al_2O_3 ; they are consistent instead with SiO_2 (quartz) and aluminosilicate-type contributions, respectively. This assignment agrees with the relative intensities predicted/reported for corundum (where the strongest peaks are located at ~35–58° 2 θ) and helps avoid misindexing the quartz reflection as corundum.

Across AT, HT, and CT, no new crystalline phases appeared within the detection limit, indicating phase stability of α - Al_2O_3 over the applied thermal histories (Figure 2). Minor signals assigned to SiO_2 and an Al_2SiO_5 -type phase were present in all conditions with much lower intensities than corundum, suggesting secondary fractions likely associated with raw material residuals or process-induced contamination in the artisanal manufacturing route.

Given the HT schedule (1350°C) and the Al_2O_3 – SiO_2 system, mullite formation cannot be ruled out; the peak profiles associated with Al_2SiO_5 can overlap with

mullite families at moderate intensity, and definitive discrimination would require Rietveld refinement and/or selective peak fitting beyond this exploratory scope.

Figure 2. DRX patterns of samples AT, HT, and CT.



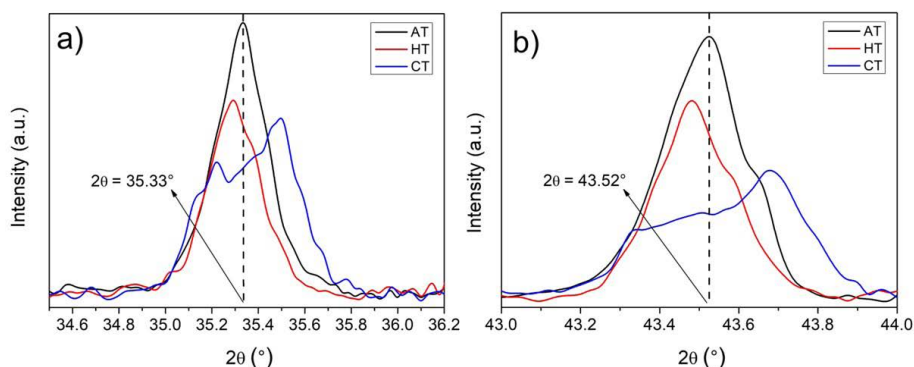
Source. Own elaboration.

A careful overlay (Figure 3) reveals small, systematic peak shifts between conditions at the strongest α - Al_2O_3 reflections (e.g., $\sim 35.1^\circ$ and $\sim 43.3^\circ$ 2θ). The HT pattern exhibits slight shifts to lower 2θ , qualitatively consistent with increased interplanar spacing (lattice expansion and/or relaxation of residual stresses after the 1350°C dwell), whereas CT shows slight shifts toward higher 2θ values, compatible with a lattice-contraction scenario (Luo *et al.*, 2020). While such trends are physically plausible for ceramics

subjected to distinct thermal histories, any strain quantification would require an internal standard and Rietveld parameter refinement to separate instrument drift from true lattice changes (Fan *et al.*, 2021; Hattali *et al.*, 2010; Rietveld, 2014).

Figure 3 shows the XRD patterns of handmade α - Al_2O_3 crucibles after AT, HT, and CT treatments. Dominant α - Al_2O_3 reflections are labeled, and low-intensity features consistent with $\text{SiO}_2/\text{Al}_2\text{SiO}_5$ are

Figure 3. Magnified views around the (104) and (113) α -Al₂O₃ reflections, highlighting small peak shifts among AT/HT/CT.



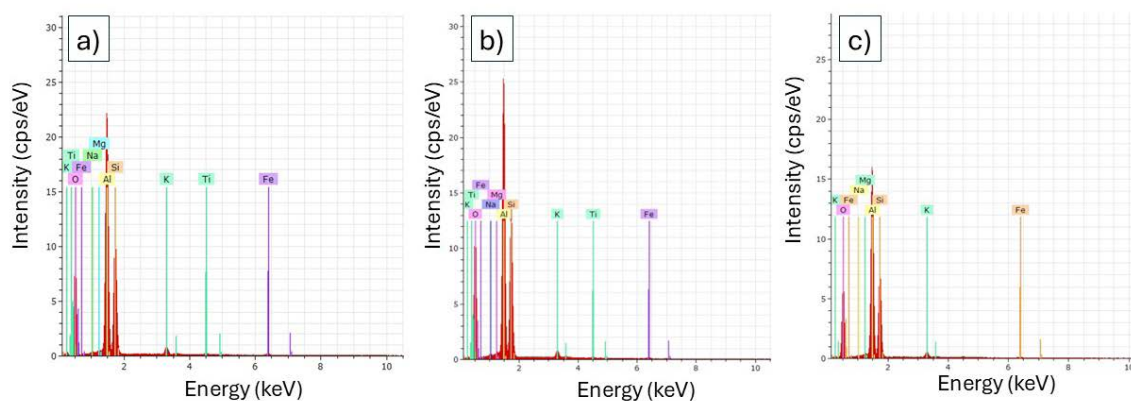
Source. Own elaboration.

Compositional characterization (EDS)

Energy dispersive X-ray spectroscopy (EDS) confirms that O and Al are the predominant elements in all conditions, with Si consistently present, supporting the XRD observation of silica/aluminosilicate secondary species (Figure 4). Trace amounts of K, Na, Mg, Ti, and Fe were detected at <1 at.% (Table 1), compatible with impurities

originating from raw materials and/or the artisanal manufacturing process. Within the resolution limits of EDS on fractured surfaces, no systematic compositional change was observed between AT, HT, and CT. The slight increase in O and corresponding decrease in Al in HT are within the typical semiquantitative uncertainty of the technique and are therefore not interpreted as evidence of volatilization/segregation (Musket and Strausser, 1980).

Figure 4. EDS spectra for (a) AT, (b) HT, and (c) CT.



Source. Own elaboration.

Table 1. Elemental composition (at.%) of handmade α -Al₂O₃ crucibles after the three thermal histories: AT, HT (1350 °C, 12 h), and CT (−195 °C, 12 h) measured by EDS on fracture surfaces.

Element	AT (at.%)	HT (at.%)	CT (at.%)
O	64.63	66.05	64.63
Al	22.82	21.01	22.82
Si	10.63	10.94	10.63
K	0.63	0.53	0.63
Na	0.58	0.72	0.53
Mg	0.45	0.53	0.54
Fe	0.16	0.12	0.17
Ti	0.10	0.10	-
Total	100	100	100

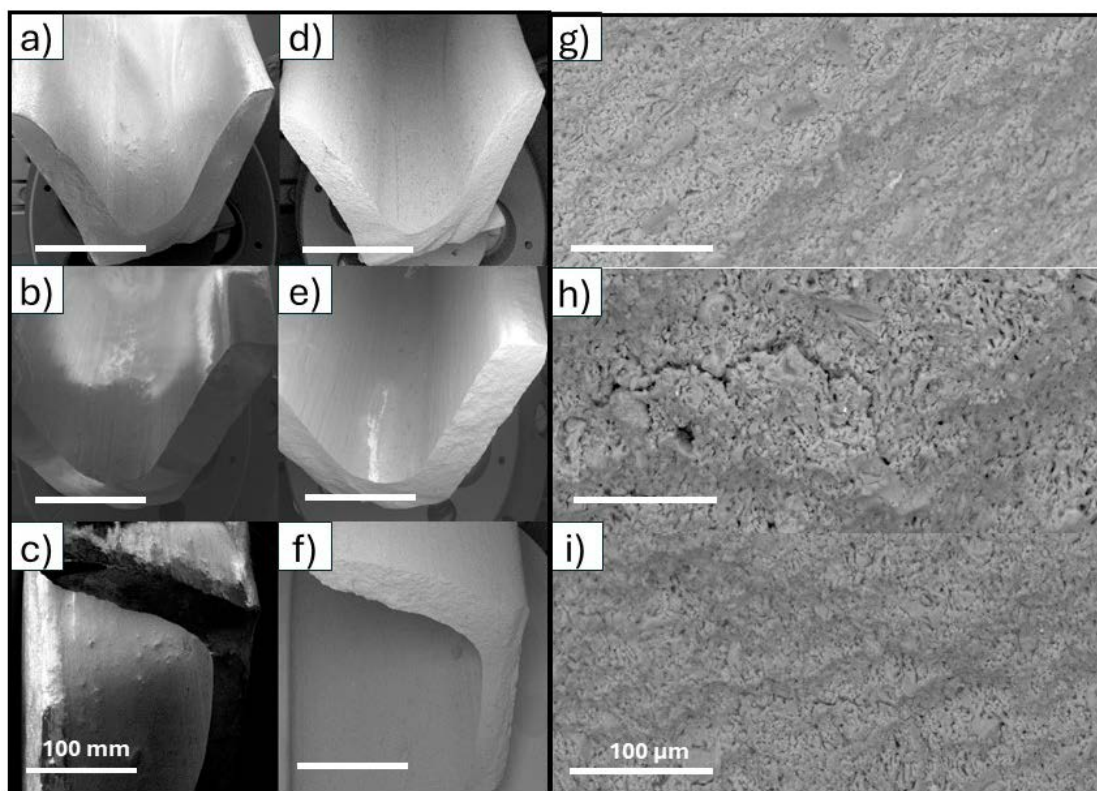
Source. Own elaboration.

Morphological characterization

Surface morphology was examined by SEM at multiple magnifications to assess the pore population, grain-scale features, and thermally induced defects in AT, HT, and CT. Representative micrographs are shown in Figure 5. At low/intermediate magnification, the three conditions exhibit a broadly similar intergranular morphology typical of sintered α -Al₂O₃, with a discontinuous pore network. At higher magnification, however, the HT specimen shows a distributed microcrack population intersecting grain boundaries and ligament bridges, whereas CT does not display a pervasive microcrack network within the same magnification range, consistent with literature reports of thermally driven microcracking in oxide ceramics (Sun *et al.*, 2024).

To quantify porosity differences, fracture micrographs were analyzed by ImageJ/Fiji (calibration with the scale bar, background correction, fixed threshold segmentation, and binary cleanup with a minimum feature size), following best-practice digital image analysis procedures (Schneider *et al.*, 2012). For each condition, *n* representative fields were processed, and the equivalent pore area (projected, μm^2) was extracted. The resulting mean $\pm$ SD values were 6.51 ± 1.60 (AT), 8.12 ± 1.67 (HT), and 5.38 ± 1.22 (CT), indicating a larger projected pore size after the 1350 °C dwell (HT) and a smaller value after cold exposure (CT) (Figure 5). The rank order HT > AT > CT was consistent across the analyzed fields. Variations of pore size with thermal exposure are well documented in ceramics and porous glass ceramic systems, where high temperature holds can promote coarsening, crack formation, and redistribution of porosity (Guo *et al.*, 2022; Nigay *et al.*, 2017).

Figure 5. SEM surface morphology of handmade α -Al₂O₃ crucibles AT (a–c, g), HT (1350 °C/12 h; d–e, h), and CT (–195 °C/12 h; f, i) conditions. Left and center columns (a–f) show low-magnification views (scale bar = 100 μ m), whereas the right column (g–i) presents high-magnification micrographs (scale bar = 100 μ m) showing grain-scale porosity and the distributed microcrack network observed in HT (h).



Source. Own elaboration.

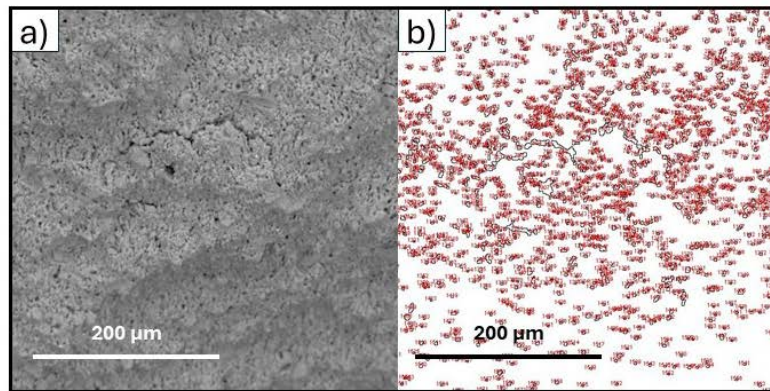
Mechanistically, the increase in equivalent pore area for HT can arise from pore coalescence/coarsening and grain boundary relaxation during the high temperature dwell, while the decrease for CT may reflect closure of narrower ligament voids in the projected fracture surface and a shift of the detectable pore population.

Because fracture surface stereology preferentially samples open porosity and 2D projections, these interpretations remain qualitative; a volumetric cross check (e.g., the Archimedes method) would better

resolve the open/closed pore balance as illustrated by the segmentation workflow in Figure 6.

In brittle ceramics, larger and/or more connected defects increase stress concentration and tend to lower flexural strength while raising scatter, whereas refined pore populations improve characteristic strength and reliability, trends repeatedly observed in Al₂O₃ systems and relevant to the mechanical response discussed later (Guo *et al.*, 2022; Ikesue and Aung, 2023; Nigay *et al.*, 2017).

Figure 6. Pore stereology on SEM fracture surfaces (example shown for AT).



Note. (a) Raw secondary electron micrograph; (b) segmented pore mask overlaid on the same field (red) used to compute the equivalent pore area (μm^2). Scale bar = 100 μm . The same ImageJ/Fiji workflow (fixed threshold and minimum feature size) was applied to AT, HT, and CT; report as mean $\pm$ SD over n fields per condition.

Source. Own elaboration.

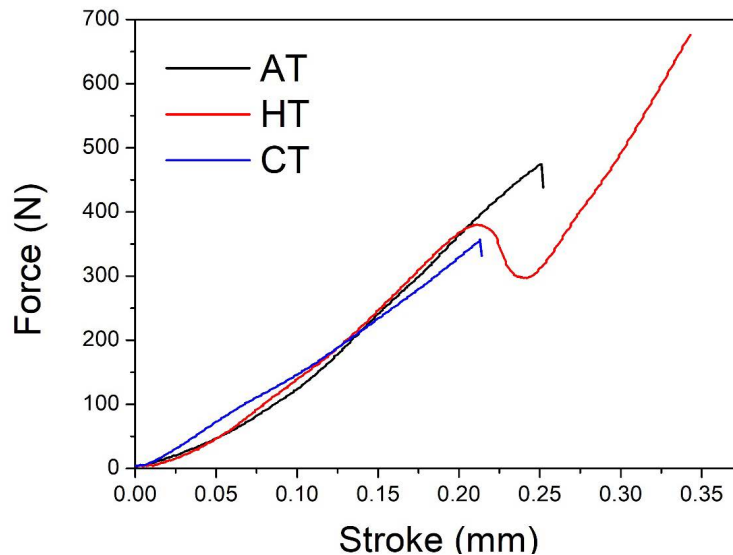
Mechanical testing (three point bending)

To assess the effect of thermal history on mechanical response, three point bending tests were performed on one crucible per condition (AT, HT, CT) using a universal testing machine (Shimadzu AG-IS, 5 kN cell). The span (L), width (b), and thickness (d) at the loaded section were measured immediately prior to testing. Figure 7 plots the resulting force-displacement curves. In the initial linear regime, the CT specimen exhibits a slightly higher slope (apparent stiffness) than AT and HT; as the displacement increases, the HT specimen attains the highest peak force and, critically, shows a pre-failure nonlinear segment (arrow in Figure 7) before final fracture. At room temperature, macroscopic plasticity is not expected in alumina; such nonlinearity is instead consistent with stable propagation of preexisting microcracks and distributed

crack-bridging events that dissipate energy without immediate catastrophic failure. The behavior aligns qualitatively with the microcrack network observed in HT by SEM (Section 3.3).

Quantitative outcomes are summarized in Tables 2-3 (peak force F_{max} , displacement at peak load, apparent flexural stress, and maximum strain). In our dataset, HT reached the largest F_{max} and displacement at peak load, indicating the highest load bearing capacity and the largest elastic plus nonlinear deflection prior to failure. Conversely, CT showed the lowest F_{max} and displacement, consistent with the most brittle response among the three conditions. Because $n=1$ per condition, these differences must be regarded as comparative/indicative rather than statistically representative; a full characterization will require ASTM C1161 compliant bars with ≥ 10 replicates per condition to enable Weibull analysis and size scaling.

Figure 7. Force-stroke curves from three point bending at room temperature for handmade α -Al₂O₃ crucibles after the three thermal histories: AT (as received, black), HT (1350 °C/12 h, red), and CT (−195 °C/12 h, blue).



Source. Own elaboration.

Table 2. Three-point bending results for handmade α -Al₂O₃ crucibles after the three thermal histories: AT (as-received), HT (1350 °C/12 h), and CT (−195 °C/12 h). Peak force and displacement at peak are reported together with flexural stress and strain at peak.

Sample	MaxForce (N)	MaxDisp (mm)	MaxStress (N/mm ²)	MaxStrain (%)
AT	474.063	0.251	42665.6	0.04183
HT	675.625	0.343	60806.3	0.05717
CT	357.031	0.213	32132.8	0.03550

Source. Own elaboration.

Taken together with the SEM observations, the microcracks observed only in the HT condition can rationalize the load-bearing behavior when interpreted through the lens of fracture mechanics in ceramics. It is important to distinguish between open porosity, which weakens ceramics when pores are large, interconnected, and randomly oriented, and thermally induced distributed microcracking, which can

operate as a toughening mechanism. As discussed by Clegg and subsequent studies on polycrystalline alumina (Brahmadathan and Lakshmana, 2024), a network of fine, controlled microcracks at the grain scale can increase the apparent fracture resistance by promoting crack deflection and branching and distributed energy dissipation without necessarily reducing flexural strength, provided that critical flaws (large pores)

do not increase in proportion (Jia *et al.*, 2026; Nigay *et al.*, 2017; Brahmadathan and Lakshmana, 2024). This behavior is consistent with microcrack toughening, in which numerous distributed microcracks facilitate deflection, branching, and arrest of the main crack, increasing the effective fracture surface area and the energy dissipated during failure (Barros *et al.*, 2021). According to the Evans & Faber model (1984), microcrack toughening is effective when grain size is on the order of 10–50 μm and microcrack density is sufficiently high to deflect the main crack without forming a percolating fracture path. Although K_{IC} was not measured in this study, the pseudo plastic segment in the HT force-stroke curve (Figure 6) constitutes indirect evidence of a distributed energy dissipation mechanism consistent with microcrack toughening (Barros *et al.*, 2021; Maiti *et al.*, 2022).

Table 3 summarizes the bending modulus (E) and the reported flexural strength (FS) for each condition. The highest E is found in HT (331.78 GPa), followed by AT (238.87 GPa) and CT (106.17 GPa). In the literature, the flexural strength of alumina typically lies in the ~300–550 MPa range for dense, high purity bodies, values characteristic of a hard yet intrinsically brittle ceramic (Shakirzyanov *et al.*, 2025). Conversely, E is known to decrease systematically with porosity; for example, aluminas with ~9% porosity may show $E \approx 114$ GPa, whereas porosity levels of 20–50% can reduce E to ~17–83 GPa (Asmani *et al.*, n.d.; Nakajima *et al.*, 2019). Within the exploratory nature of our dataset, the present E values are compatible with an estimated porosity of ~5–15%, coherent with the artisanal sintering route of the crucibles. A volumetric porosity measurement (e.g., the Archimedes method) is recommended for future work to validate this correlation.

Table 3. Young's modulus (E) and flexural strength (of) from three point bending of handmade α -Al₂O₃ crucibles after the three thermal histories: AT (as received), HT (1350 °C/12 h), and CT (–195 °C/12 h), measured at room temperature .

Sample	E (GPa)	FS (Mpa)
AT	238.87	4.98
HT	331.78	6.91
CT	106.17	2.21

Source. Own elaboration.

In contrast to the toughening-like response inferred for HT, the CT condition exhibited a different mechanical signature relevant to prolonged low-temperature storage or liquid nitrogen assisted syntheses. The smaller equivalent pore area (5.38 μm^2) and lower

flexural strength reported for CT suggest that rapid cooling to –196 °C may produce differential contraction of the crystal lattice (also reflected by right-shifted XRD peaks), introducing microstresses without developing an observable microcrack network.

This, together with the reduced E (106.17 GPa), indicates that artisanal alumina crucibles are less suitable for cyclic cold use unless thermally consolidated beforehand.

To understand the origin of these differences across conditions, structural and compositional results were integrated. No phase transformation was detected by XRD after thermal treatments, supporting structural stability. Likewise, EDS revealed no elemental segregation. These findings indicate that the observed mechanical variations are primarily associated with microstructural evolution and deformation mechanisms (e.g., porosity architecture and microcracking) rather than bulk compositional changes. Given that phases and stoichiometry were preserved at both high and low temperatures for all three specimens, the mechanical behavior can be attributed mainly to morphology and microstructure.

Overall, these results help contextualize the application envelope of the artisanal crucibles. The bending strengths we obtained in this study are lower than the ~300-550 MPa range reported for dense, high-purity alumina but comparable to the ~20-30 MPa reported for slip-cast Al_2O_3 crucibles with moderate porosity (Fan *et al.*, 2023b). This suggests that the crucibles studied here, particularly under the HT condition, possess adequate mechanical resistance for melting at moderate temperatures (below the melting point of most non-ferrous metals), whereas they may be insufficient for more demanding applications such as steel or high-temperature alloy melting above ~1500 °C.

Conclusions

X-ray diffraction demonstrated that $\alpha\text{-Al}_2\text{O}_3$ (corundum) is the dominant phase in all conditions (AT, HT, CT), with no formation or disappearance of crystalline phases after thermal treatments at 1350 °C and -196 °C, confirming high structural stability of alumina within the explored temperature window. Minor $\text{SiO}_2/\text{Al}_2\text{SiO}_5$ -type signals, consistent with aluminosilicate residues from processing, were present at low intensity and did not alter the phase field. The mechanical response can therefore be attributed primarily to $\alpha\text{-Al}_2\text{O}_3$ coupled with microstructural rather than compositional changes.

SEM and pore stereology corroborated thermal-history-dependent defect populations: a larger projected pore area with distributed microcracking in HT and a smaller projected pore area in CT. This trend is consistent with reports on porous alumina and other refractories, where thermal history governs pore size, connectivity, and morphology, directly impacting stiffness, flexural response, and apparent toughness. In the crucibles studied, the slight decrease in projected pore area for CT and the thermally induced microcrack network in HT rationalize the observed bending behavior: pores act as stress concentrators and fracture origins, with larger pores being most deleterious, whereas fine distributed microcracks at the grain scale dissipate energy through crack deflection and branching, temporarily impeding unstable propagation. Consequently, the HT microstructure exhibited greater resistance

to crack advance and a pre-failure nonlinear regime in bending, while AT and CT showed a more brittle-like response.

Overall, the artisanal α -Al₂O₃ crucibles studied here deliver mechanical performance compatible with moderate-temperature melting operations below the melting points of most non-ferrous metals. However, their flexural behavior, consistent with porous and slip-cast alumina, indicates they are not suitable for the most demanding high-temperature applications, such as steel or advanced-alloy processing above approximately 1500 °C, unless densification and defect control are improved. Given $n=1$ per condition and the use of non-standard specimen geometries, these conclusions should be interpreted as comparative and indicative. Full validation will require ASTM C1161-compliant bars with at least 10 replicates per condition, Weibull statistical analysis, fractographic evaluation of failure origins, Archimedes porosity measurements, and thermal-shock cycling tests to define practical service windows.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

Declaration of generative AI and AI assisted technologies in the writing process

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