



REGULAR ARTICLE

Sintering Conditions and their Effect on Alumina's (α -Al₂O₃) Mechanical Properties

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The current study explores the influence of sintering conditions on alumina derived through natural sintering. The nanopowder employed as the starting material was α -alumina (α -Al₂O₃), sourced from Baikowski (Baikalox-BMA15, France), boasting a purity of 99.99 % and an average particulate size of 150 nm. A wet process was utilized to prepare the slip, with 24 hours facilitating the deagglomeration of 50 g of Alumina powder. To eliminate potential contamination, the slip was cast within PVC molds and positioned on an alumina support. A comprehensive drying cycle was meticulously conducted to reduce the likelihood of cracking. This included steps such as boiling water, silica gel, dry medium exposure, oven treatment, and finally, a furnace. To negate the influence of organic additions and remnant gaseous species, the slip underwent a debinding process at 600 °C. The sintering process was executed at varying temperatures ranging from 1200-1500 °C, with 100 °C intervals, and holding durations of 1h and 3 h. The Grindo-Sonic device was applied to measure Young's modulus using a dynamic method, whereas the mechanical strength was ascertained through a four-point bending test. Samples sintered for 3h and 1h demonstrated breaking strengths of 352 and 641 MPa and 164 to 528 MPa respectively. Additionally, porous samples were fabricated using 5 % wheat starch as a porogen. The porosity within 1 h and 3 h varied between 0.01 %-0.18 %, and 0.01 %-0.1 % respectively, whereas, for porous samples, the porosity fluctuated between 0.1 %-0.35 %. The results showed that the samples sintered for a holding time of 3 h were denser than those sintered for 1 h. A 100 % dense sample was achieved at 1400 °C for 3 h. The analysis revealed that a longer holding duration of 3 h resulted in superior mechanical properties.

Keywords: Alpha Alumina, Porogen, Natural sintering, Microscopic structure.

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1. INTRODUCTION

The downsizing of powders to the nanoscale has a significant impact on numerous areas of research, such as materials engineering, contributing to advancements in various fields. Nanostructures exhibit improved mechanical properties and have good resistance to chemical attack (chemical inertia), a high hardness, and a high modulus of elasticity. These good attributes make these materials suitable for many optical, electronic, mechanical, and biomedical applications [1, 2].

Several authors have used alumina as a basis for composite materials [3-7]. Nanostructured ceramic products can be produced by two routes (wet and dry) [8]. The drying phase poses a challenge in the wet method due to the occurrence of cracking. Despite this difficulty, efforts have been made to find the best drying conditions after casting of the slip. To optimize these conditions and minimize the occurrence of cracking during the drying cycle, prismatic samples (45 × 50 × 10 mm³) and bars (45 × 10 × 6 mm³) were treated with progressive drying. Several researchers have created porous samples by incorporating different types of starch, including corn starch, potato starch, and rice starch [9, 10], as well as flour [11], and naphthalene [12, 13].

The aim of this study is to create both nonporous and

porous samples by incorporating wheat starch, and investigate how the mechanical properties of sintered alumina are affected by varying sintering conditions such as temperature and holding time.

2. METHODOLOGY AND EXPERIMENTAL PROCEDUR

2.1 Sample Preparation Protocol

The initial powder employed in this study was commercially sourced α -nanosized alumina (Baikalox-BMA15, Baikowski), which boasted a purity level of 99.99 % and an average particle size measuring 150 nm. To fabricate the samples, slip casting methodology was employed [4]. A template is a tool for enforcing a standard layout and look and feel across multiple pages or within content regions. It provides stricter standardization control of the documents.

Wet shaping methods require drying of the slug mixture before the sintering stage. Generally, these methods use additives to facilitate the shaping of the suspension for demoulding. These additives must be eliminated before the sintering stage to obtain homogeneous and dense materials. Among the most widely used liquid shaping processes is slurry casting (casting on a plaster mould,

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casting on a porous alumina mould and pressure casting). The selection of this method was based on its capability to produce samples with a high level of density [14-16]. To create the suspension, 50 g of BMA15 powder was dispersed in 50 g of deionized water, and hydrochloric acid was added to achieve a pH value of 3.

HCl served as a dispersant that was absorbed on the surface of the particles, and because of the resulting congestion of the particle space, it stabilizes the particles (steric effect). In addition, the load sometimes associated with dispersants also influenced the electrical forces by increasing the surface load, resulting in electrosteric repulsion when both electrostatic and steric action exist.

The suspension underwent effective electrostatic dispersion. To prevent contamination, the suspension was subsequently deagglomerated for a duration of 24 hours in a ball mill containing alumina balls.

In a similar manner, another slurry was prepared for porous samples. However, to ensure a uniform and homogeneous mixture for the porous samples (consisting of 50 g of powder and 5 % wheat starch with a diameter

of 20 microns), the mixture was first homogenized using a three-dimensional mixer for a period of 30 minutes prior to grinding. The suspension was dispersed in the same manner as the slurry that did not contain a porogen. It was found that the duration of grinding was dependent on the quantity of material being ground. We chose to use a dry matter ratio of 60 % (240 g powder to 160 g water), but this necessitated a doubling of the grinding time. The slurry's pH level decreased during grinding, a change that corresponded with the increase in the powder's specific surface area. We monitored the dispersion state and pH every 2 hours to study how the slurry evolved over time.

The pH was adjusted back to 3.08 by adding drops of a diluted solution. The particle size distribution was bimodal and contained several micron-sized agglomerates before grinding. After grinding for 24 hours, a monomodal particle size distribution of 150 nm was achieved. The final particle size distribution measured $d(0.9) = 0.262 \mu\text{m}$, $d(0.5) = 0.163 \mu\text{m}$, and $d(0.1) = 0.118 \mu\text{m}$. To remove air bubbles, degassing was carried out for 3 to 5 minutes prior to casting.

Table 1 – Process of the drying optimization test

Procedure	Samples					
	1	2	3	4	5	(T°)
Humid envir. (boiling water)	3 days	3	3	3	3	
Humid envir. (silica gel)	4 days	5	6	7	7	
Dry environment	0 day	0	0	1	2	25 °C
Steam room	5 days	5	5	5	5	50 °C
Debinding	13 hours					
Sintering	13-18 hours					
Number of days	12	13	14	16	17	
Observations	Not cracked	Not cracked	Not cracked	Not cracked	Not cracked	Bars
	Cracked	Cracked	Cracked	Not cracked	Not cracked	Plates

2.2 Natural Sintering

In our study, we opted for natural sintering due to its simplicity and cost-effectiveness. The sintering process was carried out according to a specific cycle, as described in reference [17].

Initially, the samples were subjected to gradual heating at a rate of 1.6 °C per minute until reaching the desired sintering temperature, which ranged from 1200 to 1500 °C. Subsequently, the samples were held at this temperature for a duration of either 1 hour or 3 hours, allowing for the necessary diffusion and consolidation of the material. Once the sintering process was completed, the samples underwent natural cooling within the furnace, ensuring gradual temperature reduction to avoid thermal stresses.

To facilitate further experimentation and analysis, the plates and bars were machined into bars with dimensions of $36 \times 4 \times 3 \text{ mm}^3$. To achieve a mirror-like surface, the bars underwent grinding and polishing, ensuring a smooth and reflective finish. This preparation process

was crucial to ensure accurate measurements and observations during subsequent testing and analysis.

The utilization of natural sintering, coupled with meticulous sample preparation, allowed us to explore the properties and characteristics of the materials in a cost-effective manner while ensuring reliable and consistent results.

2.3 Characterization

Scanning Electron Microscopy (SEM) played a crucial role in the analysis of the fracture surfaces of both porous and nonporous alumina samples that were created through natural sintering. This technique allowed for a detailed examination of the surface morphology and microstructural features.

To determine the overall density of the sintered samples, Archimedes' principle was employed. This principle involves immersing the samples in water and measuring the displacement of the water to calculate the sample density. For the measurement of Young's modulus, a dynamic and nondestructive method was utilized. This

property, which is sensitive to both microcracking and porosity, was determined using equation (1). The dimensions of the specimen used for this measurement were $36 \times 4 \times 3 \text{ mm}^3$. A Grindo-Sonic type device was employed to measure the frequency of the specimen's transverse vibration, from which Young's modulus was derived.

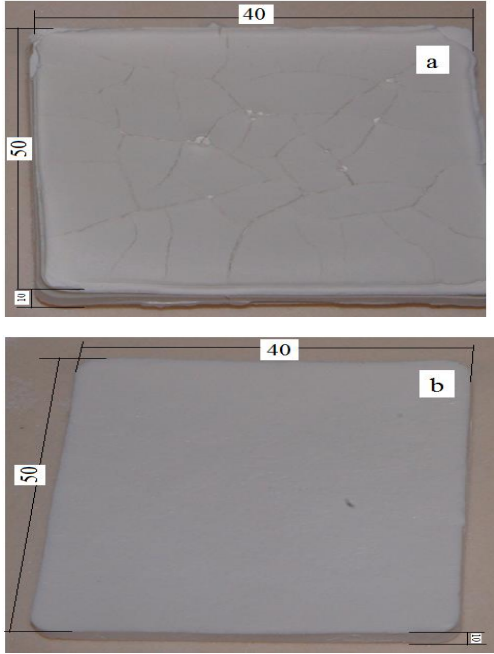


Fig. 2 – Drying state: a) not controlled ($t < 15$ days), b) controlled ($t > 15$ days)

In order to eliminate any moisture trapped within the pores, the prepared specimens underwent a steaming process at a temperature of $50 \text{ }^\circ\text{C}$ for a duration of 24 hours. This step was crucial to ensure accurate and reliable measurements of Young's modulus by eliminating any potential influence of moisture on the material's mechanical properties.

The combination of SEM analysis, Archimedes' principle, and the nondestructive measurement of Young's modulus provided crucial insights into the microstructure, density, and mechanical behavior of the sintered alumina samples.

$$E = 0,94642 \left(\frac{m}{B} \right) \left(\frac{L}{W} \right)^3 f^2 T(v), \quad (1)$$

where m refers to the mass while (W , B , L) denote the height, width, and length of the specimen, respectively. The resonance frequency is represented by f and $T(v)$ is the correction factor which is contingent on the fish coefficient and the W/L ratio. The Vickers indenter was used to measure both the hardness and stress intensity factors by applying a load of 100N . Indentations were made at the center of the meticulously polished surface of the samples. K_{10} was determined by eq. (2) as described by Anstis [18].

$$K_{10} = 0,016 \left(\frac{E}{H} \right)^{1/2} \left(\frac{P}{C^{3/2}} \right), \quad (2)$$

where, E is the Young's modulus of the material, P is the indentation load, c is the length of the crack measured from the center of the indentation, and H is the hardness.

In order to determine the stress at the point of break, a 4-point bending test was employed. This testing method required the preparation of samples in bar form with dimensions of $36 \times 4 \times 3 \text{ mm}^3$, ensuring a sufficient area for accurate stress measurements. It was crucial to maintain good parallelism and surface conditions of the samples to minimize any potential sources of error. During the testing process, a load rate of 0.5 mm/min was applied to the samples, ensuring a controlled and gradual application of force.

The stress at break, denoted as σ_R , which represents the highest stress experienced by the specimen, was calculated using equation (3). This equation provided a quantitative measure of the stress concentration at the point of failure. The 4-point bending test allowed for the assessment of the material's structural integrity and its ability to withstand applied loads. By determining the stress at break, valuable information was obtained regarding the material's mechanical strength and its resistance to fracture. The integration of this testing methodology, along with the use of precise sample preparation techniques, facilitated accurate measurement and analysis of the stress at the point of break in the tested specimens, offering insights into the material's mechanical behavior and its potential applications.

$$\sigma_R = \frac{3P_{\max}(L-l)}{2BW^2}, \quad (3)$$

here, P_{\max} is the applied load; (L , l) represent the distances between the lower and upper supports, respectively, with values of 10 and 22; B is the sample width; and W is the sample height.

3. RESULTS AND DISCUSSION

3.1 PH Effect

Fig. 2 illustrates the pH value of 4.65 after an 8-hour grinding period. The slurry necessitated a minimum of 40 hours of grinding to maintain a pH level that did not exceed 5. Nevertheless, the introduction of an inadequately diluted solution adversely affected the samples post-sintering,

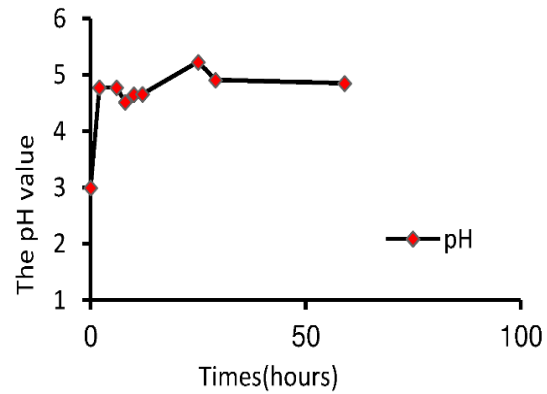


Fig. 2 – pH values over time resulting in a weaker composition. This observation underscores the importance of careful dilution during the grinding phase to ensure optimal strength in the final

sintered product.

3.2 Microstructure

The sintering temperature and dwell time played a crucial role in determining the microstructure and properties of the sintered material. At a temperature of 1200 °C, fine grain sizes ranging from 320 to 346 nm were achieved, as illustrated in Fig. 3a and b. Conversely, at a higher temperature of 1500 °C, coarse grain sizes ranging from 1430 to 1872 nm were observed, as shown in Fig. 3c and d. These grain sizes were achieved with stage durations ranging from 1 to 3 hours for each condition. When sintered at lower temperatures, there was no significant alteration in grain size compared to the average particle size of the initial powder, as depicted in Fig. 3a and b. However, porous samples sintered at 1500 °C for 3 hours exhibited noticeable grain coarsening, while still maintaining pore confinement within the grain boundaries, as depicted in Fig. 4a and b.

The process of grain size coarsening was facilitated by increasing the sintering temperature and/or dwell time, as supported by previous research [19]. This coarsening phenomenon was attributed to grain boundary migration, as demonstrated in Fig. 3c and d. Furthermore, the impact of stage duration was more pronounced at elevated temperatures, as evidenced by Fig. 5a and b.

These findings emphasize the intricate relationship between sintering parameters, resulting microstructure, and material properties, underscoring the importance of precise control over temperature, dwell time, and stage duration during the sintering process.

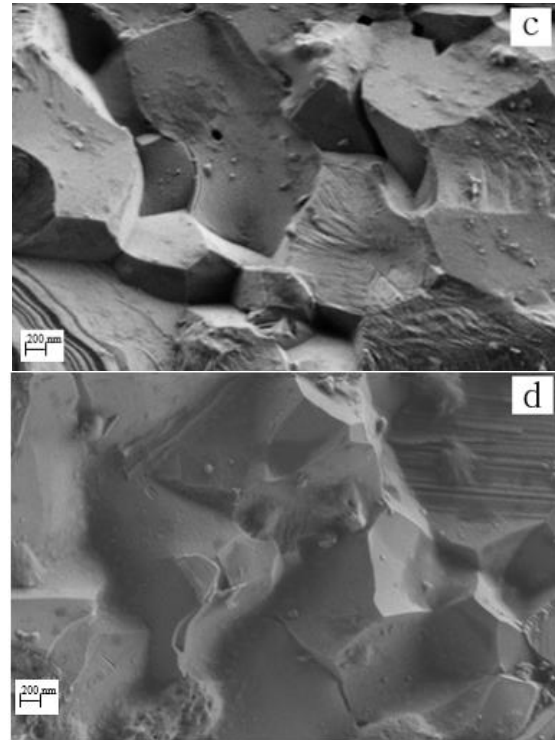
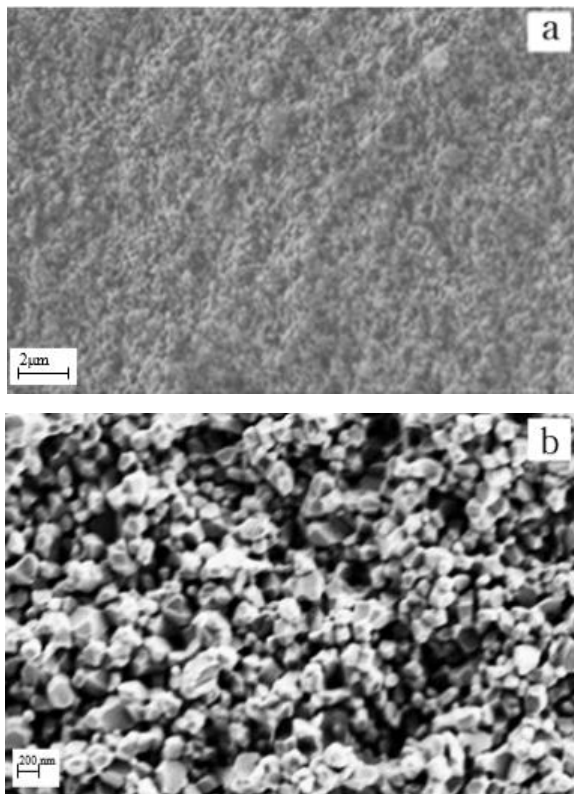


Fig. 3 – The influence of sintering temperature on grain size for microstructures achieved through natural sintering: a) 1200 °C for 1 h, grain size (Φ) = 320 nm, b) 1200 °C for 3 h, Φ = 346 nm, c) 1500 °C for 1 h, Φ = 1430 nm, d) 1500 °C for 3 h, Φ = 1872 nm

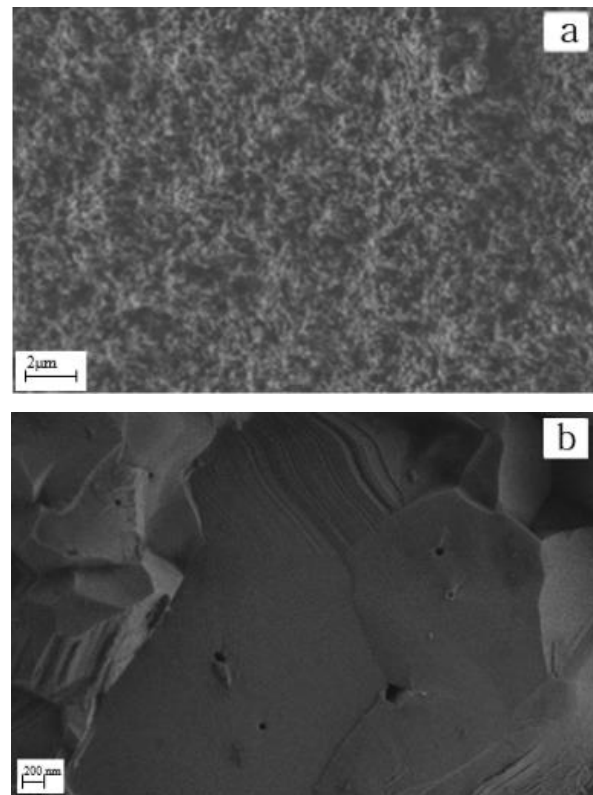


Fig. 4 – The impact of temperature on both grain size and pore size in microstructures developed via natural sintering: (a) at 1200 °C for 3 h, grain size (Φ) equals 244 nm, (b) at 1400 °C for 3 h, Φ = 1572 nm

3.3 Dwell Time Effect

Figure 6 portrays that the samples that underwent a 3-hour step time were noticeably denser compared to those that were sintered for just an hour. Particularly, a sample that was sintered at a temperature of 1400 °C for a duration of 3 hours showed a density of 100 %, implying zero porosity. This observation underscores the significance of both sintering temperature and duration in achieving optimal density and minimizing porosity. However, it was also observed that temperatures beyond 1400 °C led to an increase in porosity owing to the process of grain coarsening. This suggests that while higher temperatures can speed up the sintering process, they can simultaneously compromise the microstructure of the material by inducing grain growth. Hence, maintaining an appropriate balance between sintering temperature and time is crucial for achieving optimal material properties.

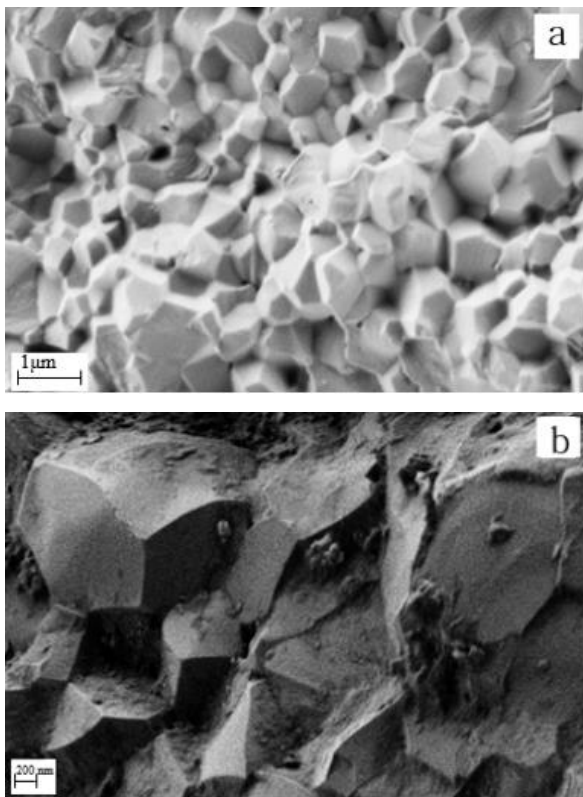


Fig. 5 – The effect of the hold time on the grain size of the resulting microstructures: a) 1400 °C/1 h, $\Phi = 1161$ nm, b) 1400 °C/3 h, $\Phi = 1666$ nm

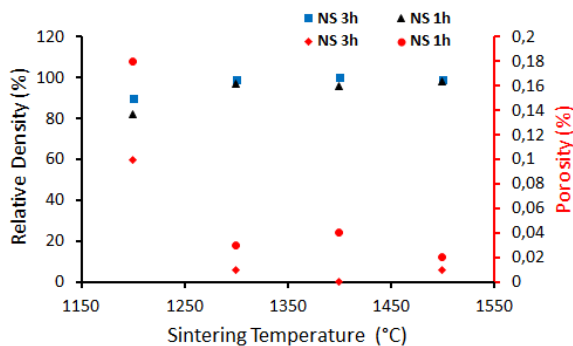


Fig. 6 – The porosity variation in relation to the sintering temperature (°C)

3.4 Young's Modulus

Figure 7 illustrates a direct correlation between grain size and Young's modulus, with an increase in grain size leading to higher values of Young's modulus. The samples sintered at 1200 °C for 1 hour exhibited both the minimum and maximum values of Young's modulus. However, for the porous samples containing 5 % wheat starch, the relationship between grain size and Young's modulus was almost monotonic, with values ranging between 118 and 144 GPa. This was primarily due to the presence of porosity, which induced a decrease in mechanical properties. Figure 8 further supports this observation, showing an inverse relationship between Young's modulus and porosity. As porosity increased, the Young's modulus decreased, as the presence of pores weakened the sample. The sample sintered at 1200 °C for 1 hour with a porosity of 0.18 % exhibited the minimum value of Young's modulus. Conversely, for the porous samples, the maximum value of Young's modulus was obtained at a porosity of 0.1 %. These findings underscore the significant impact of both grain size and porosity on the mechanical properties of the sintered material, highlighting the need to carefully control these parameters to achieve the desired Young's modulus.

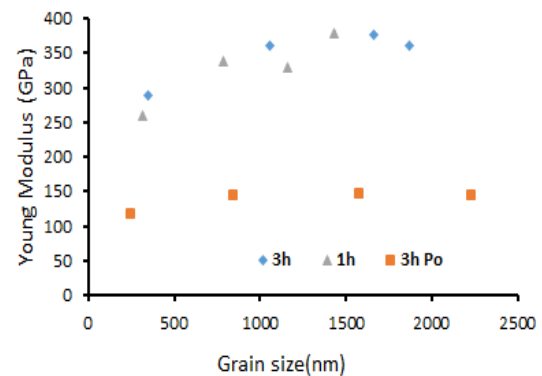


Fig. 7 – The Young's modulus variation as a function of grain size

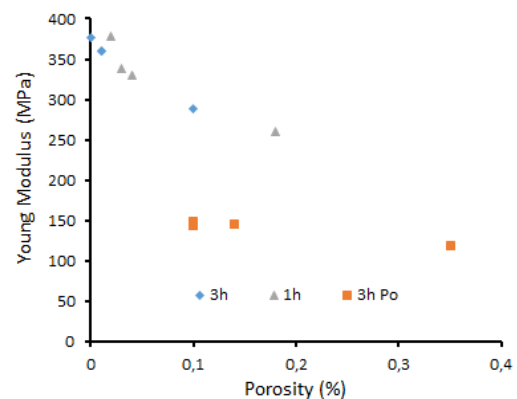


Fig. 8 – The variation in the Young's modulus in response to changes in porosity

3.5 Fracture Strength

The fracture strength of the sintered material exhibited a notable decrease as porosity increased [20]. It was observed that a lower porosity level contributed to improved mechanical strength. The maximum fracture

strength value recorded was 641 MPa, achieved under the specific conditions of sintering at 1440 °C for a duration of 3 hours. However, it should be noted that there was a dispersion in the measured values due to the random distribution of defects within the material. Conversely, the porous samples displayed lower stresses at the point of breakage. Among these samples, the minimum stress value was observed in the porous sample sintered at 1200 °C for 3 hours, measuring approximately 122 MPa, as illustrated in Fig. 9. This discrepancy in stress values further emphasizes the impact of porosity on the mechanical properties of the sintered material, with lower porosity generally leading to higher fracture strength. The relationship between porosity, fracture strength, and sintering conditions highlights the importance of carefully controlling porosity levels during the sintering process to achieve desired mechanical performance.

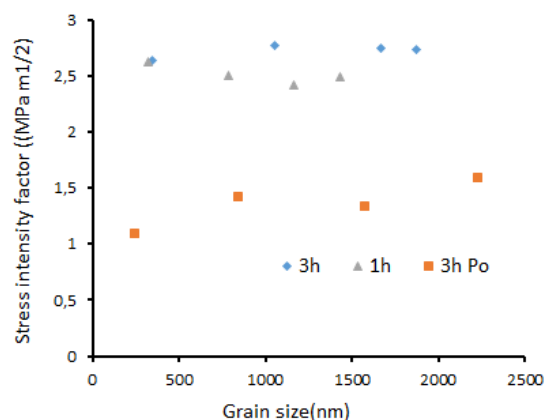


Fig. 9 – The variation in the stress intensity factor as a function of the grain size

3.6 The Stress Intensity Factor K10

Figure 10 demonstrates a clear sensitivity of the stress intensity factor to grain size in both naturally sintered and porous samples. The highest value was observed in the sample sintered at a temperature of 1300 °C for a duration of 3 hours, recording a notable value of 2.77. This finding suggests that the specific temperature and duration of sintering play a significant role in influencing this factor. In contrast, the porous samples exhibited lower values, ranging between 1.1 and 1.59. This indicates that the presence of porosity can significantly affect the material's resistance to stress propagation. Therefore, while sintering conditions can be manipulated to optimize the stress intensity factor, the

inherent porosity of the sample must also be considered to achieve desirable mechanical properties. This highlights the need for fine-tuning these parameters to optimize the performance of sintered materials.

4. CONCLUSION

The outcomes of this study can be distilled into several key findings. Firstly, the processing conditions applied precipitated the formation of diverse microstructures. These ranged from fine to coarse, demonstrating the extent to which the processing parameters can influence the resultant microstructural characteristics of the sintered samples.

Furthermore, the fracture surfaces of samples created under various conditions were meticulously examined using Scanning Electron Microscopy (SEM). This allowed for an in-depth evaluation of the microstructural features and provided key insights into the influence of processing conditions on microstructural evolution. The observations made through SEM were instrumental in the subsequent analysis of the microstructures.

The porosity of the nonporous samples varied based on the holding duration during the sintering process. For samples held for 1 hour and 3 hours, the porosity ranged between 0.01 % and 0.18 %, and 0.01 % and 0.1 % respectively. In contrast, the porous samples had a porosity that oscillated between 0.1 % and 0.35 %, highlighting the substantial influence of processing conditions on porosity.

In terms of densification, samples that were held for 3 hours exhibited superior results compared to those held for 1 hour. Remarkably, zero porosity was achieved at a temperature of 1400 °C over a duration of 3 hours. However, it was noted that above 1500 °C, the density reduced due to grain coarsening, indicating a limit to the effective sintering temperature for densification.

The study also observed a noticeable decline in Young's modulus, a property related to the stiffness of a material, as porosity in the porous samples increased. This highlighted the detrimental effect of porosity on the material's mechanical properties and reinforced the need to control porosity during sintering.

Lastly, the sintered samples kept for 3 hours demonstrated a fracture strength between 352 and 641 MPa, which was higher than that of the samples sintered for 1 hour or 3 hours. The latter varied respectively from 164 to 528 MPa and from 122 to 439 MPa. This underscores the fact that longer holding durations can improve fracture resistance, thus enhancing the overall performance of the sintered samples.

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Умови спікання та їх вплив на механічні властивості оксиду алюмінію (α - Al_2O_3)

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Стаття присвячена дослідженню впливу умов спікання на оксид алюмінію, отриманого шляхом природного спікання. Нанопорошок, використаний як вихідний матеріал, був α -оксидом алюмінію (α - Al_2O_3), отриманим від Bai-kowski (Baikalox-ВМА15, Франція), із чистотою 99,99 % і середнім розміром частинок 150 нм. Для приготування шликера використовувався вологий процес, при цьому 24 години сприяли деагломерації 50 г порошку оксиду алюмінію. Щоб усунути потенційне забруднення, шликер був відлитий у ПВХ-форми та розміщений на підкладці з оксиду алюмінію. Комплексний цикл сушіння був ретельно проведений, щоб зменшити ймовірність розтріскування. Це включало такі етапи, як кип'ятіння води, силікагель, висушування середовища, обробка в печі та, нарешті, піч. Щоб звести нанівець вплив органічних добавок і залишкових газоподібних форм, шликер пройшов процес роз'єднання при 600 °C. Процес спікання виконувався при змінних температурах від 1200-1500 °C, з інтервалами 100 °C, і тривалістю витримки 1 год. і 3 год. Пристрій Grindo-Sonic застосовувався для вимірювання модуля Юнга за допомогою динамічного методу, тоді як механічна міцність була визначена за допомогою випробування на вигин у чотирьох точках. Зразки, спечені протягом 3 годин і 1 години, продемонстрували міцність на розрив 352 і 641 МПа і від 164 до 528 МПа відповідно. Крім того, пористі зразки виготовляли з використанням 5 % пшеничного крохмалю як пороутворювача. Пористість протягом 1 години та 3 годин змінювалася між 0,01 %-0,18 % та 0,01 %-0,1 % відповідно, тоді як для пористих зразків пористість коливалася в межах 0,1 %-0,35 %. Результати показали, що зразки, спечені протягом часу витримки 3 години, були щільнішими, ніж ті, спечені протягом 1 години. 100 % щільність зразка була досягнута при 1400 °C протягом 3 годин. Аналіз показав, що більша тривалість витримки 3 години призвела до кращих механічних властивостей.

Ключові слова: Альфа-глинозем, Пороген, Природне спікання, Мікроскопічна структура.