

Research and Engineering Project

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Study of the effect of the electric field on the reactive sintering of an Aluminum Oxynitride (AlON)

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Summary

English

Ceramics are non-metallic, inorganic materials like clay, minerals, and oxides. They are typically hard, chemically stable, and can withstand high temperatures and pressures. Ceramics generally have poor electrical conductivity. Their properties depend on their atomic structure, the types of atoms, and how these atoms are bonded and arranged. Ceramics are used in both traditional and advanced applications such as aerospace, biomedical, electronics, and energy. One important example is AlON, a type of transparent ceramic.

γ -AlON is a solid solution of Al_2O_3 and AlN, produced by methods including carbonization, solid-state reaction, or chemical vapor deposition. It shares similar strength, hardness, and optical qualities with these materials but offers more flexibility in size and shape.

Sintering is a process that forms a solid mass by applying heat and pressure without melting the material. It also enhances material properties.

This study focuses on the effect of an electric field on the reactive sintering of AlON. Electricity is passed through the matrix or material to fuse powder particles. The Flash Sintering method will be used, allowing densification at lower temperatures and shorter times.

Parameters such as temperature, sintering time, pressure, electric current, and atmospheric composition will be varied to optimize transparency in the final product.

French

Les céramiques sont des matériaux inorganiques non métalliques tels que l'argile, les minéraux et les oxydes. Elles sont généralement dures, chimiquement stables et peuvent résister à de hautes températures et pressions. Les céramiques ont généralement une faible conductivité électrique. Leurs propriétés dépendent de leur structure atomique, des types d'atomes et de la manière dont ils sont liés et arrangés. Les céramiques sont utilisées dans des applications traditionnelles mais aussi avancées, comme l'aérospatiale, le biomédical, l'électronique et l'énergie. Un exemple important est l'AlON, un type de céramique transparente.

Le γ -AlON est une solution solide d' Al_2O_3 et d'AlN, produite par des méthodes telles que la carbonisation, la réaction à l'état solide ou le dépôt chimique en phase vapeur. Il possède une résistance, une dureté et des qualités optiques similaires, tout en offrant plus de flexibilité en taille et forme.

Le frittage est un procédé qui forme une masse solide en appliquant chaleur et pression sans faire fondre le matériau. Il améliore aussi les propriétés du matériau.

Cette étude se concentre sur l'effet d'un champ électrique sur le frittage réactif de l'AION. L'électricité est envoyée à travers la matrice ou le matériau pour fusionner les particules en poudre. La méthode de frittage flash sera utilisée, permettant la densification à des températures plus basses et en moins de temps.

Les paramètres tels que la température, le temps de frittage, la pression, le courant électrique et la composition atmosphérique seront variés pour optimiser la transparence du produit final.

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CHAPTER I:

Bibliographic Study

I.1. Introduction

The main objective of this project is to understand the material under study, analyzing its various properties, characteristics, and industrial and/or technological applications. Subsequently, to understand the concept of sintering and learn about existing sintering techniques. Finally, to study the effect of applying an electric field on the AlON sintering process.

I.2. AlON (Aluminum Oxynitride)

AlON is a transparent polycrystalline ceramic with a cubic spinel structure composed of aluminium, oxygen and nitrogen. Also called “transparent aluminum”, has his origins in the early 1950s but the correct synthesis and refinement was reached in the late 20 th century. It arose from the need for highly resistant and optically transparent materials in the defense and aerospace industries.

Nowadays, it is being tested in numerous industries and continues to gain traction as researchers explore innovative manufacturing methods.

I.2.1 Compounds

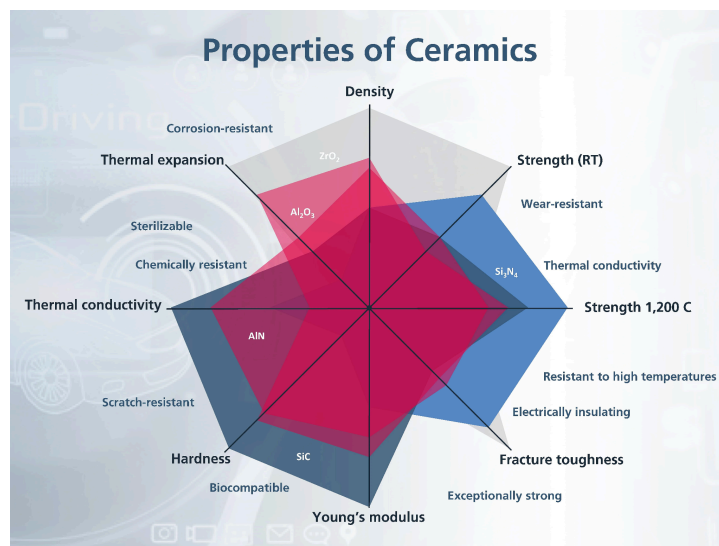


Figure 1. Properties of ceramics

Aluminum Nitride (AlN)

AlN is an excellent material to use if high thermal conductivity and electrical insulation properties are required, which makes it an ideal material for use in thermal management and electrical applications [6]. Moreover, it is a common alternative to Beryllium Oxide (BeO) in the semiconductor industry as it is not a health hazard when machined.

1) Production

It can be obtained by dry pressing and sintering, or by hot pressing with appropriate sintering aids. The result of these processes is a material that is stable at high temperatures in various inert environments, such as hydrogen and carbon dioxide atmospheres.

2) Structures and orientation

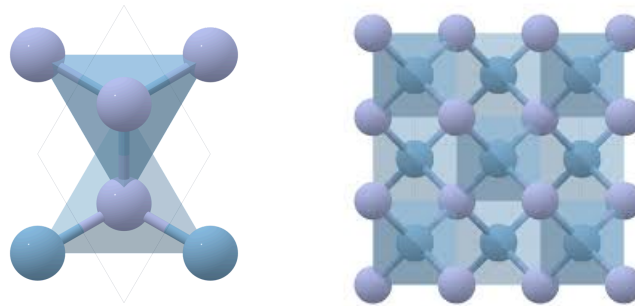


Figure 2. Crystal structure of AlN

AlN has a wurtzite structure (see figure 2) and crystallizes in the hexagonal space group $P6_3mc$ [6]. The wurtzite structure consists of two hexagonal close-packed (HCP) lattices, each containing one of the two types of atoms, interconnected along the direction of the principal axis of symmetry of the hexagonal prism, with a $5/8 c$ spacing [20].

Aluminum has three electrons in its valence shell, distributed as $3s^2 3p^1$. Nitrogen, on the other hand, has five electrons in its valence shell, distributed as $2s^2 2p^3$. In the excited state, the $3s^2 3p^1$ sublevels of aluminum give rise to four $3sp^3$ hybrid sublevels[29].

Al^{3+} is bonded to four equivalent N^{3-} atoms to form vertex-sharing AlN_4 tetrahedra. There are three shorter Al-N bonds ($1.90 \text{ \AA}$) and one longer one ($1.91 \text{ \AA}$). N^{3-} is bonded to four equivalent Al^{3+} atoms to form vertex-sharing NA_4 tetrahedra[6].

Regarding orientation, the vibrational phononic modes of the AlN film, active in the IR, are related to the respective covalent bonds. Furthermore, they absorb energy according to the crystal orientation of the AlN film and the polarization of the IR beam electric field, so this characteristic can be used to identify the film's preferred orientation. In IR analysis, the

beam's electric field is polarized parallel to the thin film surface, i.e., the incident electric field is perpendicular to the c axis.

3) Characteristics

AlN has properties, mentioned in [33][6], such as electrical insulation (good dielectric properties) and a coefficient of thermal expansion similar to Silicon. For this reason, it is useful for electronic applications with problems of high temperatures and heat dissipation. In addition, good corrosion, abrasion and oxidation resistance, stability in semiconductor processing atmospheres, and good mechanical resistance.

Some of his advantages are high thermal conductance (over five times compared to Alumina), no toxic issues, good plasma resistance, excellent thermal shock performance and enables high performing devices to work faster and better in smaller devices. However, AlN does not occur naturally and must always be synthesized from a precursor material such as Al_2O_3 [9].

4) Applications

- AlN-based PMUT: This uses piezoelectric phenomena for electroacoustic conversion, ultrasonic transmission, and reception over a wide frequency range. A circular PMUT composed of multiple electrodes was proposed for differential transduction. The design comprises an AlN layer with Al electrodes and various elastic layers.
- AlN-based microphones: Low dielectric loss, high intrinsic signal-to-noise ratio, high stiffness, and a high figure of merit for sensitivity are attractive properties of aluminum nitride (AlN). The low dielectric loss of AlN is a crucial advantage over PZT and ZnO for applications in airframe arrays, because it results in improved low-frequency microphone response. Therefore, AlN has been used in aeroacoustic applications [14].
- Industrial field: AlN acts as a sensor to identify pressure changes by generating a potential difference under the applied external voltage, based on the polarized c axis, thanks to its non-centrosymmetric crystalline structure.

Aluminum Oxide/Alumina (Al_2O_3)

Aluminum oxide (alumina) is the most widely used oxide ceramic material and has very diverse applications. In addition, it is one of the most abundant minerals on Earth, constituting a significant portion of the Earth's crust. It occurs naturally in the form of bauxite.

1) Production

It is obtained from bauxite using the Bayer process, followed by calcination. This process involves digesting bauxite ore, with a maximum of 55% alumina, in a hot sodium hydroxide solution. At temperatures close to 175°C, the aluminum dissolves and forms sodium aluminate. The high-purity alumina is filtered from the compound, and the resulting solution is transferred to a precipitator tank for cooling [27]. Upon cooling, the solution is filled with aluminum hydroxide ($\text{Al}(\text{OH})_3$) crystals. These crystals are then transferred to a kiln for calcination at temperatures close to 1260°C.

2) Structures and orientation

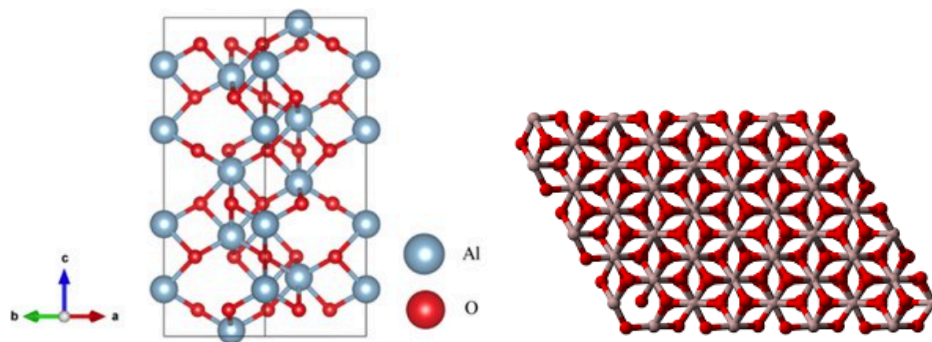


Figure 3. Crystal structure of Al_2O_3

It has a unique crystal structure that significantly influences its properties and applications (**see figure 3**). In its most stable form, alpha alumina, it adopts a hexagonal close-packed (HCP) structure consisting of densely packed layers of oxygen atoms, with aluminum ions occupying the octahedral sites between these layers [10]. This specific crystal structure gives aluminum oxide exceptional hardness and thermal stability. Other crystal forms also exist, such as the beta and gamma phases. The beta phase has a cubic crystal structure that exhibits distinctive properties compared to the alpha phase [37]. On the other hand, the gamma phase is characterized by a unique, partially disordered structure, which gives it specific catalytic properties.

3) Characteristics

It is extremely robust, with high hardness and great resistance to abrasion, corrosion, and wear. It also exhibits high resistance to thermal shock and chemical attack, even at high temperatures. Therefore, it stands out as the preferred material for low-cost production and manufacturing. It has a relatively high density and excellent electrical insulation properties, due to its dielectric strength, and thermal insulation properties.

4) Applications

- Petrochemical field: The exceptional chemical inertness of pure alumina makes it ideal for use in both oxidizing and reducing atmospheres, making it a key material for multiple applications in industrial petrochemicals.
- Industrial field: It is widely used as a filler in polymers, reinforcing their mechanical properties. In the glass industry, it improves the transparency, strength, and durability of products.

I.2.2 Structure

AlON is a transparent polycrystalline ceramic with a cubic spinel crystal structure composed of aluminum, oxygen, and nitrogen. It belongs to a class of complex oxides with the general chemical formula AB_2O_4 , where the A ions are typically cations. These are isometric cubic crystals (see figure 4).

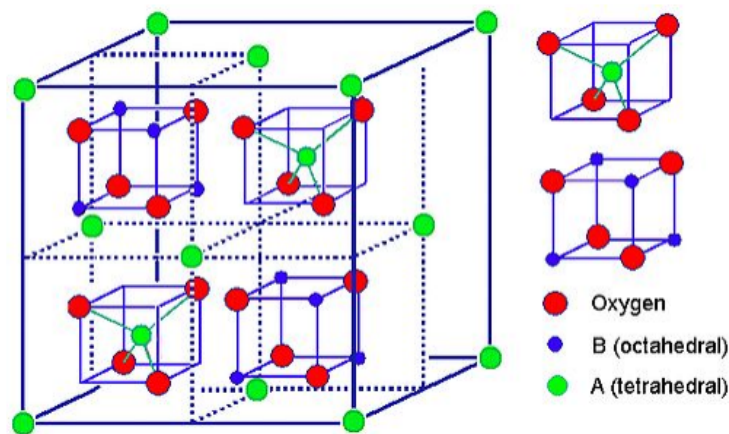


Figure 4. Crystal structure of complex oxides

To begin, it's important to understand the spinel structure upon which AlON is based. The spinel structure is represented by the formula MM'_2X_4 , where M and M' are cations coordinated tetrahedrally and octahedrally, respectively, and X is an anion (typically oxygen or fluorine). There are two types of spinel structures: normal and inverse.

In the normal spinel structure, there is a densely packed array of anions. The A-site cations occupy 1/8 of the tetrahedral holes, while the B-site cations fill 1/2 of the octahedral holes. Each unit cell contains eight formula units, resulting in a composition of $A_8B_{16}O_{32}$ (see figure 5).

Conversely, the inverse spinel structure has a closely related arrangement where the A-site cations and half of the B-site cations switch places. In this configuration, all A cations and half of the B cations occupy octahedral sites, while the remaining half of the B cations occupy tetrahedral sites (see figure 5).

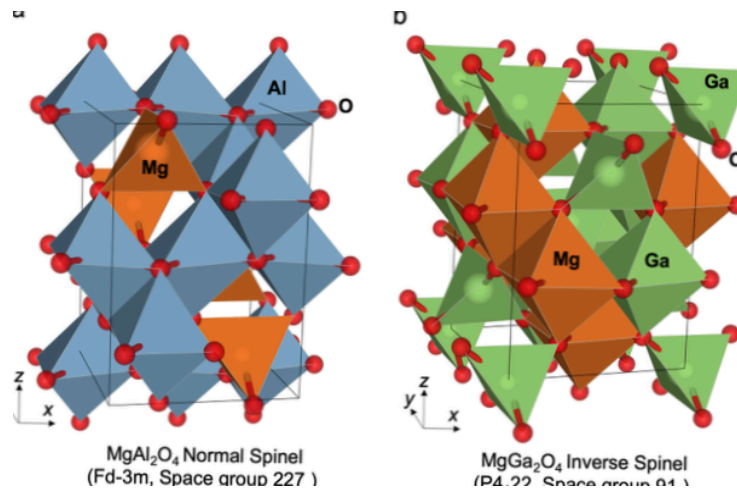


Figure 5. Normal/inverse spinel crystal structure

There are also mixed espinelas, which are intermediate between the normal and inverted structures of the espinela.

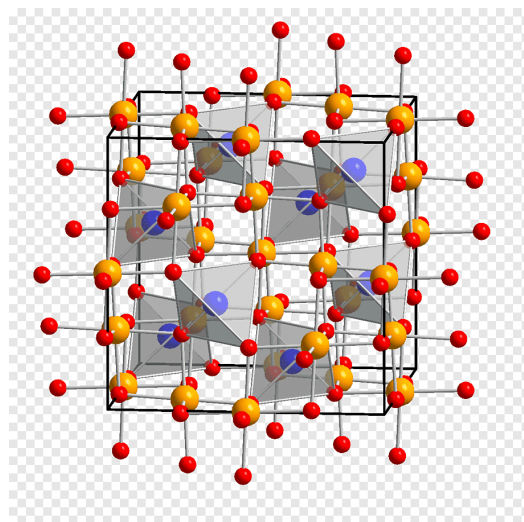


Figure 6. Crystal structure of AlON

The cubic spinel-type structure of AlON (see figure 6) has a lattice constant of approximately 7.95 Å. It is formed by replacing oxygen atoms with nitrogen atoms, resulting in an approximately cubic close-packed structure. About one-third of the aluminum atoms are in

tetrahedral coordination, while the remaining aluminum atoms occupy octahedral sites, with one site left vacant. In other words, the 32 e^- sites in the spinel crystal structure are fully occupied by O and N atoms (red), while the Al atoms (orange) occupy the tetrahedral (Al(IV)) and octahedral (Al(VI)) sites, with one vacancy between them [31].

The spinel-type aluminum oxynitride solid solution (γ -AlON) exists in the Al_2O_3 -rich region of the AlN – Al_2O_3 systems. The anionic crystal structure of γ -AlON can be defined as a solid solution of the γ - Al_2O_3 spinel phase [34]. The lattice parameter does not follow a linear relationship with the nitrogen content in γ - Al_3O_3N , deviating from what Vegard's law predicts. As the nitrogen content increases, the anions expand along the (1 1 1) direction, causing shifts in diffraction peaks and a displacement toward lower energies in the electronic density of states. Compressibility improves due to the expansion of coordination polyhedra, but the elastic properties only show slight enhancement. This suggests that improving the mechanical properties of γ - Al_3O_3N solely by increasing the nitrogen content is not straightforward. These results contribute to a better understanding of the structure and properties of γ - Al_3O_3N with varying nitrogen content.

The structure of spinel-type compounds can be modified by changing the elemental species and the ratio of cations to anions [23], making it an important factor to consider for achieving optimal optical properties.

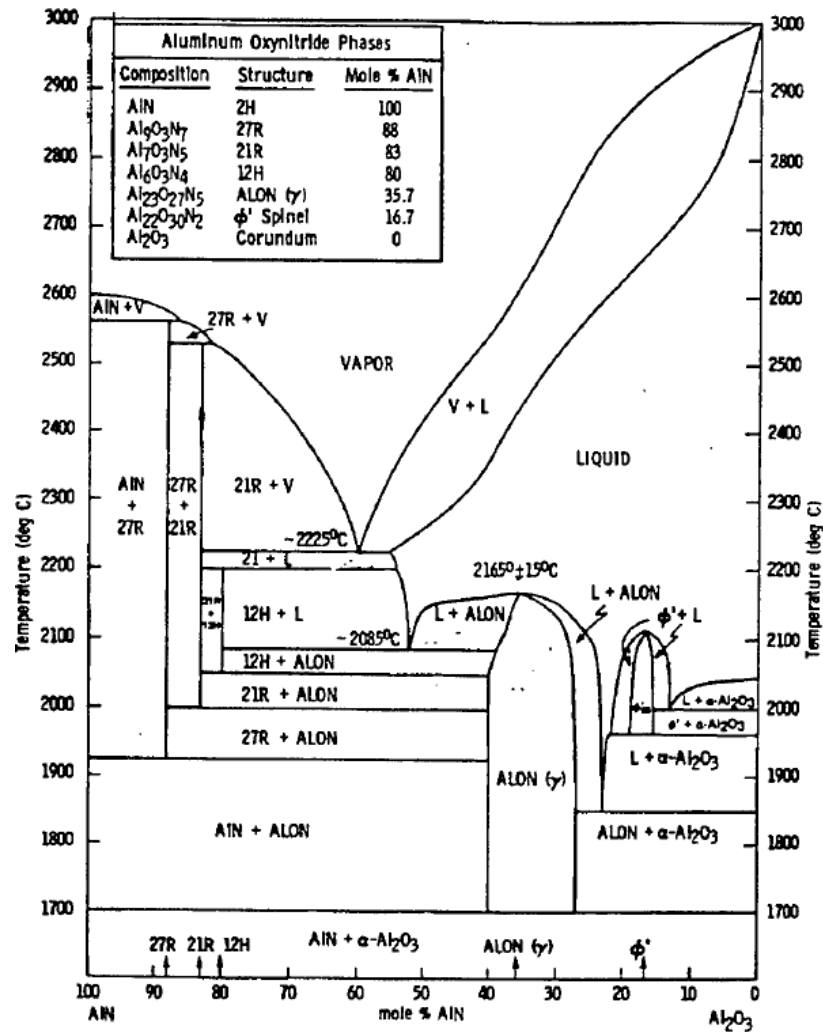


Figure 7. Phase diagram of AlON

To obtain dense and optically transparent ceramics, interest is focused on the single-phase domain of γ -ALON, since outside this compositional range, the resulting material does not present a single phase and its crystalline structure is no longer cubic, which compromises its optical and mechanical properties (see figure 7) (see table 1) .

Table 1. Proportions and regions of the AlON phase diagram

Region	Proportion Al ₂ O ₃ :AlN	Compound
AlON-Al ₂ O ₃	13:1	Φ- Al ₂ O ₃
	9:1	δ-Al ₂ O ₃
	10:2	ξ-Al ₂ O ₃ or Φ'- Al ₂ O ₃
AlON	9:5	Al ₂₃ O ₂₇ N ₅ = AlON
AlN Polytypoids	1:4	12H
	1:5	21R
	1:7	27R
	1:14	32H

I.2.3 Properties

Physical properties

- 1) **Mechanical Properties:** This material stands out for its high strength, being capable of withstanding bullet impacts. It is approximately four times harder than fused silica glass, slightly harder than magnesium aluminate spinel, and nearly as hard as sapphire. It exhibits excellent resistance to wear, abrasion, and compression, allowing it to remain intact under extreme conditions. However, despite its hardness, brittleness can be a limiting factor in certain applications.
- 2) **Melting Point:** It has a very high melting point due to the strong covalent bonds between aluminum, oxygen, and nitrogen atoms. Its melting point is generally close to that of alumina (Al₂O₃), around 2050 °C, although it may vary slightly depending on nitrogen concentration. It also remains structurally stable at temperatures up to approximately 1200 °C.
- 3) **Optical Transparency:** The material is transparent in both the visible and infrared regions of the electromagnetic spectrum. In areas near the ultraviolet and infrared regions, it can reach transparency levels above 80%.
- 4) **Low Relative Density:** Despite its strength, it is a relatively lightweight material thanks to the presence of vacancies in its crystal lattice. Its density may vary depending on nitrogen content.
- 5) **Refractive Index:** It has excellent refractive performance in homogeneous regions, making it suitable for high-precision optical applications.
- 6) **Thermal Properties:** It features good thermal conductivity and acts as a dielectric

material with a wide band gap (around 6.2 eV). It can withstand high temperatures without losing its structural or functional properties.

7) **Acoustic Properties:**

It has a high sound propagation velocity, which indicates a rigid and dense structure, suitable for advanced acoustic applications.

Chemical properties

- 1) **Chemical Stability:** The material is extremely resistant to corrosion and oxidation, even in harsh environments. The presence of nitrogen enhances its resistance to chemical degradation compared to pure alumina (Al_2O_3), particularly in environments where acidic gases are present.
- 2) **Resistance to Acid and Base Attack:** It shows significant resistance to both strong acids and strong bases, maintaining structural integrity under chemically aggressive conditions.
- 3) **Reactivity with Water:** Like alumina, it is minimally reactive with water, which provides high stability in aqueous or humid environments.
- 4) **Behavior with Gases:** It can react with certain gases, especially at elevated temperatures. Nitrogen may partially substitute for oxygen, influencing the material's electronic structure and properties.
- 5) **Compatibility with Metals:** As a ceramic material, it does not form alloys or homogeneous mixtures with metals under standard conditions. However, at high temperatures, it may interact with certain metals depending on the specific chemical environment.
- 6) **Reactivity with Oxygen:**
In the presence of oxygen, the material remains stable. The combination of oxygen and nitrogen in its crystal structure prevents easy decomposition. However, under extreme conditions of temperature and pressure, some reactions may occur that alter its structure.

I.2.4 Applications

Thanks to the excellent optical transparency, strength and hardness, ALON optical ceramic has emerged as the material of choice for a number of military and commercial applications.

Optical Applications

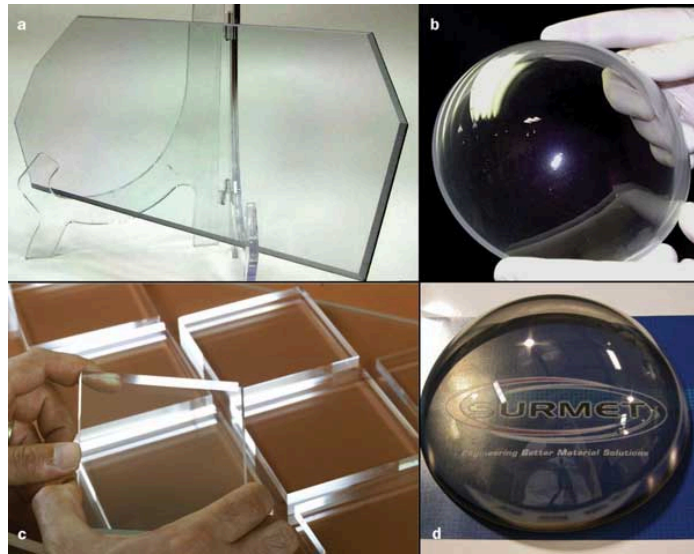


Figure 8. Optical applications of ALON

IR Windows and Domes: windows, domes, and lenses used by many major defense companies. ALON optical components are now being used in such diverse applications as laser windows for underwater towed arrays, lenses, missile domes and FLIR windows/domes. Several ALON prototype domes have been produced. A significant cost component of finished domes is in the fabrication process. To minimize the cost of the domes, the blank yields must be taken into consideration. Fixturing considerations also play a part in set up time for thin domes. Detailed cost savings have been demonstrated for the fabrication of ALON domes versus sapphire domes. The largest challenge facing polycrystalline optical ceramics is the ability to produce large components of high optical quality and uniformity. On the other hand, the powder process is only limited by the size of the available equipment.

Defense Applications

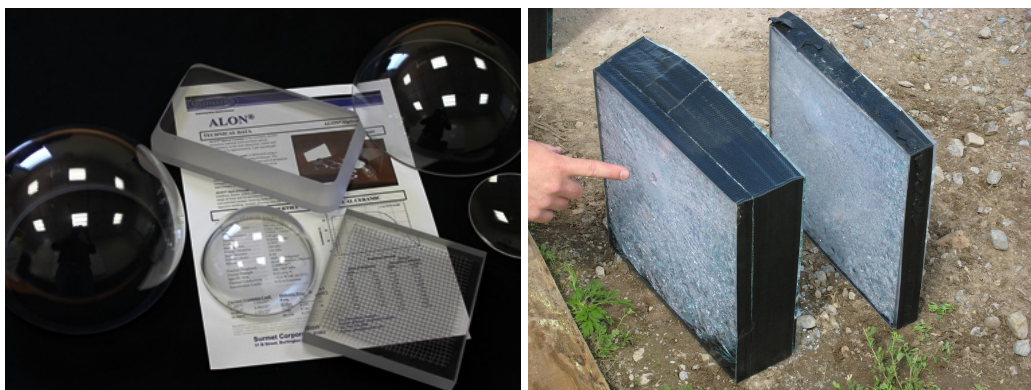


Figure 9. Defensive applications of ALON

Transparent Armor: In terms of volume, the largest potential market for ALON is transparent armor. These include applications on land vehicles as well as aircraft and water craft. When compared to conventional glass/plastic laminates used against armor piercing rounds, ALON transparent armor provides equivalent protection at half the weight and thickness. These results indicate a potential for hundreds of pounds of weight savings per vehicle.

Furthermore, ALON transparent armor has demonstrated superior multi-hit performance against armor piercing threats. There are currently needs for large armor transparencies for land, air, and sea based vehicles. Initial process development was conducted to manufacture flat windows out of which the curved window was fabricated. To save costs the forming and processing of curved window blanks is also under development. This will save on material by reducing waste material usage. It will also decrease generation time and costs.

Commercial Applications



Figure 10. Commercial applications of ALON

ALON products are being manufactured for use and evaluation in a number of commercial applications. The high hardness and durability of ALON make it suitable for use as scanner windows seen in everyday use at supermarkets and other retail stores. Scratch and impact resistance, as well as optical clarity and low cost, are essential. ALON continues to find new applications in semiconductor manufacturing equipment because of its outstanding chemical resistance and its ability to be manufactured in complex shapes and in the large sizes required for these components (plasma delivery tubes, wafer carriers, shower heads, and process enclosures) . ALON has demonstrated excellent durability when exposed to many of the corrosive chemical processes used in the electronics industry. ALON not only offers a more economical option than sapphire, but is also a more etch-resistant alternative than quartz in various applications. This means that, although the purchase price of ALON may be higher than that of quartz, its total long-term cost could be lower due to its much longer lifespan in corrosive environments.

I.3. Sintering

Sintering is a manufacturing process used to produce various components from mixtures of metallic or ceramic powders [30]. It involves the thermal transformation of bulk materials into compact solids at temperatures below their melting point. The key phenomenon in sintering is solid-state diffusion, which occurs along crystal interfaces and through vacancies. During the process, significant changes take place in the microstructure.

For successful sintering, the material must be homogeneous. One advantage is that the powder used in sintering never reaches a liquid phase [28]. Additionally, several parameters must be carefully controlled. Variations in temperature, sintering time, pressure, and atmospheric composition directly influence the characteristics of the final product.

Sintering occurs naturally in mineral deposits and is widely used as a manufacturing process for materials such as ceramics, metals, and plastics.

Sintering stress is the driving force behind densification. It depends on surface tension, relative density, particle size and shape, as well as grain and pore size, all of which change with the microstructural evolution during the sintering process.

Sintering is employed to enhance various material properties, including thermal and electrical conductivity, strength and structural integrity, and translucency.

The duration of the process varies depending on several parameters, as noted in [32], and can range from just a few milliseconds to over 24 hours.

I.3.1 Steps

Can be described as an interval of geometric change in which the pore shape change is totally defined or an interval of time during which the pore remains constant in shape while decreasing in size. Below are the stages of the process (**see figure 11**) , studied in [18]:

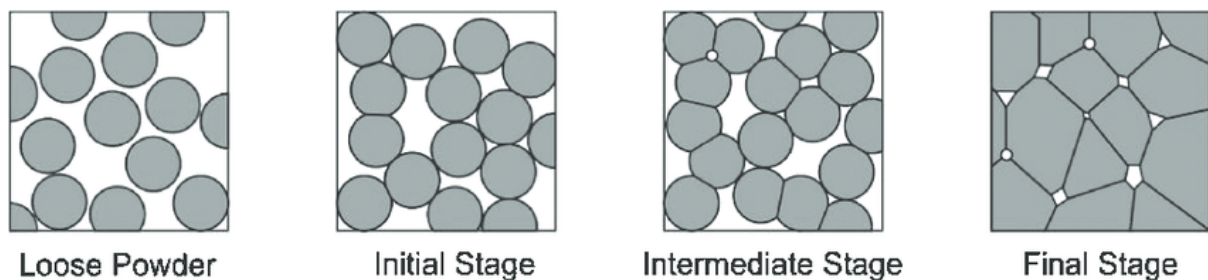


Figure 11. Stages of the sintering process

Initial Step

This is the point-contact stage and does not produce dimensional changes. Initially, it causes the particles in contact to form grain boundaries at the contact points through diffusion. The higher the initial compaction density, the greater the material's coherence. In this initial stage of sintering, necks begin to form at the contact points between adjacent particles. No dimensional changes are observed, and porosity does not decrease. Neck formation is driven by the energy gradient resulting from the different curvatures of the particles and the neck. Mass transport occurs through surface diffusion during the early stages of neck growth.

Intermediate Step

This stage begins when adjacent necks start to intersect, typically when the ratio $D/2R$ reaches 3.0. During this phase, densification and grain growth occur. The packing density and coordination number of the green body are crucial in this stage. A high green packing density enables rapid sintering with relatively few pores in the final object.

Pore channel closure takes place, where interconnected pore channels are sealed off, isolating the porosity due to neck growth. Another contributing factor is the formation of new contact points as a result of pore contraction within the pores themselves.

Very low green packing densities, often associated with low coordination numbers, can lead to grain coarsening without densification. In extreme cases, this may result in open-pore structures that lack structural integrity.

Initially, the pores form a network of interconnected cylindrical channels interrupted by necks. By the end of this stage, the pores become smoother and begin to narrow and isolate from one another. Bulk mass transport mechanisms dominate, causing material to migrate from within the particles to their surfaces, resulting in flattening and densification at points of contact.

Final Stage

This stage begins when most of the pores have closed. As sintering progresses, the pores, which formed an interconnected network during the intermediate stage, become isolated from one another. This final stage is much slower than the initial and intermediate stages.

As grain growth continues, pores tend to detach from grain boundaries and become more spherical. Pore shrinkage is the most critical aspect of final-stage sintering. Solid material must be transported to the pores, and there must be a path for the gas inside the pores to escape to the surface. As a result, the overall volume of the sintered mass decreases.

Smaller pores are eliminated, while larger pores may grow, a process known as Ostwald ripening. In some cases, pore growth during the final stage of sintering can actually reduce the density, as gas pressure in larger pores can inhibit further densification.

I.3.2 Types

There are several types of sintering, depending on the material being bonded or the specific sintering process used (see figure 12) .

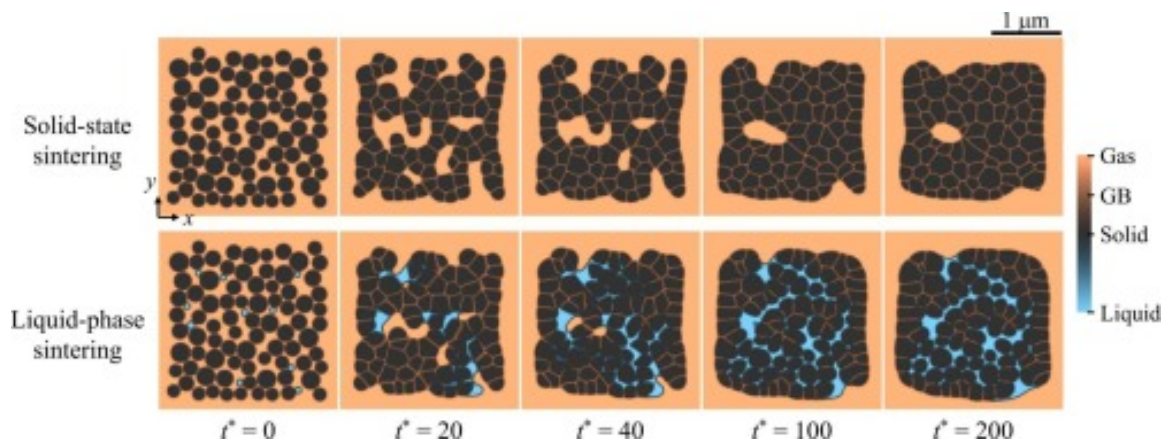


Figure 12. Solid and liquid phase sintering

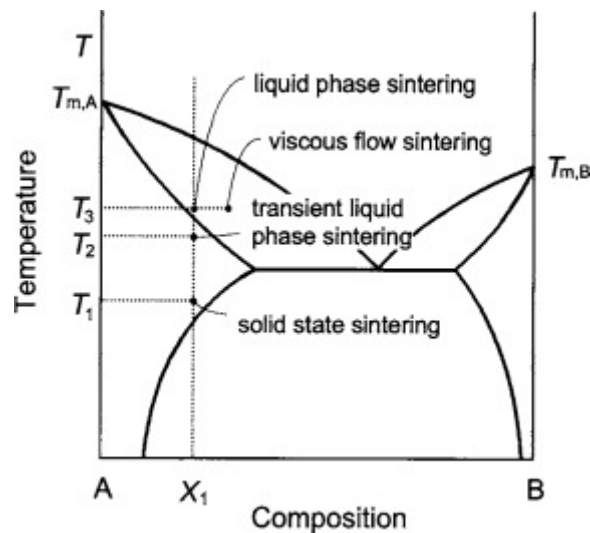


Figure 13. Composition/temperature diagram of solid and liquid phase sintering

Solid-State Sintering

This is the most common sintering process and generally involves heating powdered materials below their melting point, allowing them to bond and form a solid structure [11]. In this case, surface energy is usually a relatively small driving force.

As mentioned in [8], this method offers several advantages, such as low synthesis cost, high productivity, and a straightforward synthesis procedure. However, it also has drawbacks, including the tendency to form impurities and the typically higher temperatures required to obtain a single-phase ferrite.

Liquid-Phase Sintering

Liquid-phase sintering involves adding an additive to the powder to be sintered. This additive melts, and the resulting liquid is absorbed into the pores, causing the grains to rearrange into a more favorable packing configuration. When capillary pressures are high and particles are close together, atoms dissolve and precipitate in regions of lower chemical potential.

For the process to be effective, the additive must melt before sintering begins. Additionally, good wettability between the solid and liquid phases is essential.

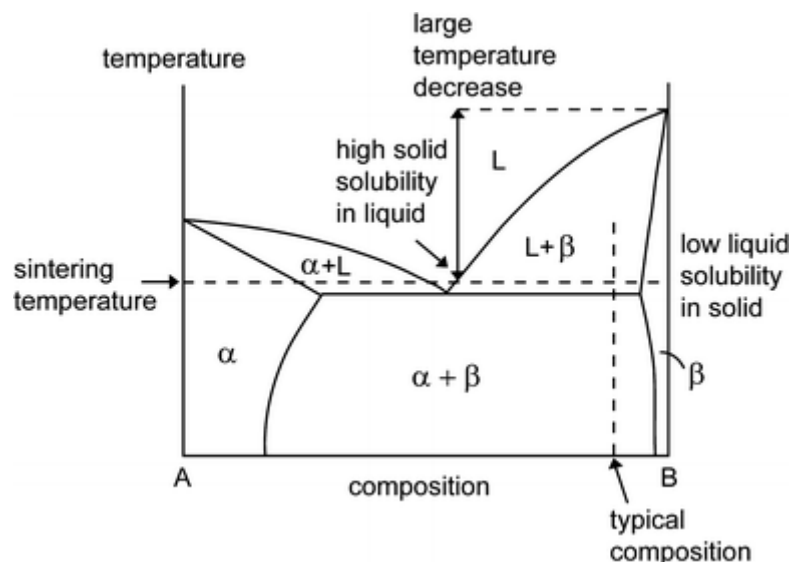


Figure 14. Composition/temperature diagram of liquid phase sintering

- 1) Liquid phase permanent

Maintains the liquid phase throughout the ceramic sintering process, improving particle bonding and material properties [11]. Promotes capillarity to draw liquid into open pores, resulting in grain movement and improved packing.

2) Transitory liquid phase (TLPS)

Consists of a temporary liquid phase that is solidified during the process. It is used to produce large-scale products made of ceramics, metals and metal-matrix ceramics. These materials must be mutually soluble, wetting the liquid to the solid and creating a high diffusion rate. It supports lower sintering temperatures and thinner microstructures, resulting in better material properties [11].

Pressure assisted

A process in which high densification is achieved by the application of external pressure during sintering. The temperature can be reduced to almost half the melting point of the ceramic. In addition, differential sintering can be avoided by using pressure assisted sintering, which is critical for maintaining microstructure and mechanical properties. It differs from traditional sintering in that, apart from using heat, it also introduces mechanical pressure, which compacts the material more effectively.

I.3.3 Concepts

Driving Forces

Sintering is driven by various forces and mechanisms that help bind and densify the powder particles to form a solid structure, which are influenced by a variety of factors.

1) Interface/inner surface energy reduction

The interface can be grain boundary or surface. It is the fundamental driving force for sintering.

Reduction occurs by the bonding of particles. Smaller particles have higher surface energy, which provides a strong driving force. As the particles coalesce, the total surface area decreases, resulting in a more stable, lower energy, minimum energy state [22].

It is a main driving force behind the initial stages of sintering, where particles begin to form necks and bonds at contact points. Mass transport is enhanced by diffusion and plasticity (**see figure 15**) .

$$\Delta(\gamma A) = (\Delta\gamma)A + \gamma(\Delta A) \quad (I)$$

being γ interface energy density and A interface area

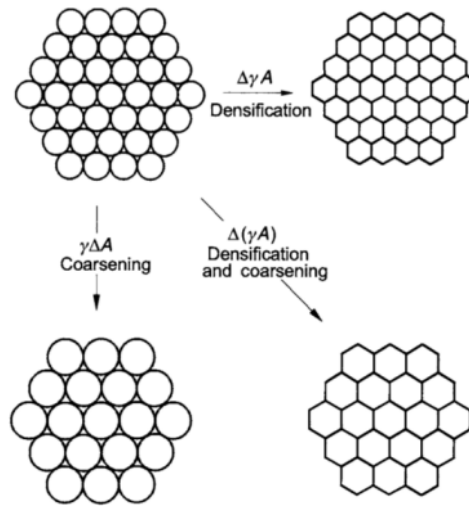


Figure 15. Densification and enlargement of grains

In the final, fully dense body, the particle surfaces are, for all practical purposes, removed. Throughout this process, a grain boundary area is created, and since it is smaller than that removed (and so is its energy), the net energy of the sintered body decreases, generating the driving force for sintering (see equation 1).

2) Diffusion mechanisms

Sintering processes are mainly regulated by solid-state diffusion mechanisms that are activated by heat. Diffusion occurs at the molecular level due to random motion and concentration gradients (see figure 16). It is a type of mass transfer.

- a) Surface diffusion: atoms move along the surface of particles, which is crucial in the early stages of neck formation between particles.
- b) Diffusion along grain boundaries: atoms move across the boundaries between particles, promoting compaction and bonding of the material.
- c) Bulk diffusion (massive): atoms move through the internal structure of the material, participating in the overall densification.

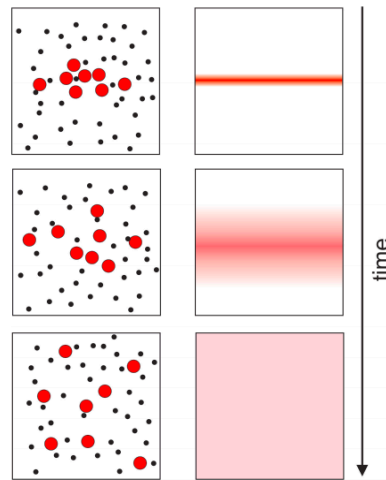


Figure 16. Diffusion in the sintering process

It can be described by Fick's laws, as they are fundamental to understanding diffusion.

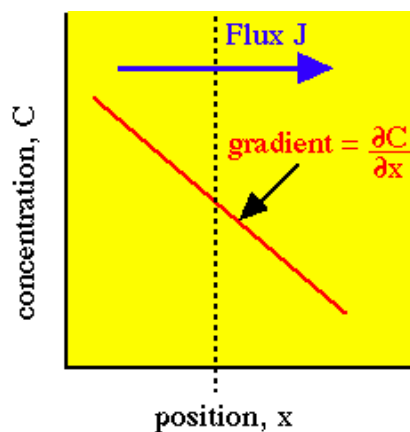


Figure 17. Fick's First Law

- Fick's first law: (see **figure 17**) as stated in [17], the diffusive flux is proportional to the concentration gradient, negative to indicate that it occurs in the direction of decreasing concentration (see **equation 2**). The problem with this law is that for it to apply well, the conditions throughout the system are constant. The flux input has to be the same as the flux output.

$$J [1/s \cdot m^2] = - D \frac{dC}{dt} \quad (2)$$

being J diffusive flux, D diffusivity and $\frac{dC}{dt}$ concentration gradient

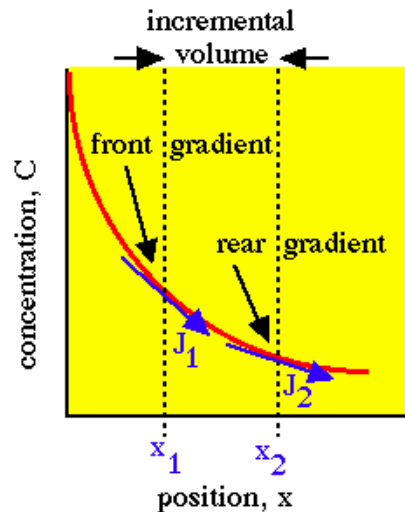


Figure 18. Fick's Second Law

- Fick's Second Law: (see **figure 18**) describes the rate of accumulation or decrease of concentration within the volume as proportional to the local curvature of the concentration gradient [15], i.e. the concentration changes over time. Requires initial and boundary conditions (see **equation 3**).

$$\frac{dC}{dt} [1/s \cdot cm^3] = D \left(\frac{d^2C}{dx^2} \right) \quad (3)$$

being x the position

The factors that influence diffusion are:

- Temperature: the higher the temperature, the higher the diffusion rate, which accelerates the sintering process.
- Concentration gradient: the higher the gradient, the higher the diffusion rate (more molecules moving down the gradient).
- Particle size: smaller, spherical particles have higher diffusivities because they have lower interactions with the medium and other molecules [16].

3) Applied external pressures (Laplace pressure)

The Laplace pressure (see **figure 19**) value is defined as the product of the surface tension by interfacial area curvature. It is the excessive pressure of the phase created by the closed surface (see **equation 4**).

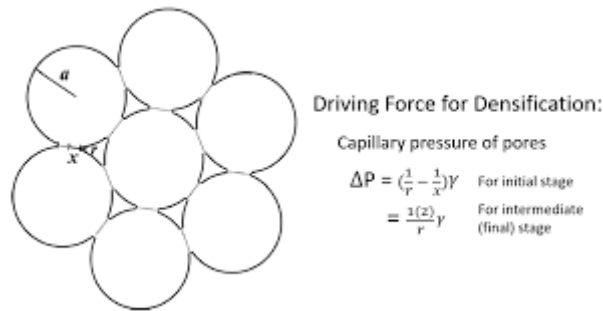


Figure 19. Laplace pressure

$$\Delta P = \gamma \cdot \left(\frac{1}{r} - \frac{1}{x}\right) \quad (4)$$

being γ a constant, x and r curvature at the given point

It improves the sintering process, especially in techniques such as hot pressing or plasma sintering. Pressure helps the rearrangement of particles and the removal of porosity, resulting in increased densification.

The application of pressure can also reduce the sintering temperature required, making the process more energy efficient [22].

Thinly divided materials should experience a decrease in their cellular parameters as the grain size decreases [13].

I.3.4 Techniques

Applying different techniques to ceramic materials with the same composition allows differences in the microstructure and performance of the products obtained.

Conventional Sintering (CS)

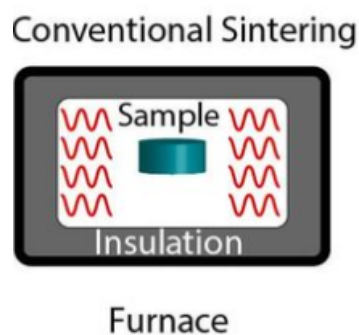


Figure 20. Conventional Sintering (CS)

It is the simplest technique and consists of heating a previously prepared powder compact to ambient temperature, without applying external pressure.

Pressureless Sintering (PS)

In this process, no external driving force is required, as it is provided by the surface free energy of the ceramic powder. This leads to a reduction in the total surface area of the powder and a decrease in interfacial energy. It is a simpler and more common process.

The resulting products generally have lower performance compared to those obtained through hot pressing, hot isostatic pressing, or atmospheric sintering. However, its advantages include not requiring special equipment, being cost-effective, and allowing the preparation of products with complex shapes and batch production [25], while avoiding density variations.

Gas Pressure Sintering

This process involves the sintering of ceramic products at high temperature and pressure using pressurized nitrogen or other inert atmospheres. It helps prevent decomposition. Applying pressure from all sides to the ceramic material results in a more solid, compact, and simultaneously strong and durable structure.

The advantages include improved density and mechanical properties, as well as a finer and more uniform grain microstructure.

Hot-Pressing Sintering (HP)

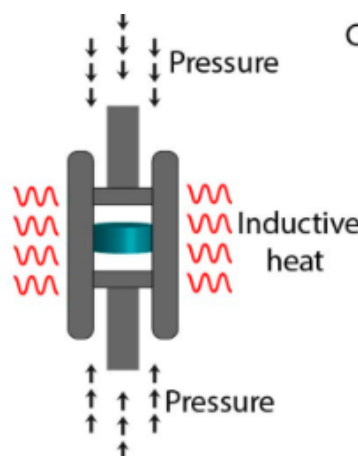


Figure 21. Hot-Pressing Sintering (HP)

This process involves mechanical pressure, where ceramic powder is placed inside a mold and then heated to a selected temperature under a certain pressure. The external pressure

complements the driving force, allowing densification to occur in a relatively short time, resulting in a fine and uniform grain microstructure [25].

The advantages include improved mechanical properties, reduced sintering time or temperature, and enhanced mechanical performance at high temperatures.

Hot Isostatic Pressing Sintering (HIP)

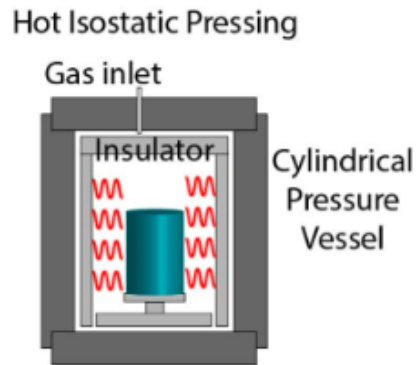


Figure 22. Hot Isostatic Pressing Sintering (HIP)

This is a technique that uses inert gases, typically nitrogen or argon, as pressure-transmitting media. The process involves placing the product in a sealed container and subjecting it to uniform pressure from all directions at temperatures between 900–2000 °C and pressures ranging from 100–200 MPa (simultaneous pressure-assisted sintering).

Among its advantages are the production of high-density products, as it effectively reduces porosity [26], along with excellent uniformity and outstanding performance. It also offers short production times, fewer processing steps, low energy consumption, and minimal material loss.

Spark Plasma Sintering (SPS)

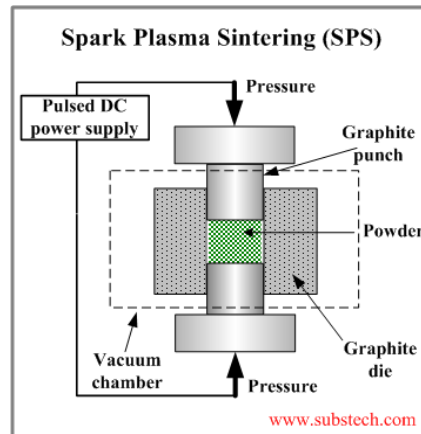


Figure 23. Spark Plasma Sintering (SPS)

This is a technique that uses pulsed electric current for pressure-assisted sintering, which causes self-heating of the material being processed (**see figure 23**). The energy from plasma discharges between particles leads to rapid and dense sintering. The process involves Joule heating, as in conventional hot pressing, and plastic deformation caused by the applied pressure.

Joule heating states that the heat generated per second is equal to the electrical power absorbed, or the power dissipated [12] (**see equation 5**).

$$P = I^2 \cdot R \quad (5)$$

Electricity also passes through the powder, generating heat precisely where the particles touch, which causes them to heat up very rapidly from the inside. This technique offers distinct advantages over the others, such as rapid heating and low sintering temperature. It results in ultrafine grains with no significant anisotropy.

Microwave Sintering (MS)

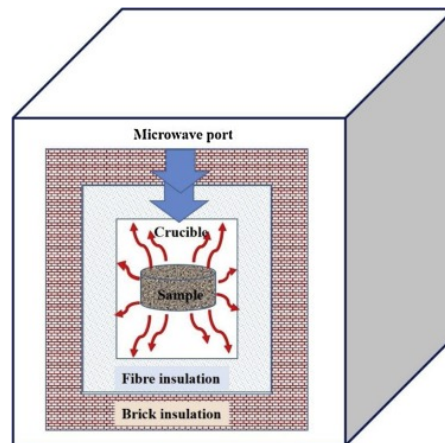


Figure 24. Microwave Sintering (MS)

This technique is based on utilizing the dielectric loss of ceramic materials in electromagnetic microwave fields to synthesize and densify them. During the process (**see figure 24**), the materials absorb microwave energy and convert it into molecular kinetic and potential energy within the material [25].

As a result, uniform heating is achieved, with minimal internal temperature gradients and high heating and sintering rates. Ultimately, this enables rapid sintering at low temperatures, improving the mechanical properties of ceramic materials. Since it does not require an external heat source, it is highly efficient and energy-saving. It offers high production efficiency and a low unit cost [25].

Two-steps Sintering (TSS)

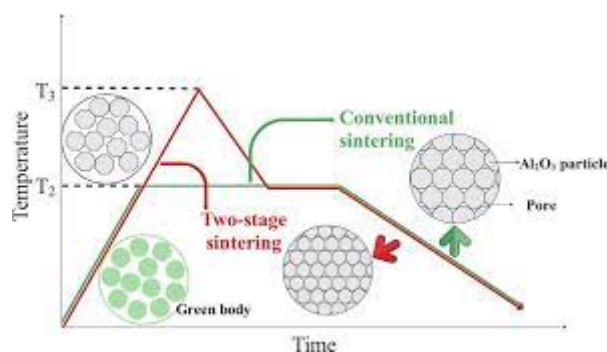


Figure 25. Two-Step Sintering (TSS)

This method is one of the simplest and most cost-effective for obtaining materials with near-theoretical density and controlled grain growth without the addition of dopants [70]. It is an efficient technique for producing ceramics with a fine-grained microstructure, high density, and relatively low cost. The process involves heating the sample in two stages.

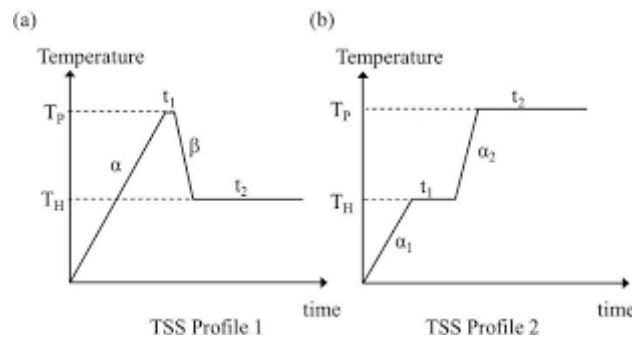


Figure 26. Two-Step Sintering (TSS) with (a) high temperature in the first step (b) low temperature in the first step

- 1) With a high sintering temperature in the first step.

It is widely used to produce fully dense ceramics with controlled grain growth due to the higher initial sintering temperature. Certain conditions must be met at the end of the first sintering step. Specifically, the sample must reach a critical density to ensure that the densification process can continue during the final sintering stage. This critical density varies depending on the material.

- 2) With low sintering temperature in the first step.

In this case, the samples are initially heated to lower temperatures and then to higher temperatures. The first step involves a pre-thickening process to remove the volatile fraction and to smooth the porous channels.

I.4. Electric Field

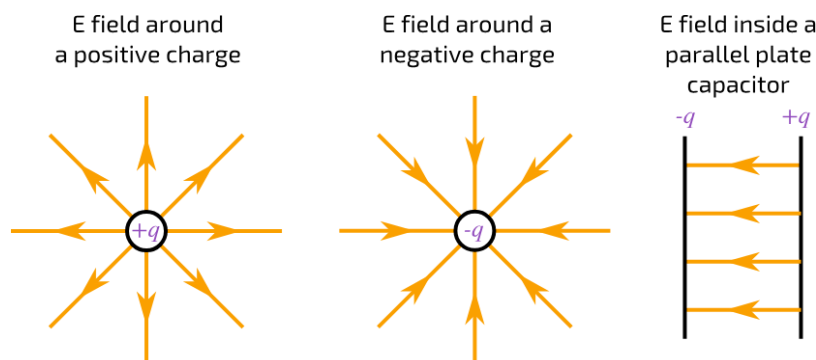


Figure 27. Fundamentals of the electric field

Flash sintering is an innovative technology for the electric field-assisted sintering of ceramic materials that is gaining increasing importance today, as it allows for the production of highly dense powders in short periods of time [7]. It is a non-conventional method of powder densification involving the application of an electric discharge with resistive heating and pressure. As a result, chemically clean grain boundaries are generated, along with new resistivity peaks in superconductors.

An electric field exists (**see figure 27**) in any region where a charged particle is subject to a force that depends solely on the particle's charge and position. It is defined as the force per unit charge that would act on a small, positively charged particle placed at that point [4] (**see equation 6**).

$$E [V/m] = \frac{F}{q} \quad (6)$$

being q the size of a charge and F the force that q is subject at some point

Some studies have found that the sintering temperature decreases with the intensity of the electric field, even reaching room temperature. However, increasing the electric field can lead to other effects on the experiments.

When an electric field is applied to a ceramic material at a specific temperature, a sudden increase in the electric current within the sample occurs. Due to the thermal effect of the current, the ceramic can achieve full densification in a very short time [5]. Electric fields enhance shrinkage in both axial and radial directions without causing appreciable changes in the internal structure that would indicate a preferred direction.

The sintering stress increases almost linearly with the applied electric field. In contrast, the uniaxial, volumetric, and shear viscosities decrease [1].

Since the energy required for this process is much lower than in traditional methods, the energy consumption is low. Furthermore, due to the extremely short sintering time and the low temperature required, grain growth can be controlled through instantaneous sintering. Additionally, the necessary hardware is minimal, consisting only of a power source, cables, and a sintering furnace.

In addition to the heating and increase in conductivity with temperature, an increase in atomic diffusion and mobility also occurs, caused by the formation of defects (Frenkel pairs) induced by the electric field. However, the electric field alone is not sufficient to form Frenkel pairs unless it reaches values of 10^9 V/m, which is unrealistic. As an alternative, the formation of Frenkel pairs mediated by nonlinear phonons with intense local strain fields has been proposed, as mentioned in the study in [2].

I.5. Graphite sheets

Graphite is a natural form of carbon that features a two-dimensional, planar molecular structure. It appears in the form of flakes, meaning multiple layers of graphene held together by weak bonds. As the most stable form of carbon under standard conditions, it is used as the reference state for defining the heat of formation of carbon compounds. This non-metal is a good conductor of heat and electricity and possesses high strength and stiffness. Additionally, it remains resistant and stable at temperatures above 3600 °C.

The sheets used allow for a reducing environment to prevent AlON from oxidizing and losing its properties. As previously mentioned, graphite withstands high temperatures without degrading, making it a good support material. It can also help distribute heat evenly throughout the pellet.

An increase in the particle size of the carbon can lead to a corresponding increase in the particle size of the synthesized AlON powders [19].

Using graphite sheets during the sintering process results in carbon contamination in the final samples, which is a problem when aiming for transparency. Undesired phases may form due to reactions between the graphite and the sample. Although graphite helps prevent oxidation, it can interfere with specially controlled atmospheres (such as nitrogen or vacuum) if not properly managed.

Efforts are being made to use alternative materials to reduce the contamination caused by graphite. For this reason, tantalum has been proposed as a substitute.

I.6. Conclusion

The initial chapter provides a detailed overview of aluminum oxynitride (AlON), highlighting its remarkable mechanical, optical, and thermal properties. These characteristics give AlON high potential for use in advanced technological applications, particularly in the defense and precision optics sectors. Furthermore, a critical review of sintering methodologies was conducted, with special emphasis on those assisted by electric fields. This analysis underscores the importance of precise control over processing parameters to achieve efficient densification and optimize the functional properties of the ceramic material.

CHAPTER II:

Experimental analysis

II.1. Introduction

The following is a detailed explanation of the procedure carried out to obtain the sample pellets and their sintering process in the furnace, as well as their characterization. The equipment used is also described, and a flow diagram of the HP furnace system will be presented.

II.2. Raw material

For the preparation of the pellets, raw materials available on the commercial market can be used, or a custom mixture can be prepared in the laboratory. In our case, commercially available raw materials were used to create our own mixture. The AlN and Al₂O₃ (alumina) powder containers were sourced from the company Alteo, specialists in the production of specialty aluminas.

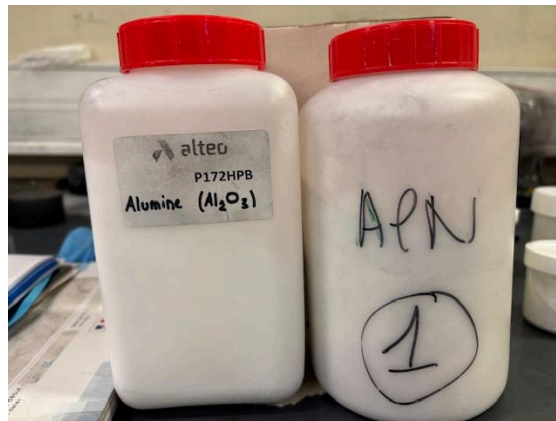


Figure 28. AlN and Al₂O₃ powders

The Al₂O₃ used has an exceptionally high chemical purity, with an approximate concentration of 99.9% Al₂O₃. The average particle size is initially very small (on the order of a few micrometers). Due to its highly cohesive nature, the alumina powder tends to agglomerate, forming significantly larger clusters.

In the case of AlN, the undoped version is used to optimize sintering without adding dopants, in order to avoid the formation of unwanted secondary phases.

II.3. Preparation of the samples

II.3.1 Preparation of the powders

As can be seen in the **figure [7]** , AlON is obtained between [27- 40] % molar percentage of the AlN component. However, it is necessary to know the value in mass percentage. For this purpose, the following **equation (7)** is used, which can be found in more detail in the **annex 1**:

$$W_1 = \frac{x_1 \cdot M_1}{x_1 \cdot (M_1 - M_2) + M_2} \quad (7)$$

The values in mass percentages 12.94% and 21.13% are obtained. To work the mixture, an intermediate value is taken, in this case it will be 17% AlN, being 83% Al₂O₃.

A total of 50 g of mixture has been prepared, so 41.5 g of Al₂O₃ and 8.5 g of AlN are required. It is weighed on a digital balance (**see figure 29**) and reserved in a container for the next step.



Figure 29. Electronic balance

The mixture is then introduced into a homogenising machine (**see figure 30**) , and if necessary, some aluminium balls should be added to grind the mixture to obtain finer grains. In addition, it must be taken into account that the machine has a limit value of mass that can be introduced in order for it to work properly. The range in which it must be kept is $110 < \text{mass in g} < 140 \text{ g}$. In our case, the container used weighed 86 g, leaving room for 50 g of mixture, which occupies approximately $\frac{1}{3}$ of the vessel. It was left at 1040 rpm for 1 min, twice.



Figure 30. Electronic mixer

II.3.2 Dust compaction

After mixing, the pellets are compacted. If aluminium pellets are used, sieving should be carried out inside the fume extractor hood to separate the powder from the pellets, repeating the process several times until the material is recuperated.

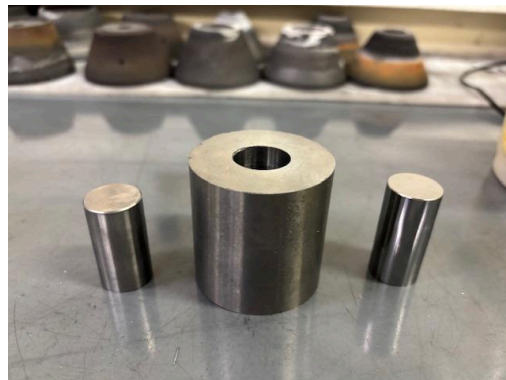


Figure 31. Aluminum matrices

To form the pellets, 2.8 g of mixture is required to obtain a thickness of 5 mm and a diameter of 20 mm and is made by uniaxial pressing.

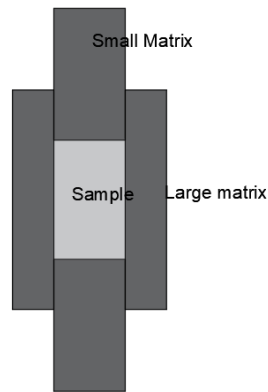


Figure 32. *Assembling the matrices with the mixture*

The process involves placing the weighted mixture into an aluminum matrix (**see figure 31**) with a 20 mm diameter cavity, in which a solid aluminum matrix has previously been inserted. Another identical aluminum punch is then placed on top (**see figure 32**). At this point, pressure can be manually applied using the 20 TON press (**see figure 33**). In our case, a force of approximately 1.6 meT (metric tons) is selected. The pellet is held under pressure for 5 minutes.

The selected force is converted into pressure as follows (**see equation 8**):

$$\sigma = \frac{F}{S} = \frac{16 \cdot 1000}{\pi \cdot 0,02^2} = 127,23 \text{ MPa} \quad (8)$$

The pressure applied during the compaction of the pellets must be higher than the pressure exerted during the sintering process in the furnace. If it were lower, the pellet could be damaged or destroyed during the process.

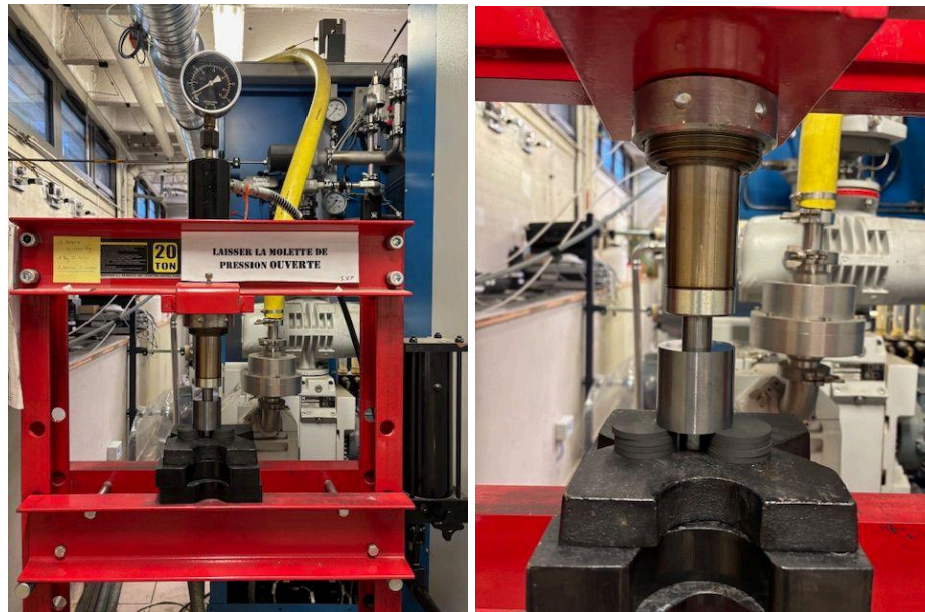


Figure 33. Manual press of 20 TON

To remove the pellet from the mold, one of the two punches inserted into the hollow matrix is taken out, and the main matrix is turned upside down. A container with cotton is placed beneath the matrix to cushion the pellet when it falls. Once everything is set up, pressure is carefully applied using the machine until the pellet drops through the bottom.

II.4. System flow diagram

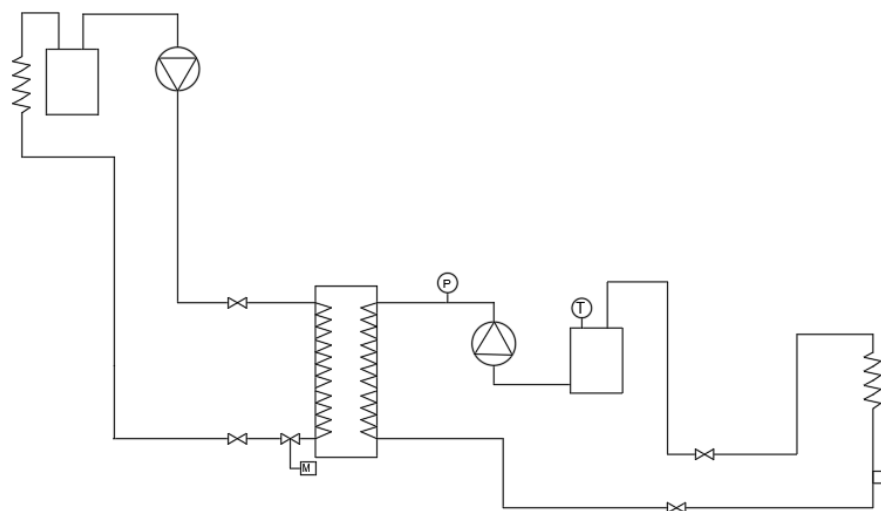


Figure 34. HP Oven Cooling System Flow Diagram

In **Figure [34]**, a flow diagram of the components that make up the cooling system of the HP furnace is shown. It consists of a plate heat exchanger supplied with water from a reservoir, which must always maintain an adequate water level (60 L), and another reservoir that provides an additional cooling liquid. The heat exchanger is also connected to the HP

furnace, into which it injects water during operation. The system also includes valves, pumps, and pressure and temperature sensors.

II.5. Assembly of the sintering oven

To carry out the sintering of our pellets, the Hot-Pressing furnace (see figure 35) in the laboratory was used.



Figure 35. HP oven

The Hot-Pressing technique involves applying heat and pressure to a material, ceramic in this case, under a protective atmosphere to produce a dense and uniform final product. The material is placed in a mold, and heat and pressure are applied simultaneously, causing the material to soften and flow, filling the mold and forming the desired product.



Figure 36. Assembling the interior of the HP oven

The sintering process of the samples was carried out using the HP Goliath furnace from the company Stein Heurtey Physitherm **Figure [35]**, which operates under pressures close to atmospheric. This equipment is a hot-pressing system equipped with a chamber measuring $\text{Ø}140 \times 190 \text{ mm}$, capable of applying uniaxial pressure up to 100 kN through a top piston (**see figure 36**). Thermal treatment can be performed under various controlled atmospheres, such as vacuum, argon, or nitrogen. Heating is achieved via a resistive element, allowing temperatures of up to 2100°C , with a heating rate of $25^\circ\text{C}/\text{min}$. The samples are placed in a cylindrical graphite mold with a maximum diameter of $\text{Ø}80 \text{ mm}$.



Figure 37. (From Left to right) Water, gas and compressed air streams for the HP furnace

For proper operation, the furnace requires several utility supplies (**see figure 37**) :

- Water flow for cooling
- Gas flow

- Compressed air flow

As previously mentioned, the pressure in the furnace must be lower than the compaction pressure of the pellet. The maximum uniaxial pressure in the HP furnace is 327 daN, which corresponds to approximately 0.33 meT, as shown in **Annex 2**.

$$\sigma = \frac{F}{S} = \frac{3,3 \cdot 1000}{\pi \cdot 0,02^2} = 26,26 \text{ MPa (9)}$$

During our tests, nitrogen was used as the atmosphere, since previous studies have shown that using nitrogen or argon results in negligible differences in the final density of AlON [24].

First, the compressed air, gas, and water cooling systems are connected, and the HP furnace is started. The pellet is carefully placed into its mold and inserted into the furnace die. Once the pellet is inside, the indicated pressure (according to **equation [9]**) is applied using the resistive element, and the furnace door is properly closed.

Next, the vacuum pump (VP) and the primary pumping valve are activated, allowing the furnace to reach a vacuum level of 2.1 mbar in order to eliminate any residual oxygen inside. Once this vacuum is reached, pressure is reapplied, since it was reduced during the vacuum phase, and the water cooling system is turned on. The partial pressure should be approximately 900 mbar. At this point, the sintering process can begin.

The temperature was increased at a rate of 12 °C per minute, with a holding time of six and a half hours at various temperatures. A variable pressure was also applied depending on the specific test. All experimental parameters were recorded by the furnace's control software.

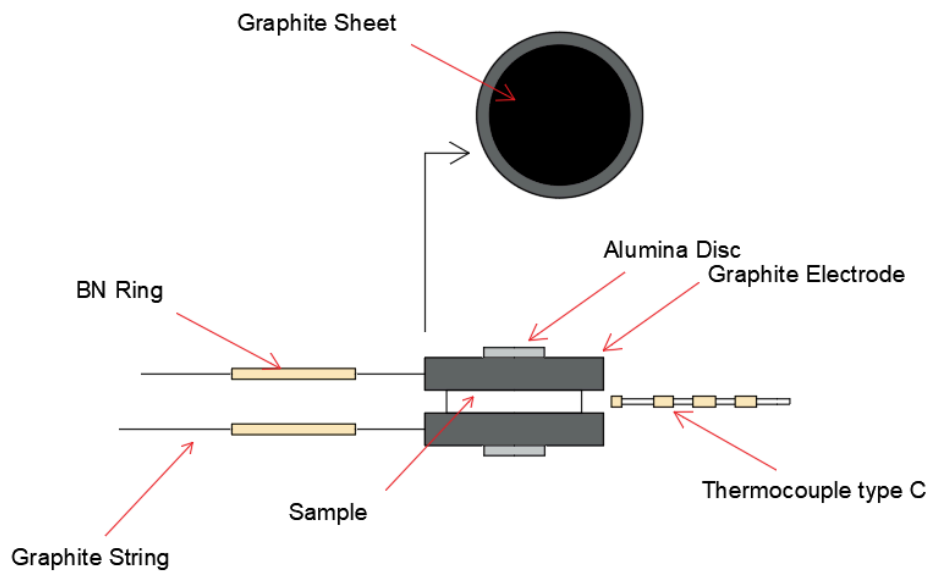


Figure 38. Internal assembly of sintering by applying electric current

For the case of sintering with an electric current, the pellet is placed between two electrodes connected to the furnace via graphite cords, surrounded by boron nitride tubes, allowing the electric current to pass through the sample. For better temperature distribution, graphite sheets are used between the pellet and each electrode, one on each side(see figure 38).

The electric current is applied directly to the sample through the upper electrode using a power supply. On top of the electrodes, two alumina insulating discs, 20 mm in diameter, are placed to electrically isolate the setup from the rest of the furnace and thus direct the current only to the sample.

Additionally, a type C thermocouple made with two wires (one tungsten and the other tungsten with 26% rhenium) is used at all times. It is placed near the pellet to measure its temperature.

A program is used to control the intensity and voltage applied to the pellet inside the oven using the thermocouple, and its temperature.

II.6. Experimental conditions for sintering

For the tests, several parameters have been used, such as the pellet dimensions, sintering temperature and time, sintering pressure, the type of atmosphere to which the pellet is exposed, the type of current, which can be alternating or direct (including voltage and current intensity), and the type of sheets used between the graphite electrode and the pellet.

In our case, the parameters varied were the sintering temperature, sintering time, and the application of electric current (**see table 2**).

Table 2. Experimental conditions of the sintering process

Parameter	Value
Pastille diameter	20 mm
Pressure applied by the piston	127 DaN aprox
Heating speed	12 °C/min
Sintering time	Variable (hr)
Intensity	1 A
Voltage	Variable (V)

At the same temperature, two tests are carried out, one without current and one with current, in order to observe the effects of the electric field during the sintering process.

The voltage is gradually applied to the specimen in 5 min step increments. At the end of each step, the voltage may or may not be increased, depending on whether the set current value has been reached. If the pellet becomes conductive, it is no longer necessary to adjust the voltage.

II.7. Characterization of the raw material

II.7.1 Al₂O₃ (AluminaP172HPB)

The alumina powder P172HPB, supplied by Alteo, is used in the preparation of our mixture. This material is characterized by its very high chemical purity, with an approximate content of 99.9% Al₂O₃. The particles have a very small average size, on the order of a few micrometers. However, due to its cohesive nature, the powder tends to clump together forming large agglomerates.

ID ?chantillon COMMANDER (Coupled TwoTheta/Theta)

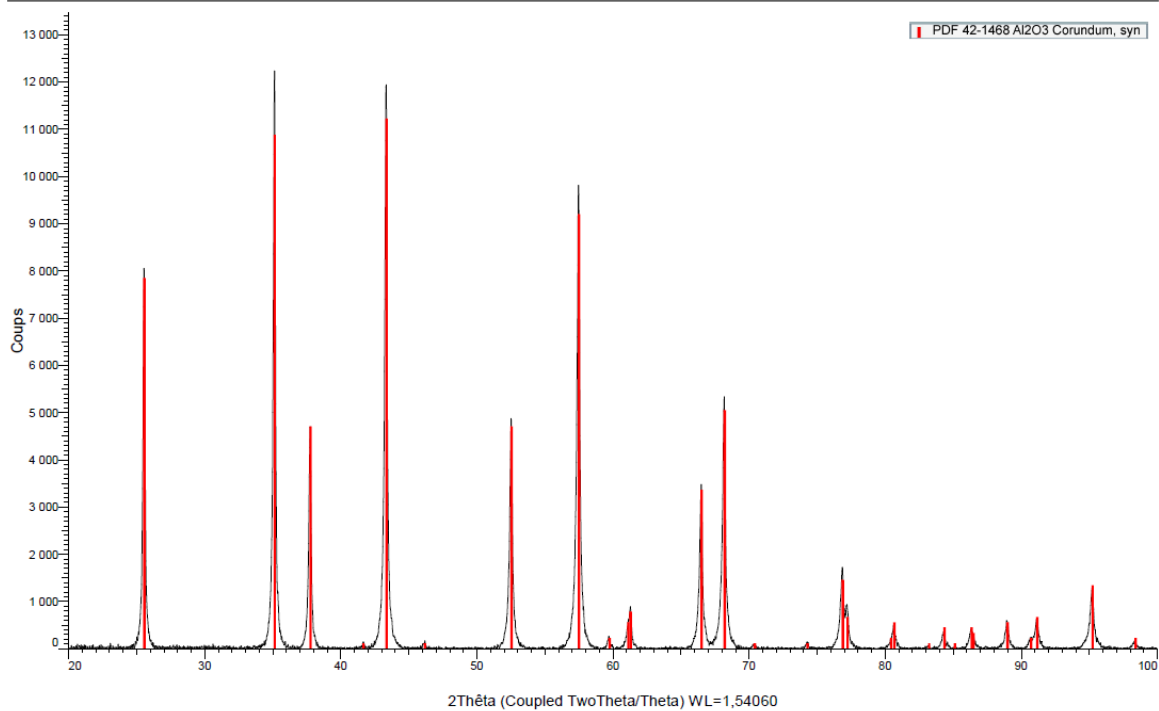


Figure 39. *Diffractogram of Al_2O_3 powder*

An DRX test (see **figure 39**) has been performed on the raw Al_2O_3 powders. It shows a structure corresponding to PDF card 42-1468, which is characteristic of a rhombohedral structure typical of α -alumina.

II.7.2 AlN

It was decided to use undoped powder with the aim of optimizing the sintering process without resorting to additives. Although dopants can help eliminate porosity, they can also cause undesirable effects, such as the formation of secondary phases and precipitates that scatter light, resulting in an opaque sintered material.

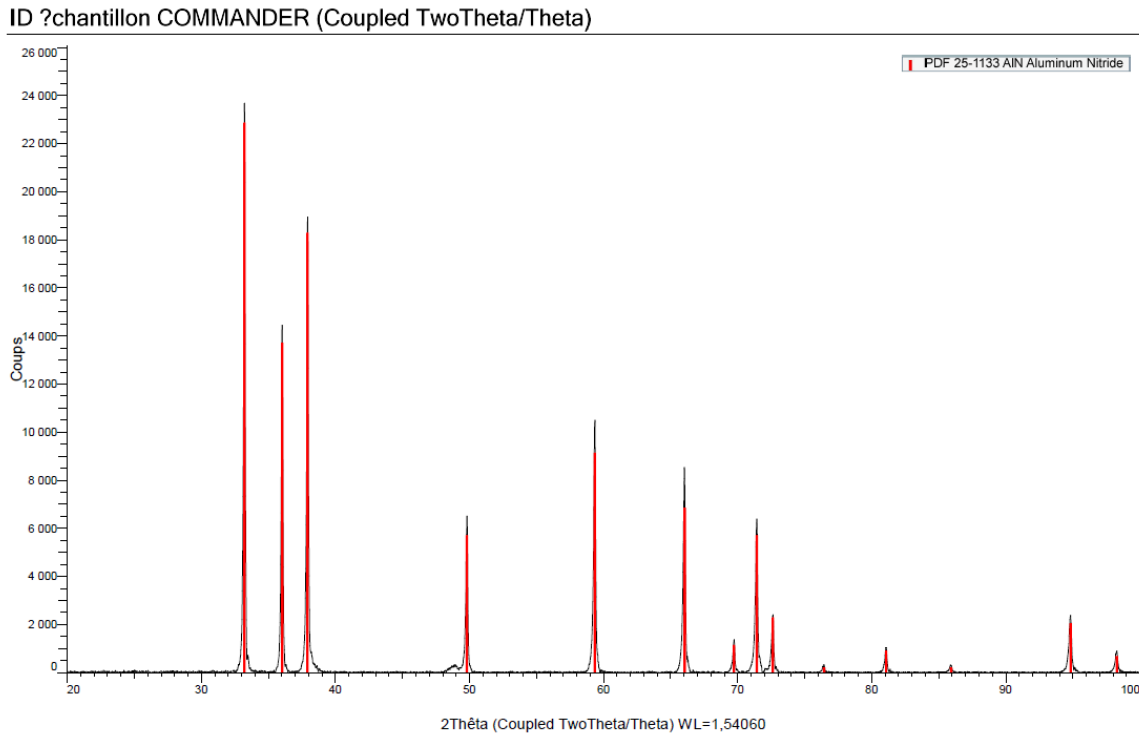


Figure 40. Diffractogram of AlN powder

In the case of AlN, the DRX (see **figure 40**) shows that the material matches PDF card 25-1133.

Additionally, SEM analysis has been performed on the raw initial powder mixture. As can be seen in the **figure 41**, the larger particle agglomerates correspond to alumina, and the smaller particle agglomerates correspond to AlN.

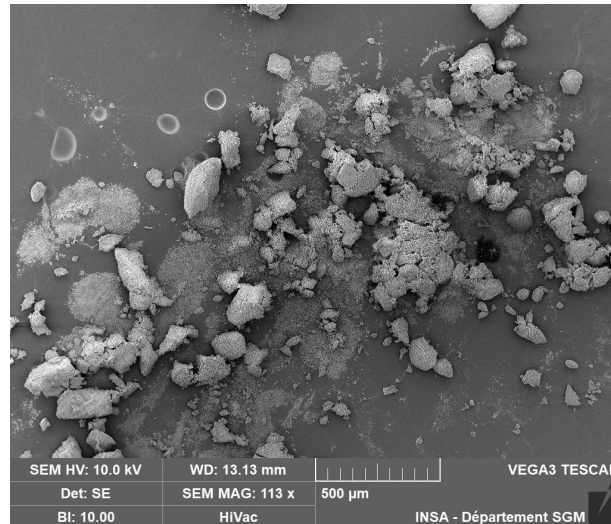


Figure 41. SEM image of the raw Al_2O_3 powder mixture

II.8. Characterization of the samples

II.8.1 Sample Preparation

Cutting

The technique consists of separating or shaping pieces using specific tools or techniques, since ceramic materials are hard, brittle, and not very ductile, making them difficult to cut with common tools as they may break. This step is important for subsequent analysis using SEM (Scanning Electron Microscopy), which provides information about the microstructure. It allows preserving the physicochemical properties without damaging the sample.



Figure 42. ISOMET 4000 compact chainsaw

A compact chainsaw model ISOMET 4000 from the BUEHLER brand is used (see figure 42).

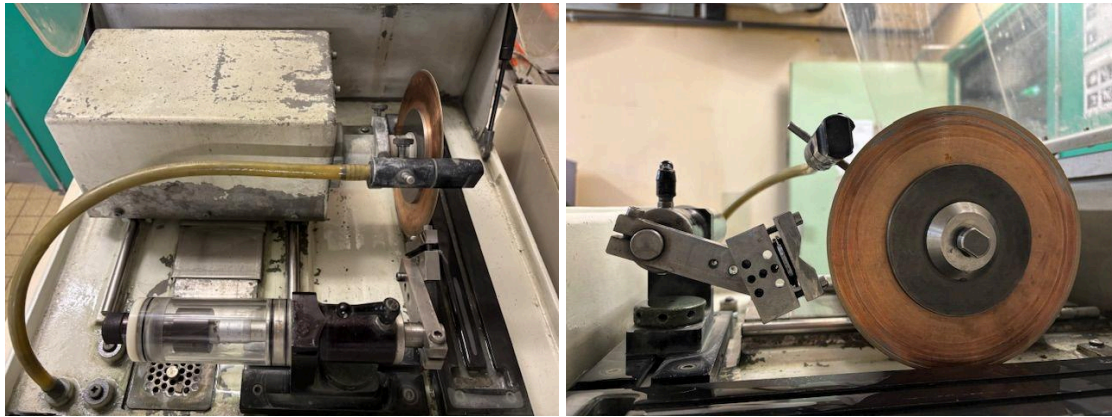


Figure 43. Interior assembly of the ISOMET 4000 compact chainsaw

It consists of a large diamond disc and two smaller aluminum discs. Additionally, it features a lubrication system (see figure 43).

The sample is marked at its midpoint and must be positioned between the precision vise and some plastic sheets, which help provide better grip of the sample during cutting, ensuring that half of the sample protrudes, leaving an extra 2 mm. Once it is confirmed that the sample is securely fixed in place, the precision vise is attached to the protruding metal arm using a nut and is positioned at a 45° angle relative to the cutting disc to shorten the cutting time. Then, the lubrication tank is checked and, if needed, filled with a bit of lubricant fluid and the rest with tap water. Next, the machine is checked to ensure everything is functioning properly, and the parameters are set. In our case, the settings were 3500 rpm for the disc's rotation speed and a feed rate of 4 mm/min, which is suitable for a material like ALON. The disc is then brought close to the sample, and the procedure can begin.

Hot-Coating

After the sintering of the samples, the first step is hot mounting, a process in which the samples are embedded in a resin block to facilitate their polishing.



Figure 44. MECAPRESS I hot coating machine

The MECAPRESS I hot coater (see figure 44) and a KonductoMet conductive resin from the BUEHLER brand are used.



Figure 45. Samples coated in resin

The process involves placing the sample with its holder inside the mounting press. Then, the resin is carefully poured in and the press is closed. The machine is turned on, and the process continues until a solid block is formed (see Figure 45). Afterwards, any excess material is lightly ground down to level the surface, allowing for proper polishing.

Polishing

The polishing step is essential to perform microstructural characterization tests, as they require a smooth and defect-free surface. It also allows us to remove the residue of the sheets used during sintering.

The technique consists of eliminating the scratches produced during the grinding operation and obtaining a mirror-like surface on the sample.



Figure 46. Mecatech 334 semi-automatic polisher

The machine used was the Mecatech 334 polishing machine from PRESI(see **figure 46**). It consists of a motor that rotates a disk on which the polishing cloth is placed. To perform proper polishing, the sample must be moved in spirals opposite to the rotation direction of the cloth. Additionally, a water flow helps to clean both the sample and the disk while polishing is carried out.



Figure 47. Diamond suspensions for polishing

Silicon carbide (SiC) abrasive discs and felt discs are used, and the polishing is done with a diamond suspension(see **figure 47**). Typically, polishing starts with the disc having the largest grain size, and then discs with progressively smaller grain sizes are used (see **table 3**).

Table 3. Operating conditions for polishing the samples

Step	Disc grain size	Granulometry (μm)	Duration (min)	Head/platter speed (rpm)	Applied force (N)	Lubrication	
Pre-polished flat	120	125	1:30	60/200	15	Water	
	220	68	1:30	60/200			
Fine pre-polishing	500	30	1:30	50/120	5		Diamond suspension
	1200	15	4	60/100			
Diamond polishing	Felt	6	10	30/60		Diamond suspension	
		3	10	30/60			
		1	15	60/200			
Cleaning	-	-	5	-	-	Water	

The first stage is to remove large irregularities and traces of other materials that may have contaminated the sample. The second stage is to refine the surface and reduce roughness. The third stage is to obtain a smooth surface. Finally, the abrasive residues are removed.

It is worth mentioning that the sample sintered at 1000 °C with current was polished using a force of 3 N, with polishing times of 1 minute and 3 minutes during the fine pre-polishing stage, as the sample was too delicate and disappeared during polishing.

Measurement of Electric Current

One of the characteristics to be verified is the electrical conductivity of the product obtained after sintering. It is the ability of a material to allow the flow of electric current through it.

*Figure 48. MX 579 METRIX voltmeter*

For this purpose, the MX 579 METRIX voltmeter is used(see **figure 48**). It is a digital instrument designed for common electrical measurements (voltages, currents, decibels). It is powered by a 220 V or 127 V, 50 Hz mains supply, with internal communication and a power consumption of 6 VA. Its dimensions are 230 x 230 x 60 mm.

To measure, the Ohms option is used to obtain the electrical resistance of the material. The pellet is placed between both measurement cables (black and red), without touching the cable ends directly with the hand. If the value reads 0, it means the resistance is infinite and, therefore, the material is an electrical insulator. Conversely, if a value is obtained, the material is a conductor.

II.8.2 Microstructural Characterization

Spectrophotometry

Spectrophotometry is a qualitative and quantitative technique used to measure the amount of light absorbed by a chemical substance. It does this by measuring the intensity of light as a light beam passes through the sample solution, based on the Beer-Lambert Law.

1) Fundamentals

As previously mentioned, spectrophotometry is based on a series of laws. First, there is Lambert's Law, which states that the intensity of transmitted light decreases exponentially as the thickness of the absorbing medium increases arithmetically. On the other hand, there is Beer's Law, which states that the intensity of a monochromatic light beam decreases exponentially as the concentration of the absorbing substance increases arithmetically, when the beam passes through a homogeneous medium. These two laws combine to form the Beer-Lambert Law [3] (see **equation 10**):

$$A = \varepsilon \cdot c \cdot l \text{ (10)}$$

being: ε =molar extinction coefficient

l = path length of the cell

A = absorbance

c = concentration of the solution

Transmittance gives us a physical measure of the ratio between incident and transmitted light intensity as it passes through the sample. The relationship between %T and concentration is not linear but follows an inverse logarithmic relationship. When the incident and transmitted

intensities are equal, the transmittance is 100%, indicating that the sample does not absorb at that specific wavelength, and thus $A = \log 1 = 0$.

The amount of light absorbed depends on the distance the light travels through the chromophore solution and on the chromophore's concentration.

2) Operation

The spectrophotometer used is the V-670 model from the brand JASCO (see **figure 49**). It can cover a spectral range from [190–3200] nm.



Figure 49. V-670 Spectrophotometer

It is double-sided and consists of a single monochromator designed to obtain more energy, which includes a dual grating: 1200 lines/nm for the UV/Vis range and 300 lines/nm for the NIR range. It features two detectors: a PMT (photomultiplier tube) for the UV/Visible range and a Peltier-cooled PbS detector for the IR range. The advanced optical technique allows for a spectral bandwidth as narrow as 0,1 nm, while ensuring high operational precision (see **figure 50**).



Figure 50. Inside the V-670 spectrophotometer

Prior to measurement, the spectrophotometer must be calibrated without any sample inside using the AUTO ZERO function. The sample should be placed in the holder after the monochromator, ensuring that the light beam passes through it and that it remains stationary during the test. Once properly positioned, the lid is closed, and the transmittance measurement begins using our software, configured with a bandwidth of 5 nm, NIR bandwidth of 20 nm, and continuous scan mode.

3) Applications

- In chemistry and biochemistry, for the quantitative analysis of substances, where analyte concentrations are determined by measuring light absorption or transmission at specific wavelengths.
- In environmental testing, especially for water and air quality analysis, detecting and quantifying pollutants.
- In the petroleum and chemical industries, to assess fuel quality by measuring key parameters and identifying and quantifying impurities.

X-Ray Diffraction (XRD)

X-ray diffraction (XRD) is a non-destructive analytical technique with numerous applications in the analysis of physical properties such as phase composition, crystal structure, and orientation of powder, solid, and liquid samples.

1) Fundamentals

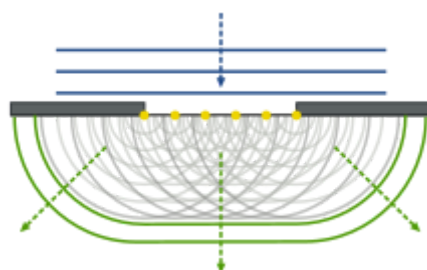


Figure 51. *Fresnel-Huygens principle*

Diffraction is a phenomenon that occurs when a wave encounters an obstacle or slit, which can act as a diffracting object. The information about the diffracting system is contained in the diffraction pattern. It is based on the Fresnel-Huygens principle (**see figure 51**), which states that every point on a propagating wavefront acts as a source of individual spherical wavelets.

The wavelength (λ) of X-rays falls within the ionizing radiation region of the spectrum, ranging between 10^{-8} and 10^{-11} meters, which is of the same order of magnitude as interplanar spacings. The electromagnetic radiation used must have a λ value comparable to the object being studied.

The types of solids that can be analyzed by XRD are crystalline solids and amorphous solids. We will focus on crystalline solids, which are characterized by periodicity, homogeneity, and anisotropy. Additionally, they exhibit long-range order. Crystals are structured by a lattice and a motif, forming the unit cell, which is the smallest repeating unit of a crystal and possesses all its symmetry properties.

Unit cells are defined by two vectors that form a parallelogram and have the same angle between them (**see figure 52**).

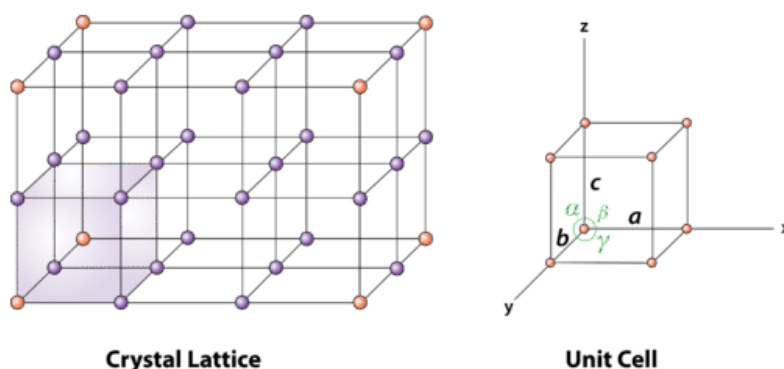


Figure 52. *Structure of a crystal and unit cell*

There are several types of unit cells, which arise from combining different geometries and the number of motifs per cell (primitive or centered). This gives rise to the 14 Bravais lattices, which, when combined with point symmetry and translational symmetry elements, result in 230 space groups.

A unit cell is defined by the parameters a , b , c (the lattice vectors) and α , β , γ (the angles formed between the vectors).

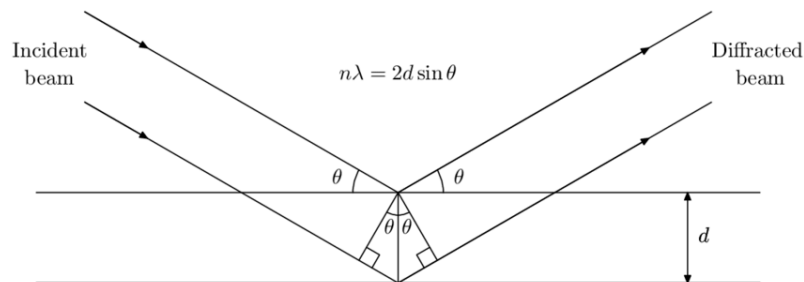


Figure 53. Representation of Bragg's Law

Bragg's Law considers the crystal planes as semi-transparent parallel surfaces for X-rays (see **figure 53**). It can be represented by the following equation (see **equation 11**):

$$2 \cdot d \cdot \sin\theta = n \cdot \lambda \quad (11)$$

being: d = distance between two crystallographic planes (*constant*)
 θ = angle of incidence of the X-rays (*variable*)
 n = order of diffraction
 λ = wavelength of the X-rays

Depending on the direction, the interferences can be constructive, which appear as peaks in the diffractogram, or non-constructive, which are not detected by the X-rays. The higher the value of n , the greater the intensity of the peaks in the diffractogram.

X-rays are generated when accelerated electrons strike a metal surface. This involves an electron displacing an electron from the anode, leaving a vacancy that is later filled by an electron from a higher energy level. Most electrons do not lose all their energy in a single collision, multiple collisions are typically required. As a result, a second type of spectrum appears, sharp peaks superimposed on the continuous radiation, known as the characteristic spectrum.

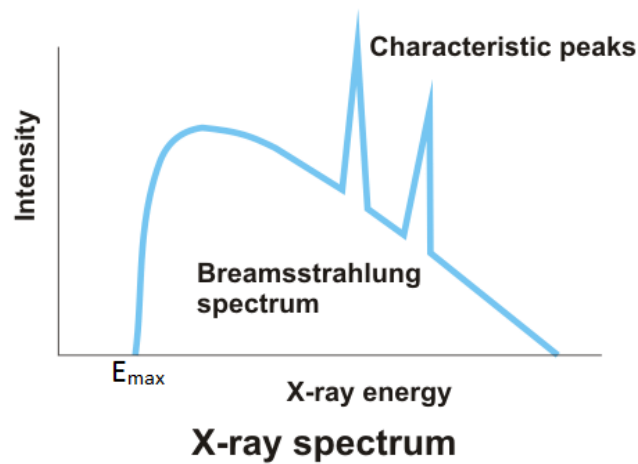


Figure 54. X-Ray spectrum

For a crystalline material, the spectrum should show only the characteristic peaks. In the case of an amorphous material, the spectrum would consist solely of continuous radiation. If the material is semi-crystalline, the spectrum would appear as shown in **figure 54**.

2) Operation

The X-ray diffractometer used was a Bruker D2 Phaser (**see figure 55**), operating at a voltage of 30 kV and a current of 10 mA, utilizing the Cu $K\alpha$ line ($1.54184 \text{ \AA}$).

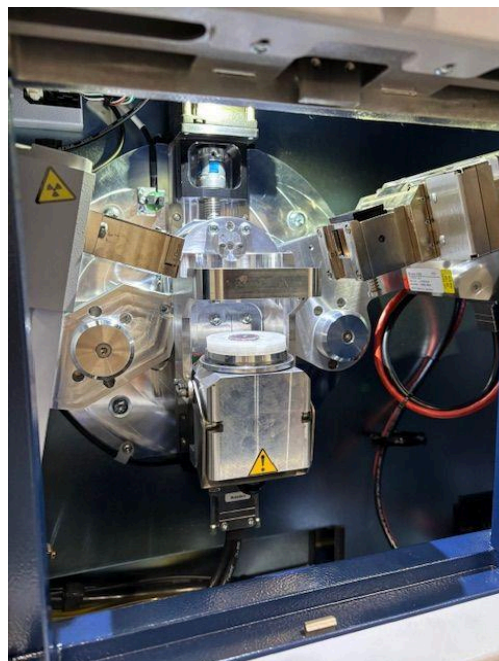


Figure 55. Bruker D2 Phaser X-ray diffractogram

The sample is placed on the sample holder using a small amount of modeling clay, as it must not move during the test. The sample should be well-polished to obtain more accurate and uniform data. Once positioned, the holder is inserted into the diffractometer, the door is closed, and the experiment begins. The data take approximately 30 minutes to be collected.

Parameters used during the experiment:

Initial $2\theta = 20^\circ$

Final $2\theta = 100^\circ$

Step Size = $0,0263^\circ$

Scan Mode: Fast continuous PSD with PSD aperture $5,826^\circ$

In addition to the previous diffractogram, it is also located in the building of Blaise Pascal. It is a Brüker AXS diffractogram (see figure 56).



Figure 56. Brüker AXS X-ray diffractogram

It consists of a Kristalloflex X-ray generator with a $0,4 \times 12$ mm (Cu) ceramic X-ray tube focal spot (see figure 57).

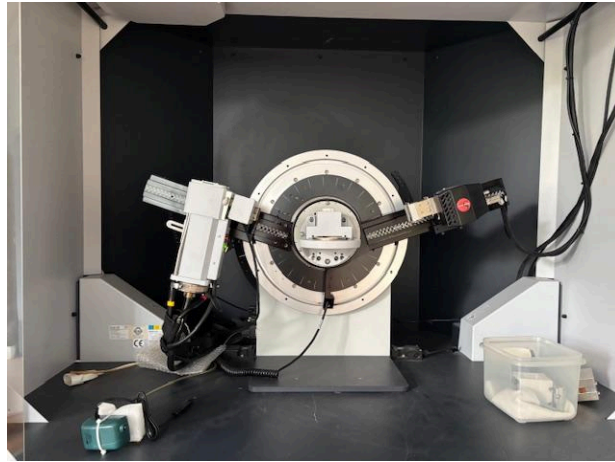


Figure 57. Interior of the Bruker AXS X-ray diffractogram

If the equipment has been switched off for several days, it is necessary to calibrate it using an aluminium sample (with a characteristic peak at $35,149^\circ$). Otherwise, enter the parameters required for the test (the same as those used in the previous diffractogram) and place the sample in the sample holder, securing it with a little plasticine to prevent movement.

From the diffractograms obtained, information such as phase identification, dosage, crystallinity measurement, evaluation of crystal size and trace residues can be extracted and structure can also be determined.

To read a diffractogram, one must know that:

- Position of the peaks: the crystalline phases present and the measurement of the lattice parameters (qualitative) are identified.
- Peak width: indicates the size and shape of the crystals, as well as traces of residues.
- Peak intensity: consists of the preferential orientation (quantitative).

3) Applications

Researchers use them as a tool to develop new materials or improve production efficiency.

Some of their main uses:

- Analysis of phase changes under other special conditions (non-environmental studies).
- Analysis of physical properties such as crystallite size (diameter), crystal orientation and residual stress (microstructure).

- They can be used on polycrystalline lamellar materials by a method called grazing incidence X-ray diffraction (GIXRD) [66].

Scanning Electron Microscopy (SEM)

A powerful technique designed to examine the surface of a material. It is used to obtain the morphology of nanostructural materials.

1) Fundamentals

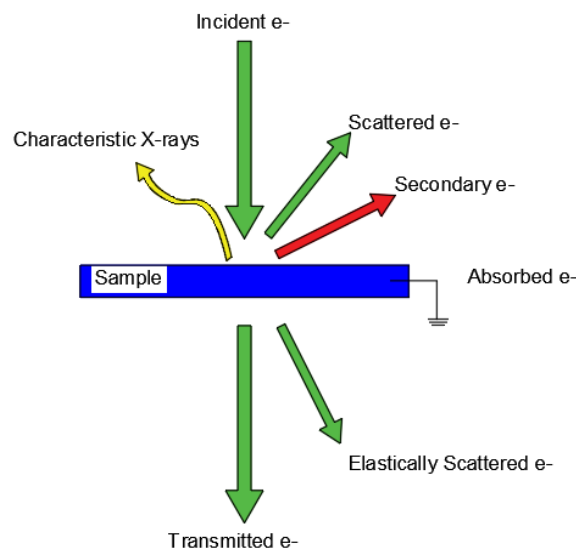


Figure 58. *Interactions of electrons with the sample*

Electrons can interact with the sample in two ways (see figure 58):

- Elastic interaction:
 - Scattered electrons: they have high energy and give us information about the atomic weight (PA) and topography of the sample.
- Inelastic interaction:
 - Secondary electrons: they have low energy and give us information about the topography
 - Characteristic X-rays: they give us information about the composition.

In addition, the transmitted electrons provide information on composition and microstructural information.

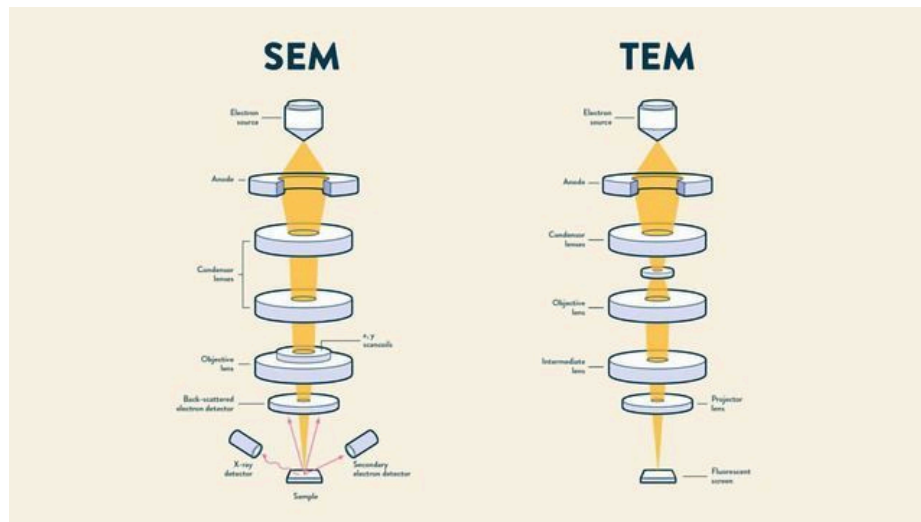


Figure 59. Types of microscopy SEM and TEM

Table 4. Characteristics of SEM and TEM microscopy

Characteristic	SEM	TEM
Electron type	Scattered	Of transmission
Image formation	Electrons captured and counted by the detector Display on the computer screen	Images directly on a screen using a charge-coupled device
Image information	3D surface images	2D internal structure image
Optimal spatial resolution	0,5 nm	< 50 pm
Maximum field of view	Big	Limited
Sample thickness	No restrictions	< 200 nm
Voltage	1 - 30 kV	60 - 400 kV
Sample preparation	Barely	Yes

2) Operation

It consists of focusing an electron beam with accelerating voltages on the sample. The interactions that take place between the electron beam and the sample produce signals, which will be picked up by the sensors. The signals will result in an image.

The volume of the sample in which the electrons interact is shaped like a bulb, with several zones in which different electrons are found.

When an electron beam is directed on a sample, some of these electrons collide with the nuclei of the atoms and bounce back without losing much energy. This phenomenon is called elastic scattering. Electrons that are deflected at an angle greater than 90 degrees are known as backscattered electrons, and the information they provide can reveal both the composition and the shape of the sample surface.

On the other hand, when the beam electrons interact with electrons and atoms in the sample, inelastic scattering can also occur. In this case, the incident electrons lose some of their energy by transferring it to the atoms in the material, causing them to be excited and releasing secondary electrons, which can also be analysed to obtain more information from the sample.

The apparatus is a TESCAN VEGA3 scanning electron microscope.

They usually consist of:

- A column maintained under vacuum.
- An electron gun comprising a source and a high voltage device.
- An electron lens assembly, an end-capacitor and a small diameter diaphragm to focus the beam onto the sample surface.
- A deflection device controlled by a scanning generator.
- A movable sample stage.
- An electron detector and signal amplification system.
- An image display system synchronously coupled to the same scanning generator.

Beforehand, the samples are polished, coated and cut in half in order to better see their microstructure during the analysis.

The samples are placed on a sample holder, on which a double-sided carbon conductor pad must be placed.

To carry out the SEM, the samples are subjected to a metallisation process, as they are not conductive, the electrons tend to be deposited on the surface of the samples, suffering a charging effect, which hinders the analysis of the images. This is done by sputtering, which involves forming a thin layer (10 nm), on the pellet, of a noble material such as Ag, Pd, Au, Pt, in our case gold. Argon gas is used for plasma formation and a gold wire is used (negative polarisation). This reduces electrostatic charging effects during imaging and improves the quality by increasing the contrast and conductivity of the sample.

After metallisation, the samples are taken to the microscope. They must be positioned and fixed correctly inside, making sure that they are sufficiently separated from each other and that they do not collide with anything. It is very important that the samples placed together have the same height.

To obtain the images, the ImageJ programme is used to observe the grain size and the degree of porosity in order to obtain more precise information about the density of the sintered pellets. The process involves looking for interesting areas in the samples and making the necessary adjustments and zooming and then measuring the areas corresponding to pores and grains. This is done by first focusing using the SE detector, adjusting the WD (the distance between the objective lens and the focusing plane), the astigmatism (image distortion) and the optical axis beam (centring). To acquire an image, use a scanning speed of 7.

This method carries some significant uncertainties, as the analysed region may not be representative of the whole sample. In addition, the manual selection of the areas, as it depends on the user's criteria, introduces a subjective component that can lead to variations between different operators.

The following conditions are used (**see table 5**) :

Table 5. Operating conditions for the SEM

Parameters	Value
Detector	SE
Scan Mode	RESOLUTION
HV	10 kV
WD	10 mm
Beam Intensity	10

3) Applications

- Microbial analysis: microorganisms can be visualised and studied in exceptional detail, providing information on morphology and microbial interactions.
- Materials characterisation: the properties, composition and microstructure of materials can be studied.
- Petrochemical: used to assess the quality and composition of water used in processing, cooling and wastewater treatment in petrochemical operations [68].

II.9. Conclusion

The second chapter provides a detailed description of the experimental methodology, covering the process from the shaping of the green compacts to their sintering in high-pressure (HP) furnaces. The proper selection of sintering conditions, materials, and equipment is critical to ensuring efficient densification of the material. The conducted experiments allow for a systematic analysis of the effect of the applied electric field during the reactive sintering of AlON, with the aim of optimizing its microstructure and enhancing the functional properties of the final material.

CHAPTER III:

Analysis of Results

III.1. Analysis and interpretation of results

After sintering the pellets and performing various tests to analyze the properties and behavior of AlON, a series of results were obtained (see table 6).

Table 6. Summary of the properties of the samples obtained

Temperature (°C)	Electric current	Sheet used between the electrodes	Conductivity	Phases observed after treatment (DRX)	Transparency	Density (g/cm ³)
1000	YES	Graphite	YES	Alumina, AlN	NO	3,6098
	NO	Graphite	NO	Alumina, AlN	NO	3,4313
1100	YES	Graphite				
	NO	Graphite	NO	Alumina, AlN	NO	
1200	YES	Graphite	NO	Alumina, AlN, a little AlON	NO	3,6637
	NO	Graphite	YES	Alumina, AlN	NO	3,6003
1300	YES	Graphite	NO	AlON, Alumina, AlN	YES	3,5940
	NO	Graphite	YES	Alumina, AlN	NO	3,1927
1400	YES	Graphite	NO	AlON, AlN	YES	3,6119
	YES	Tantalum	NO	AlON, AlN	YES	3,6435
	NO	Graphite	NO	AlON, AlN	NO	3,6099

P.S. Some essays were performed by another student in the same project.

Additionally, two other experiments were carried out in the laboratory of the Brillouin building at Lyon 1 University. A different furnace was used to synthesize the pellets. It is worth noting that the first experiment was completed successfully, although the resulting pellet contained a significant amount of diffused graphite inside, and part of the surrounding graphite furnace adhered to the pellet. The second experiment could not be completed because the ring protecting the sample became deformed. The process was stopped 35 minutes after it started. Although it was possible to reach 1400 °C, the temperature could not be maintained for a sufficient duration (see Table 7).

Table 7. Experimental data and summary of the properties of the new tests

Temperature (°C)	Diameter (mm)	Height (mm)	Mass (mg)	Compaction pressure (MPa)	Compression pressure (MPa)	Time (min)	Type of current used	Phases present after treatment (XRD)	Transparency
1400	3	3,5	55	200	600	60	Pulse	Alumina, AlN, a little AlON	NO
1400	7	3	258	370	300	35	Continuous	Alumina, AlN	NO

The information on the 1000°C current sample taken by the previous student is not available, as the pellet could not be found.

III.1.1 Interpretation of sintering curves

After sintering, the pellets, which were whitish in color before entering the furnace, come out with a more grayish tone. They have a more compact consistency than at the beginning, becoming less brittle.

For the pellets sintered with the application of an electric current, the progressive increase in voltage in stages was part of a controlled experimental approach aimed at studying the material's response to different voltage levels. However, no clear trend emerged.

Each pellet becomes conductive at different times and under varying voltages. The pellet sintered at 1000 °C reaches a current of 1 A approximately 1 hour and 15 minutes into the experiment, requiring a voltage of nearly 0,2 V, with the resistivity remaining constant at 0,011 Ohm·m. In the case of 1200 °C, the current reaches 1 A after 41 minutes, with a required voltage of nearly 50 V. Moreover, resistivity starts at 10 Ohm·m and then drops to 0 at the moment the current reaches 1 A. For 1300 °C, the current reaches 1 A after about 1

hour, with a required voltage of 18 V, and the resistivity at that point is 0,5 Ohm·m. Lastly, for 1400 °C (Graphite), a current of 1 A is reached after around 1 hour, requiring 0.7 V, and the resistivity is 0,041 Ohm·m. In contrast, under the same conditions but using Tantalum, 1 A is reached after 1 hour and 25 minutes at a voltage of 55 V. The resistivity shows similar values to the test with Graphite.

As shown in **figures [60], [61], [62], [63], [64]** the resistivity increases up to 1200 °C and then decreases in the 1300 °C and 1400 °C tests. Generally, at the moment the current reaches 1 A, the resistivity changes value. It seems that the material allows current to flow through it quite easily, except at the beginning of the 1200 °C test, where the resistivity value is somewhat higher.

In conclusion, the potential difference (voltage) required for the current to flow through each sample varies from one pellet to another, without following any apparent consistent pattern.

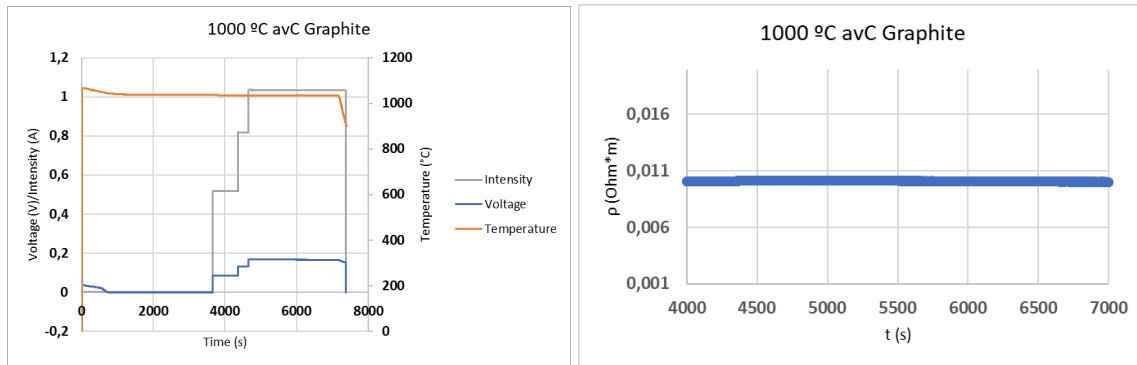


Figure 60. Representation of Tension/Intensity/Temperature vs Time and Resistivity vs Time for 1000 °C with current Graphite

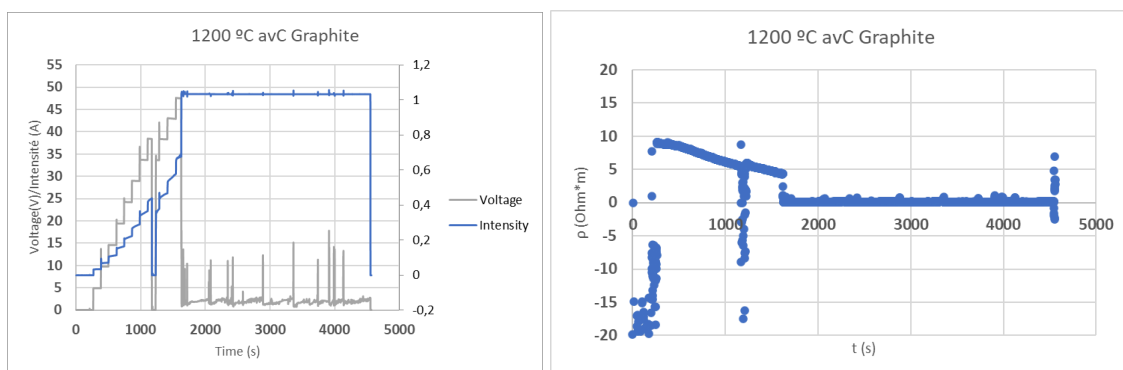


Figure 61. Representation of Tension/Intensity vs Time and Resistivity vs Time for 1200 °C with current Graphite

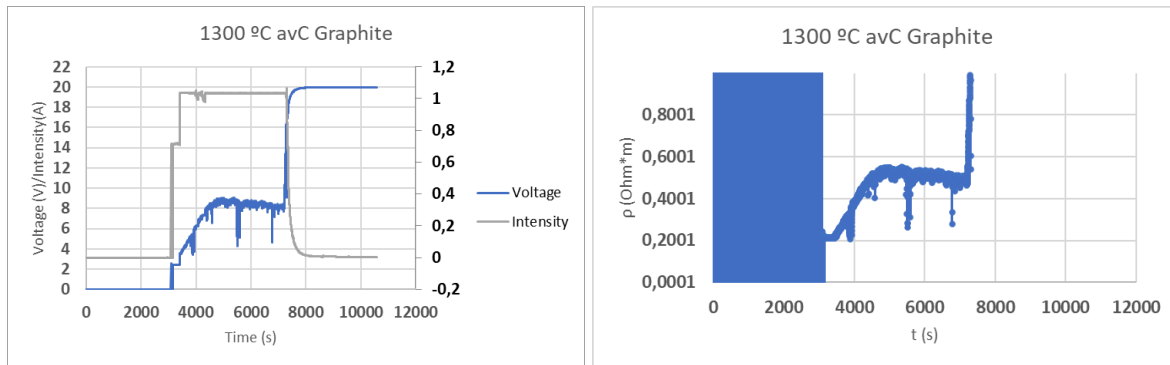


Figure 62. Representation of Tension/Intensity vs Time and Resistivity vs Time for 1300 °C with current Graphite

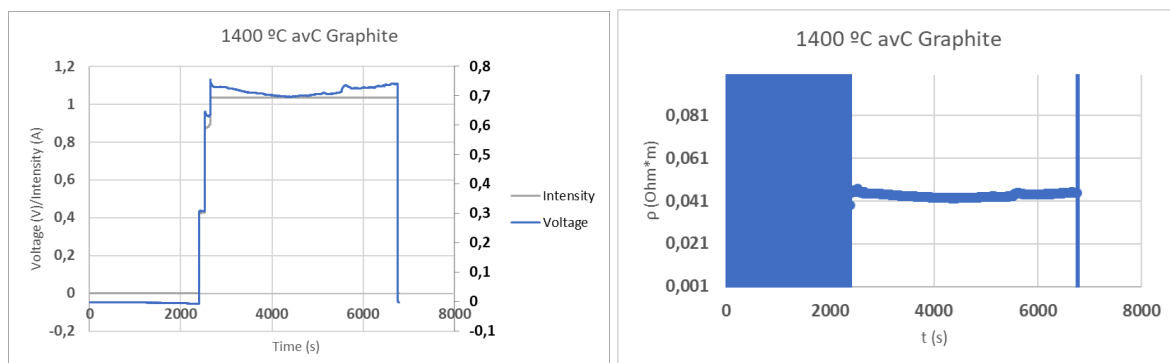


Figure 63. Representation of Tension/Intensity vs Time and Resistivity vs Time for 1400 °C with current Graphite

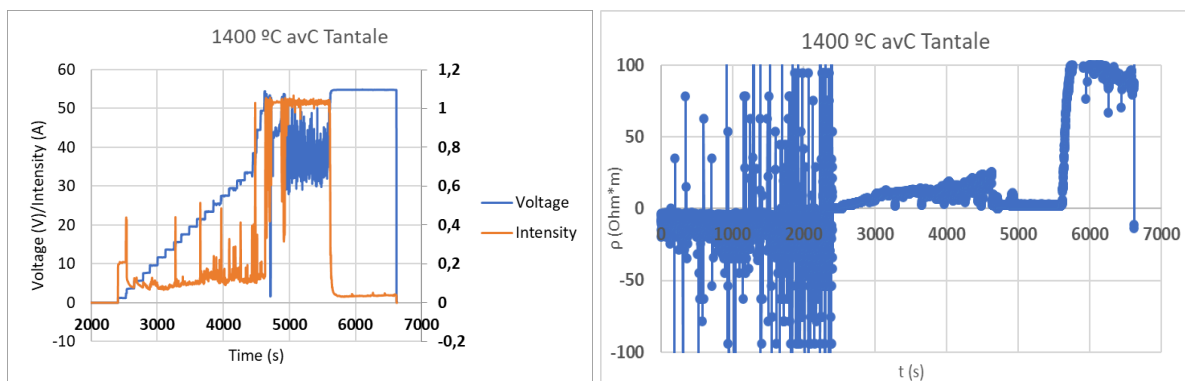


Figure 64. Representation of Tension/Intensity vs Time and Resistivity vs Time for 1400 °C with current Tantalum

P.S. An experiment was also conducted at 1400 °C with current applied, reducing the sintering time to 1 hour, using molybdenum wires to measure the potential difference between the electrodes. However, there were issues reaching the desired current intensity. In addition, both the current and voltage were lower than usual, which may have been due to

poor contact with the thermocouple. The temperature measured by the thermocouple did not match that of the furnace, which is also inconsistent.

III.1.2 Interpretation of transparency curves



Figure 65. Samples sintered at 1200 °C and 1400 °C with and without current

It can be observed that at lower temperatures, the samples exhibit a slightly grayish color, while at higher temperatures, the pellets appear darker due to the diffusion of graphite and tantalum into the samples. It is also worth noting that at lower temperatures, the application of electric current does not produce a significant visible difference, whereas at higher temperatures, the effects are more noticeable. Additionally, there is no significant visual difference between the use of graphite and tantalum (see figure 65).

Furthermore, when placing a phone flashlight behind the sample to evaluate its opacity, it is observed that light passes through the pellets sintered at 1300 °C and 1400 °C where electric current was applied. These conditions appear to promote a notable reduction in porosity and adequate grain growth, which reduces photon interaction with the pores. However, the high presence of diffused carbon during sintering considerably limits the transparency of the samples.

To assess the transparency of the samples, in addition to the flashlight test, measurements were taken using a spectrophotometer. The following graphs were obtained (see figure 66, 67, 68, 69, 70, 71). From them, it can be noted that at 1000 °C, 1100 °C, 1200 °C, and 1300 °C, transparency in the visible spectrum is zero, and in the infrared range, values vary between 0 and 0,5. Moreover, the application of electric current at these temperatures does not show differences compared to tests without current. On the other hand, at 1400 °C, when no current is applied, the results are similar to those of the other tests; however, when current is applied, an increase in transparency in the visible spectrum is observed in the form of two steps. In the infrared spectrum, transparency values reach between 3 and 4. The difference

between using tantalum and graphite lies in the size of the steps in the visible spectrum, larger with graphite, and in the final IR values, which are slightly higher with graphite.

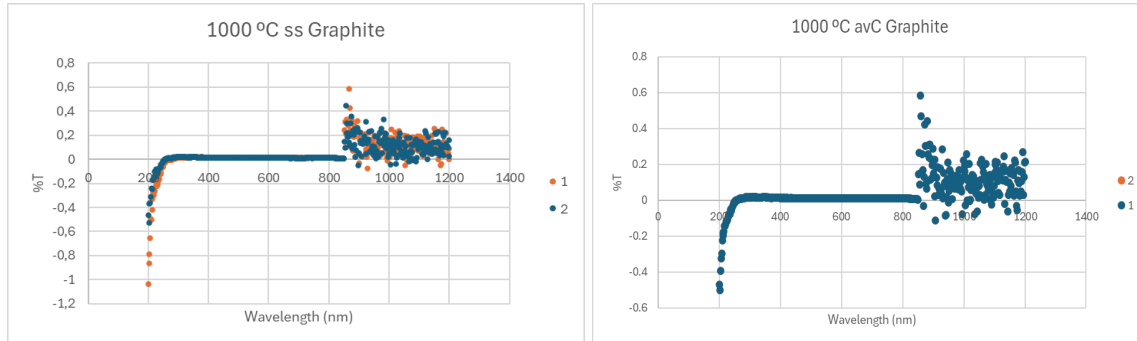


Figure 66. Representation of WaveLength vs %T for 1000 °C without (left) and with (right) current Graphite

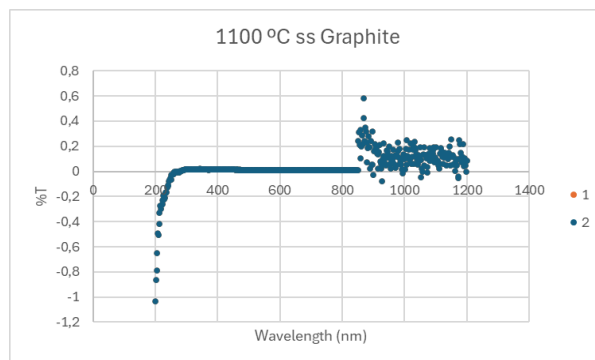


Figure 67. Representation of WaveLength vs %T for 1100 °C without current Graphite

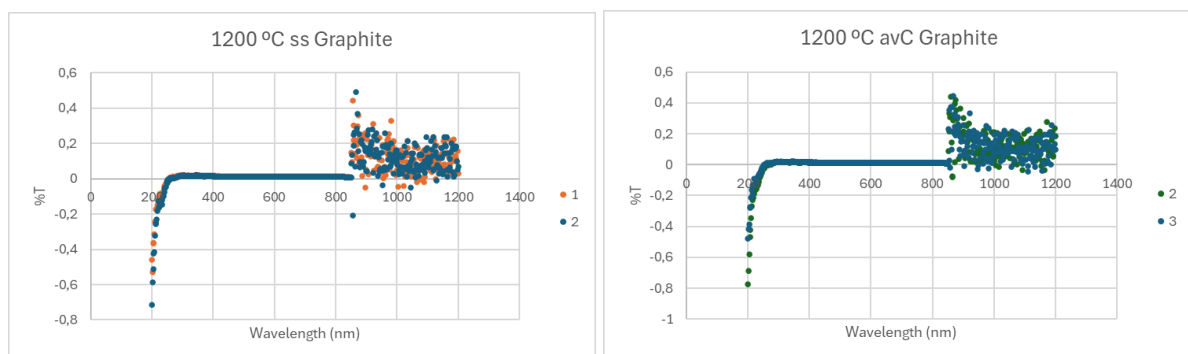


Figure 68. Representation of WaveLength vs %T for 1200 °C without (left) and with (right) current Graphite

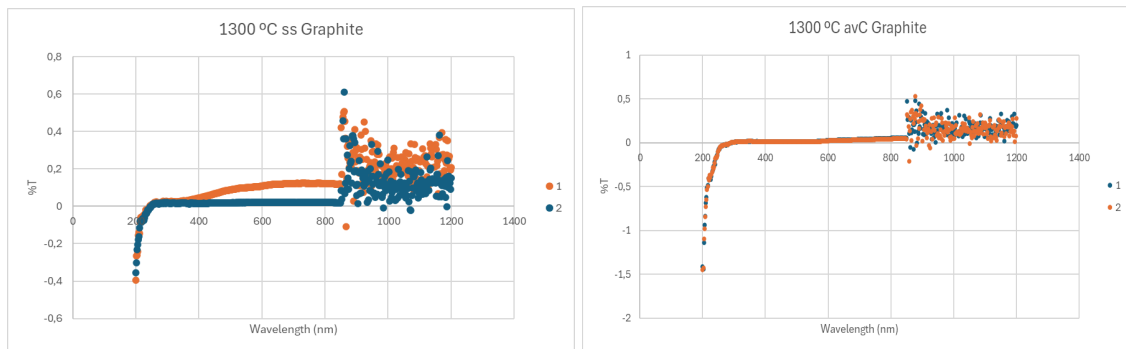


Figure 69. Representation of WaveLength vs %T for 1300 °C without (left) and with (right) current Graphite

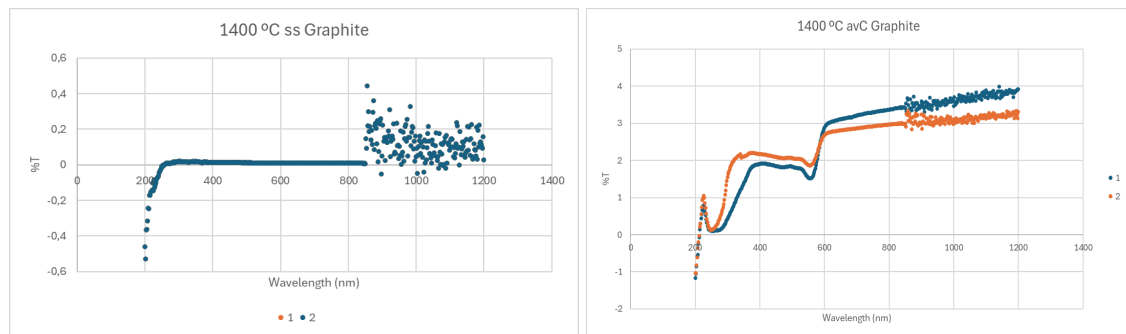


Figure 70. Representation of WaveLength vs %T for 1400 °C without (left) and with (right) current Graphite

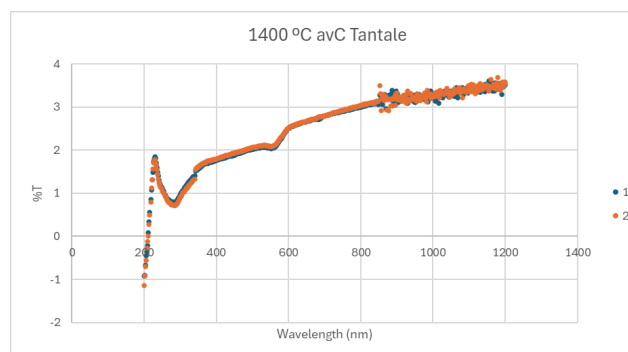


Figure 71. Representation of WaveLength vs %T for 1400 °C with current Tantalum

III.1.3 Interpretation of the density curves

To further examine the effects of temperature and the application of an electric field, an additional test was carried out, consisting of calculating the density of a small portion of the sample using a pycnometer. The results obtained are presented below:

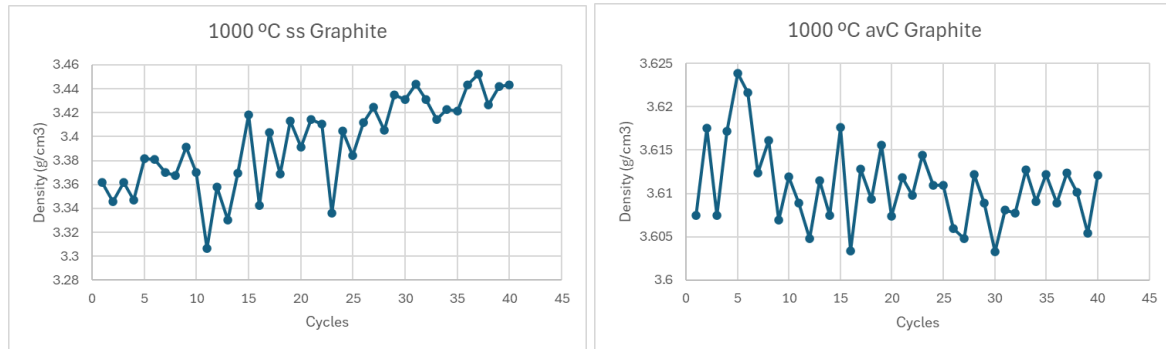


Figure 72. Representation of Density vs Cycles for 1000 °C without (left) and with (right) current Graphite

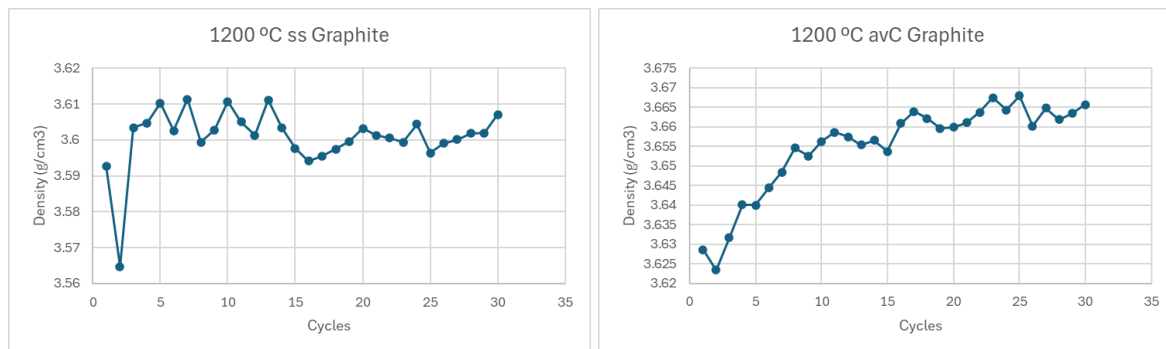


Figure 73. Representation of Density vs Cycles for 1200 °C without (left) and with (right) current Graphite

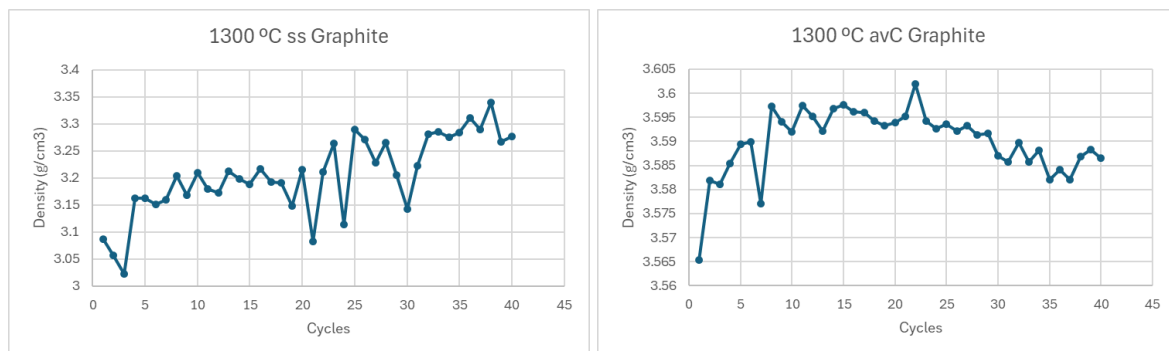


Figure 74. Representation of Density vs Cycles for 1300 °C without (left) and with (right) current Graphite

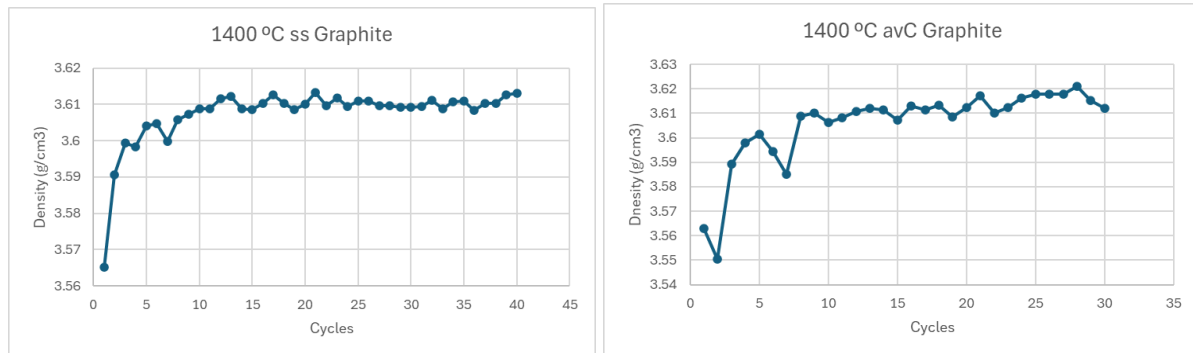


Figure 75. Representation of Density vs Cycles for 1400 °C without (left) and with (right) current Graphite

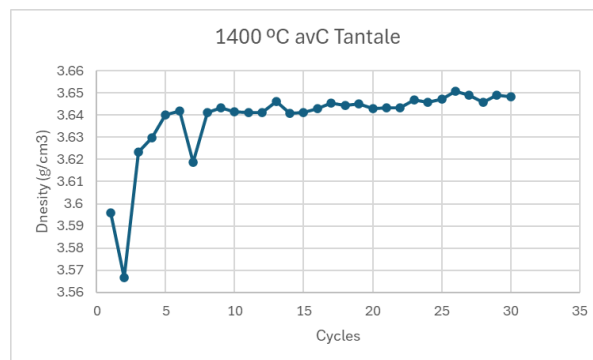


Figure 76. Representation of Density vs Cycles for 1400 °C with current Tantalum

10 purge cycles and 40 measurement cycles were performed.

From the density tests performed, it was observed that the samples subjected to an electric field showed higher density compared to those without applied current, although the difference is not very significant. Additionally, in theory, increasing the temperature should lead to higher density (see **Figure 72, 73, 74, 75, 76**). However, our results do not follow the expected trend, as the densities obtained at 1000 °C and 1200 °C are higher than those recorded at 1300 °C, which is inconsistent. This could be explained by the fact that the AlON formed has a lower density than the starting materials. Moreover, under normal conditions, an increase in temperature would typically result in higher density. This discrepancy could also be due to errors during testing, such as the selection of the sample portion to be analyzed (considering that the pellets are not homogeneous), inaccuracies in the measurements taken by the pycnometer (as they are averaged from several values), or other experimental factors.

Regarding the use of graphite or tantalum, the difference is not substantial, although slightly higher density was achieved with tantalum. At 1400 °C, the difference between applying or not applying an electric current is minimal, with both conditions yielding similar results.

III.1.4 Interpretation of the microstructure

DRX diffractograms

In the pellets sintered without the application of an electric field (see **Figure 78, 79, 81, 82, 85**), no evidence of AlON formation was detected, with the exception of the sample treated at 1400 °C. X-ray diffraction (XRD) analysis revealed that, in general, the composition of the samples remained unchanged, containing only the initial powder mixture components (AlN and Al₂O₃).

However, in the pellet sintered at 1400 °C, near-complete formation of AlON was observed, although some unreacted AlN was still detected. Additionally, carbon diffusion into the interior of the pellets was confirmed by the appearance of a characteristic carbon peak around $2\theta = 26.5^\circ$ in several diffractograms, indicating interaction between the material and the graphite environment during the thermal process.

For the tests conducted under applied electric current (see **Figure 77, 80, 83, 84, 86**), no formation of AlON was observed at 1000 °C and 1100 °C, with the samples still containing the initial components (Al₂O₃ and AlN). In contrast, at 1200 °C, 1300 °C, and 1400 °C, AlON formation was confirmed. At 1200 °C, a considerable amount of unreacted Al₂O₃ and AlN remained, along with detectable carbon. At 1300 °C, small quantities of unreacted Al₂O₃ and AlN were still present. Finally, at 1400 °C, regardless of whether graphite or tantalum was used, near-complete conversion to AlON was achieved, with only trace amounts of AlN and some diffused carbon remaining.

The identified AlON compositions include Al_{2.78}O_{3.65}N_{0.35}, Al_{2.767}O_{3.701}N_{0.299}, and Al_{2.81}O_{3.56}N_{0.44}. All these compositions correspond to face-centered cubic structures belonging to the Fd-3m space group.

Some unidentified peaks were also detected, which may correspond to secondary phases formed as a result of reactions between the sample components and the graphite sheets used during sintering. These additional phases hinder accurate identification of the target compounds and reduce the purity of the sintered materials.

ID ?chantillon COMMANDER (Coupled TwoTheta/Theta)

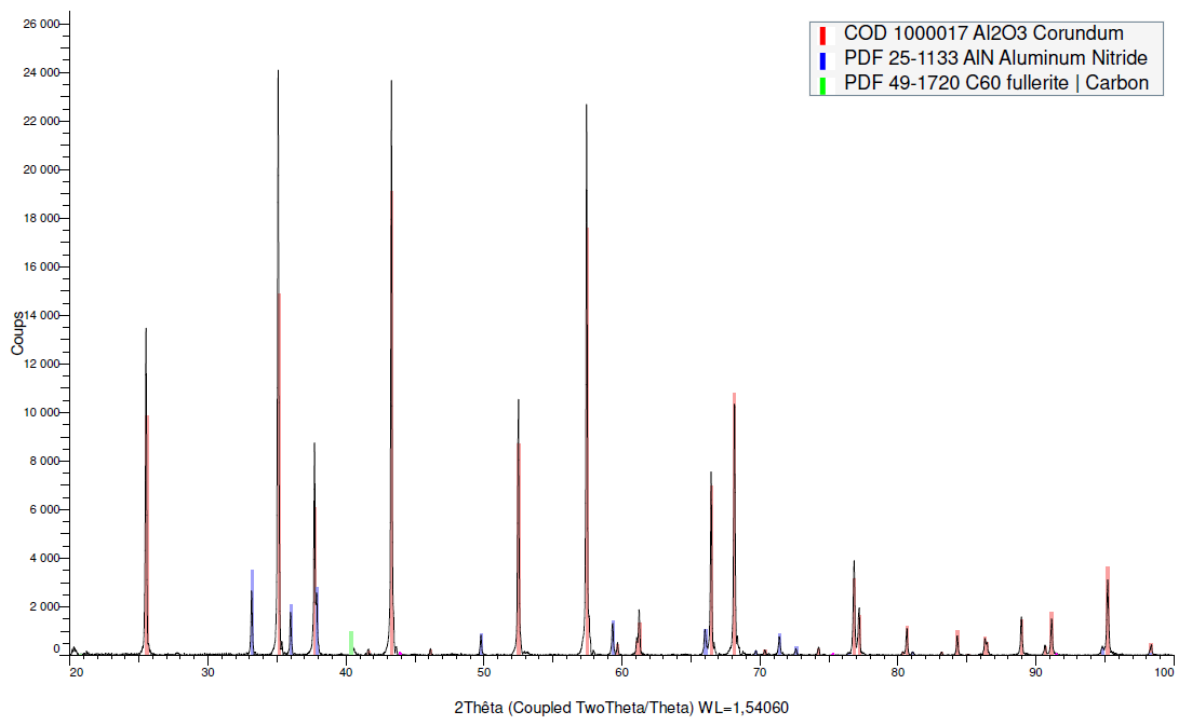


Figure 77. Representation of the diffractogram for 1000 °C with current Graphite

ID ?chantillon COMMANDER (Coupled TwoTheta/Theta)

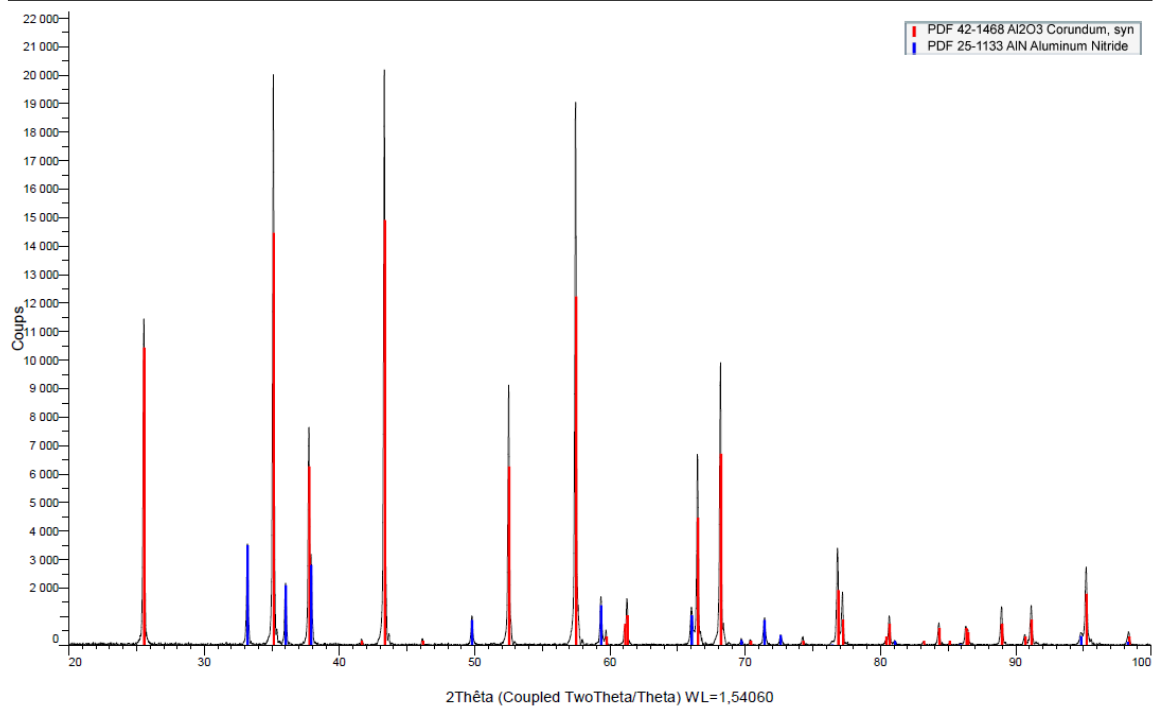


Figure 78. Representation of the diffractogram for 1000 °C without current Graphite

(Coupled TwoTheta/Theta)

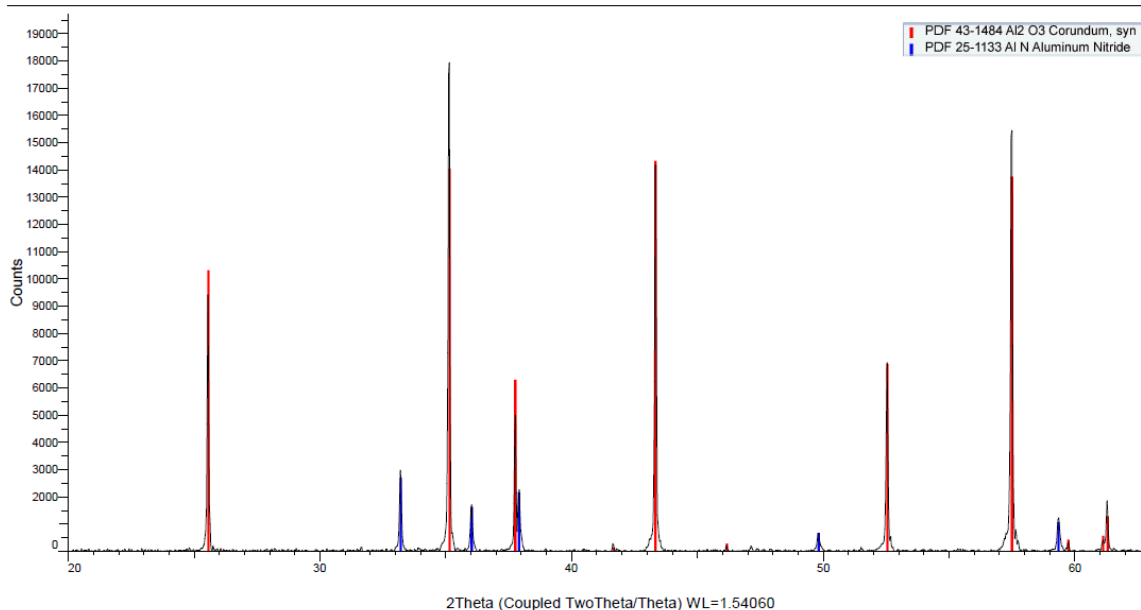


Figure 79. Representation of the diffractogram for 1100 °C without current Graphite

ID ?chantillon COMMANDER (Coupled TwoTheta/Theta)

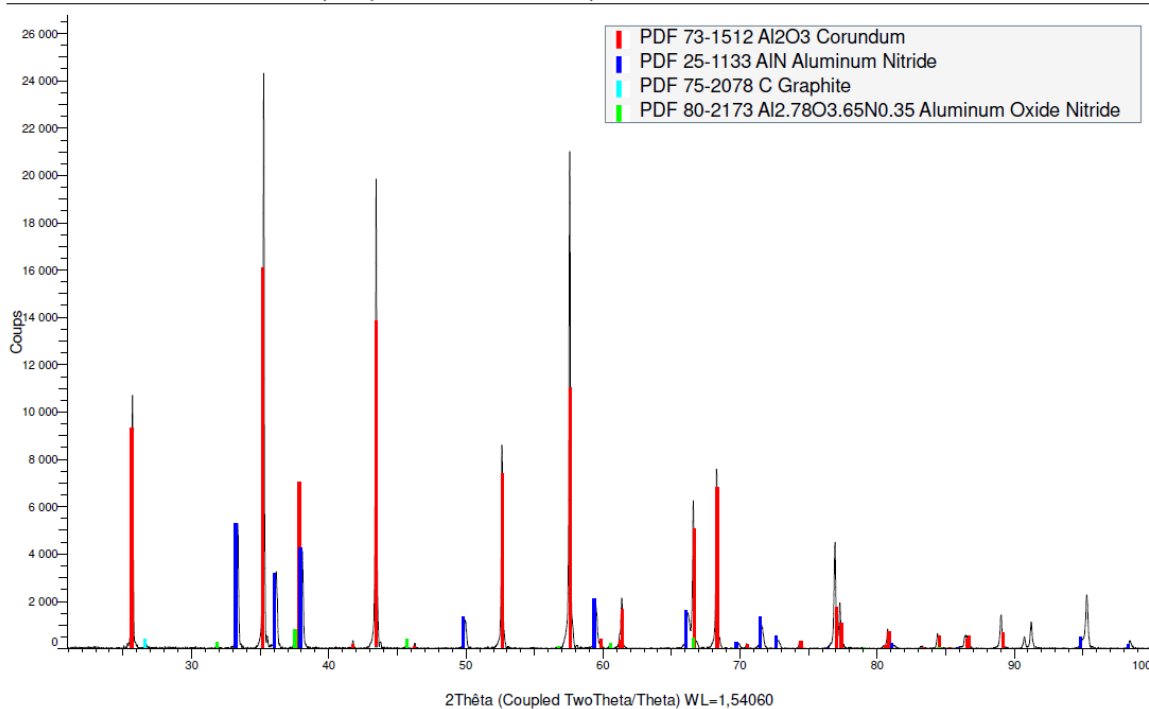


Figure 80. Representation of the diffractogram for 1200 °C with current Graphite

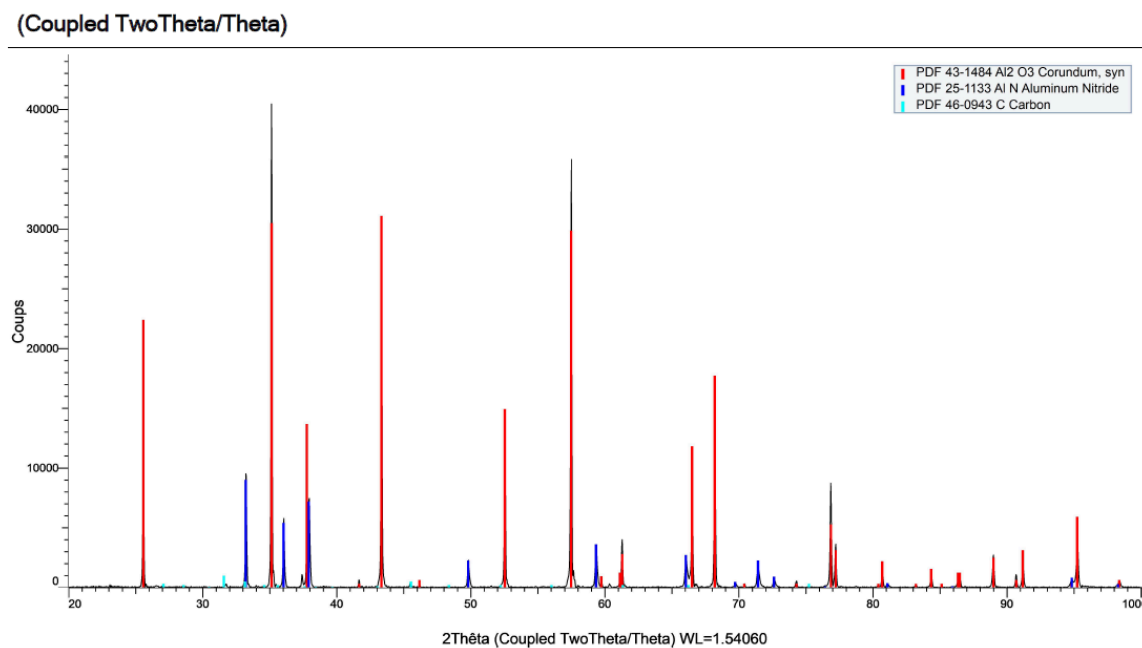


Figure 81. Representation of the diffractogram for 1200 °C without current Graphite

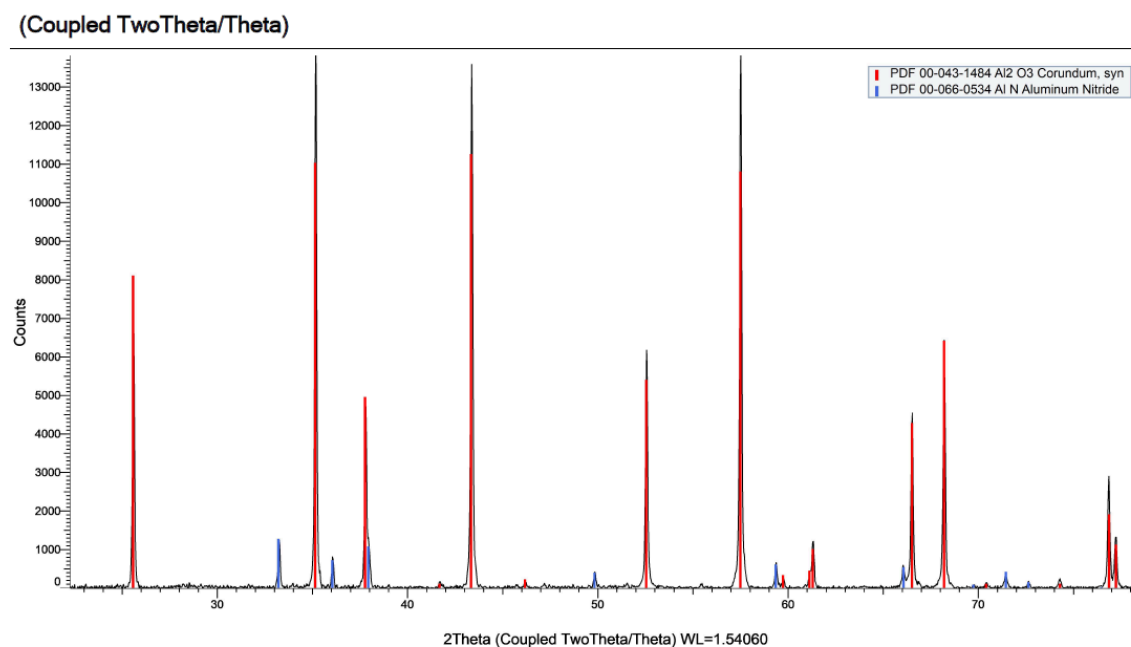


Figure 82. Representation of the diffractogram for 1300 °C without current Graphite

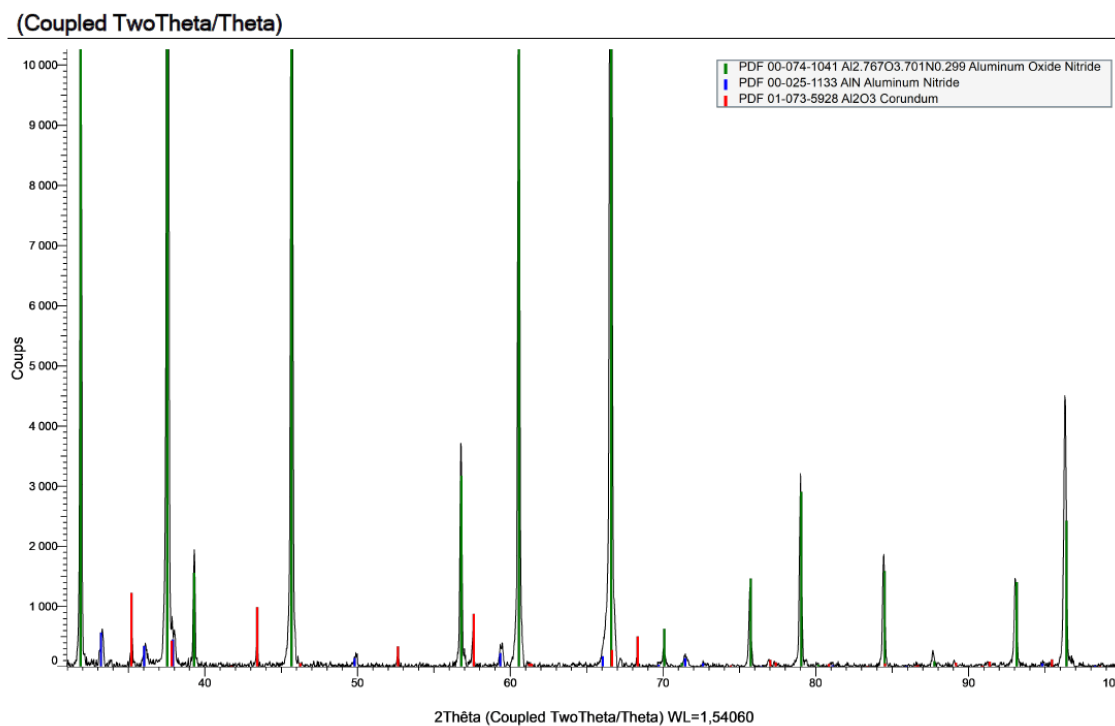


Figure 83. Representation of the diffractogram for 1300 °C with current Graphite

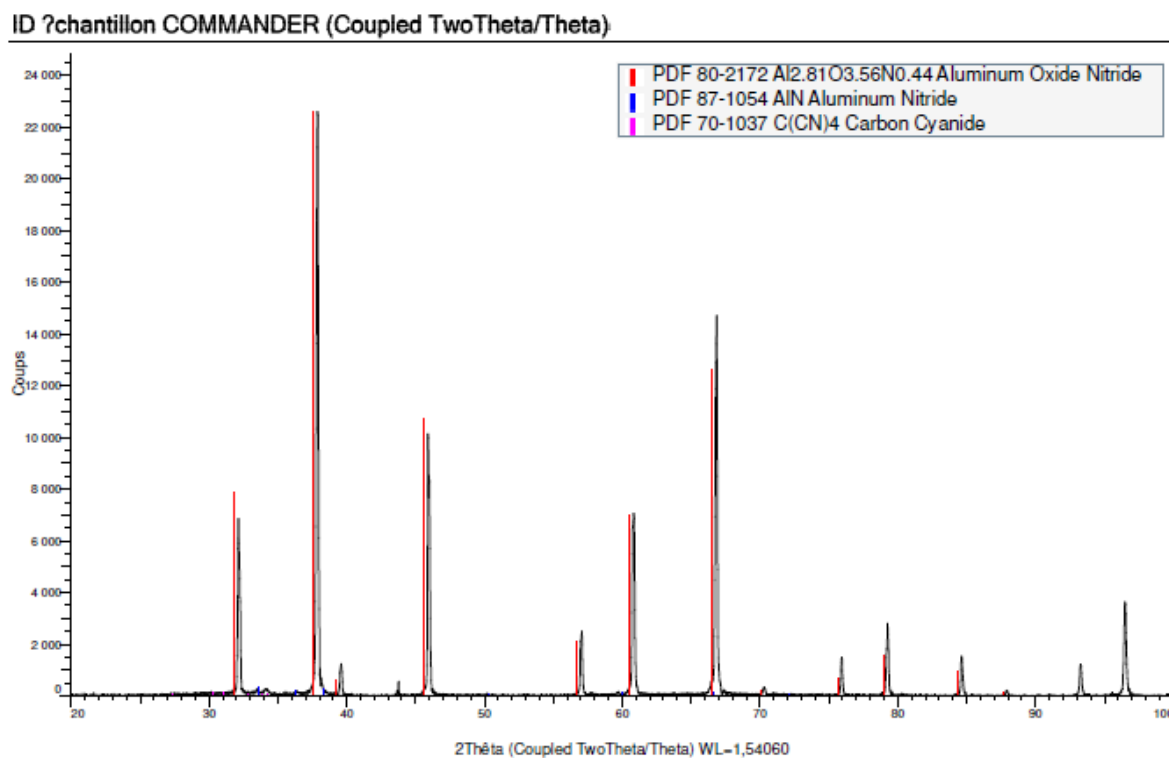


Figure 84. Representation of the diffractogram for 1400 °C with current Graphite

ID ?chantillon COMMANDER (Coupled TwoTheta/Theta)

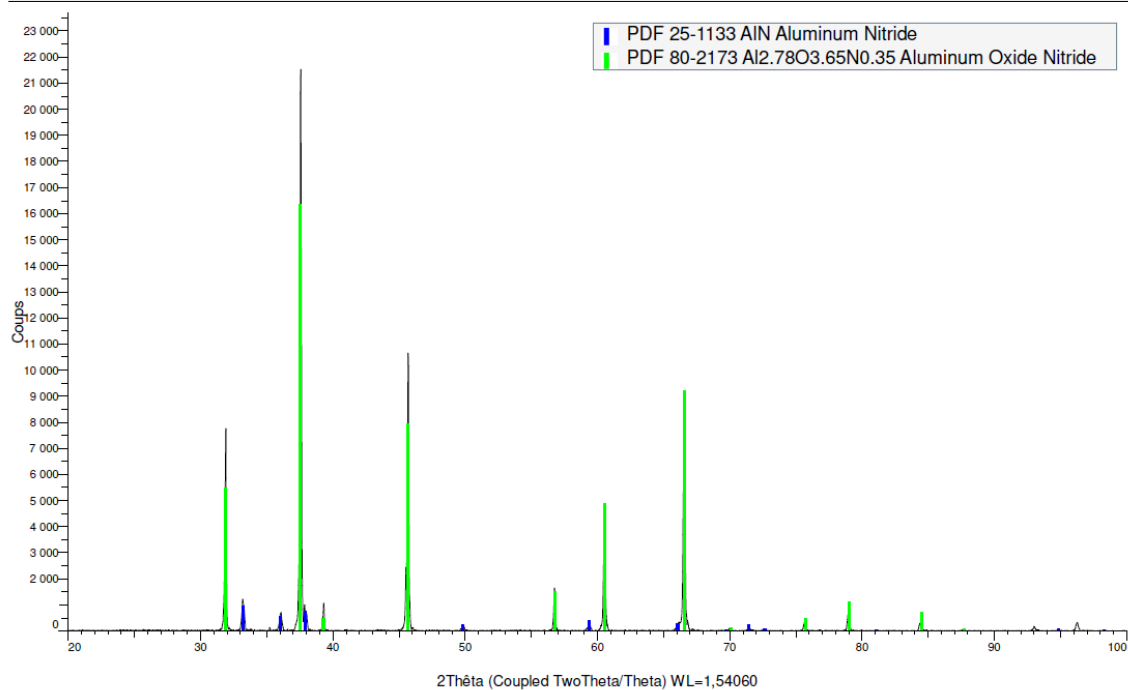


Figure 85. Representation of the diffractogram for 1400 °C without current Graphite

ID ?chantillon COMMANDER (Coupled TwoTheta/Theta)

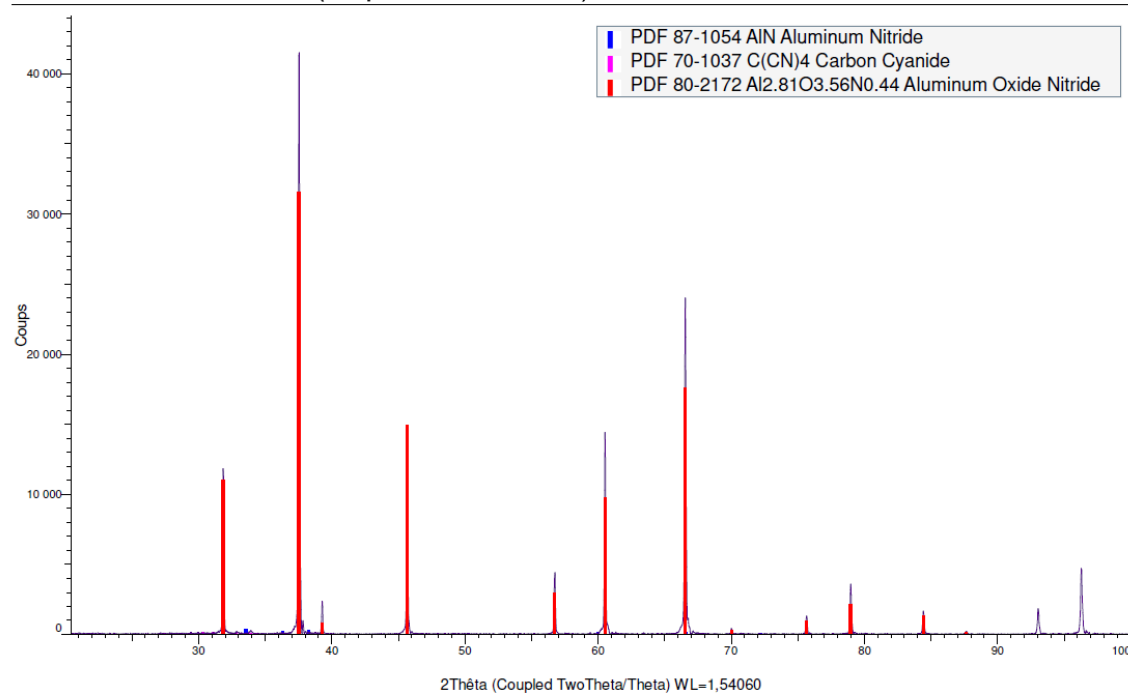


Figure 86. Representation of the diffractogram for 1400 °C with current Tantalum

SEM images

The microstructures obtained via SEM (Scanning Electron Microscopy) of the samples sintered in the HP furnace at different temperatures are shown below.

Although in both cases the pellets exhibit good densification, a significant difference in their microstructures can be observed depending on the sintering temperature and whether or not electric current was applied during the process.

The images corresponding to the pellets sintered with electric current reveal a considerably more compact structure, characterized by densely packed particles and a lower number of pores, which are also smaller in size. In contrast, the pellets processed without current show less effective densification, with higher porosity and larger gaps between particles (**see figure 87, 88, 89, 90, 91, 92**).

It is observed that, as the temperature increases, the densification of the pellet improves, reducing the number of pores and resulting in grains that are increasingly smaller and more tightly packed.

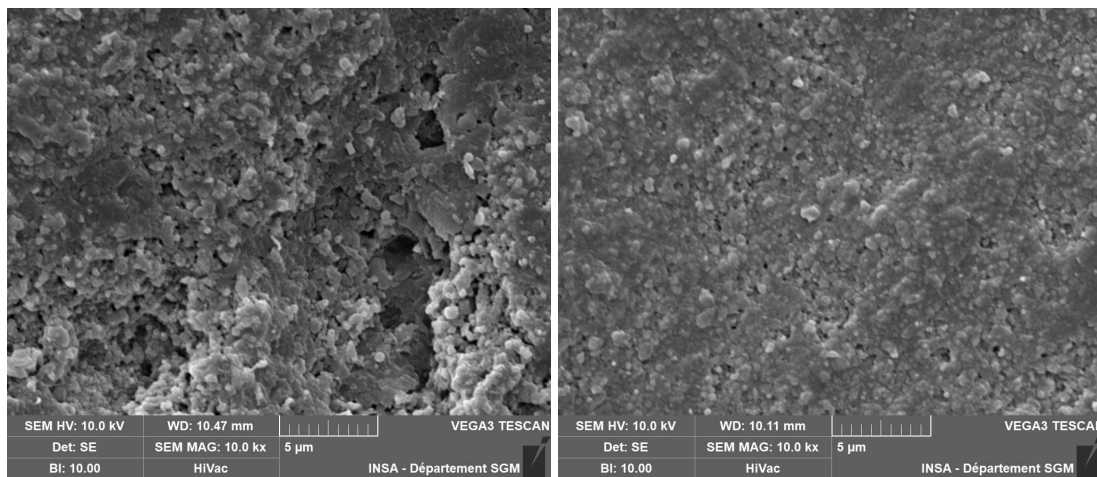


Figure 87. SEM images of the graphite pellets at 1000°C without (left) and with (right) current

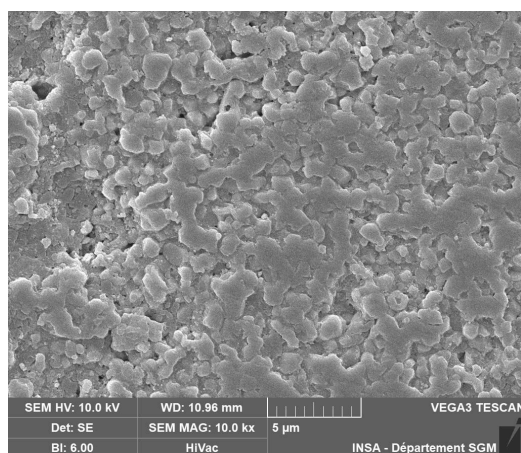


Figure 88. SEM images of the graphite pellets at 1100°C without current

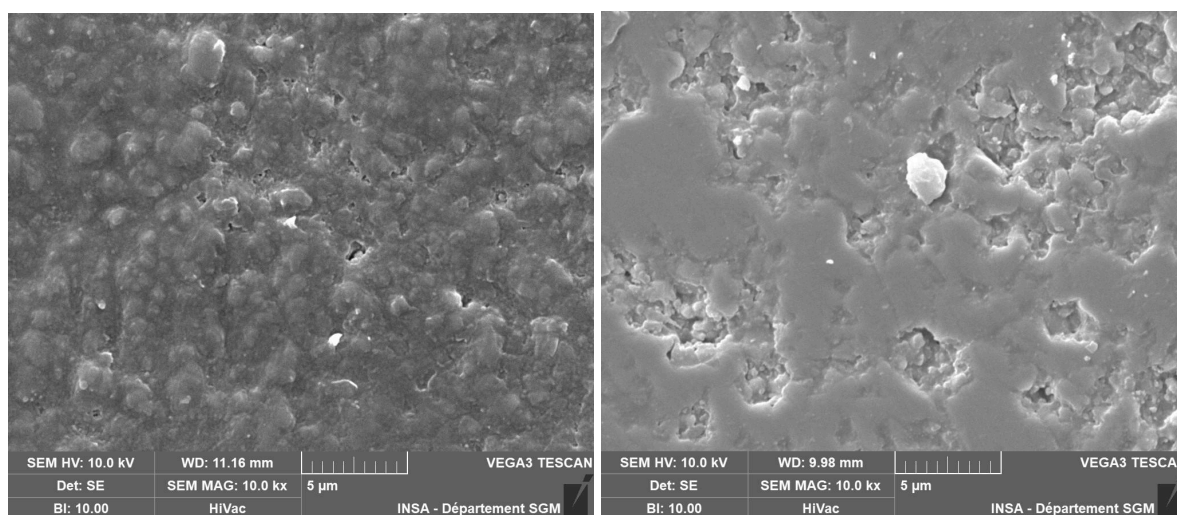


Figure 89. SEM images of the graphite pellets at 1200°C without (left) and with (right) current

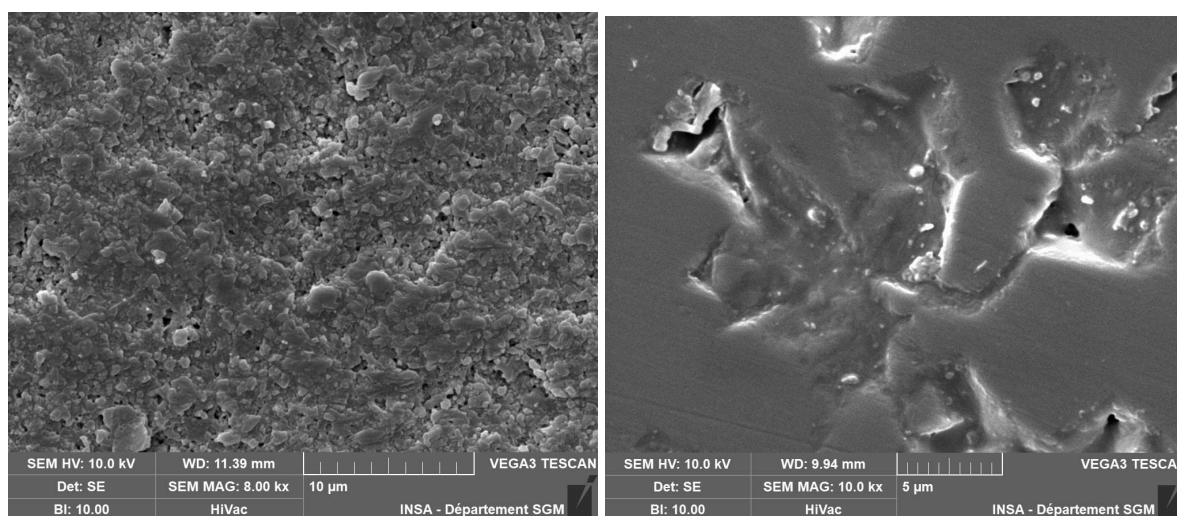


Figure 90. SEM images of the graphite pellets at 1300°C without (left) and with (right) current

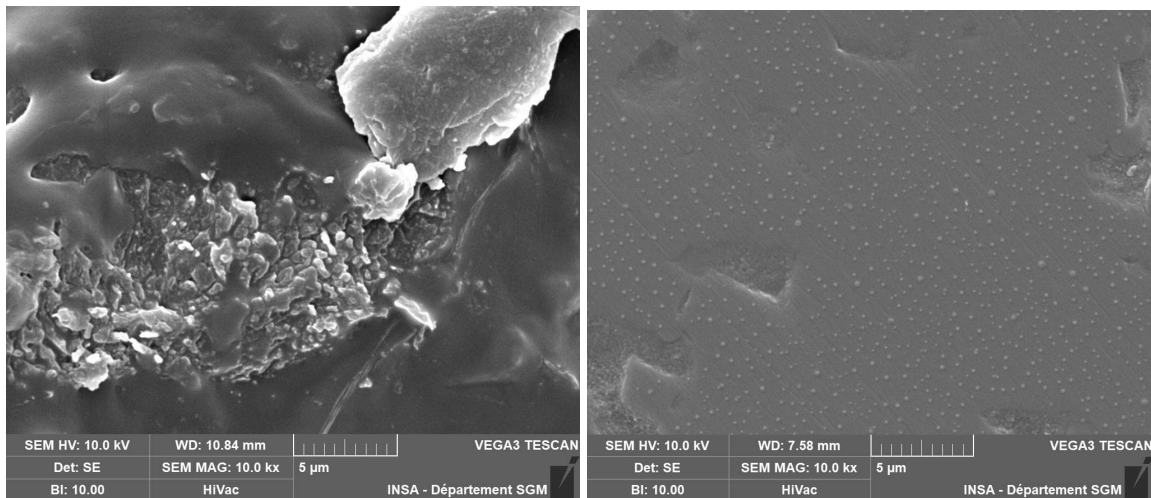


Figure 91. SEM images of the graphite pellets at 1400°C without (left) and with (right) current

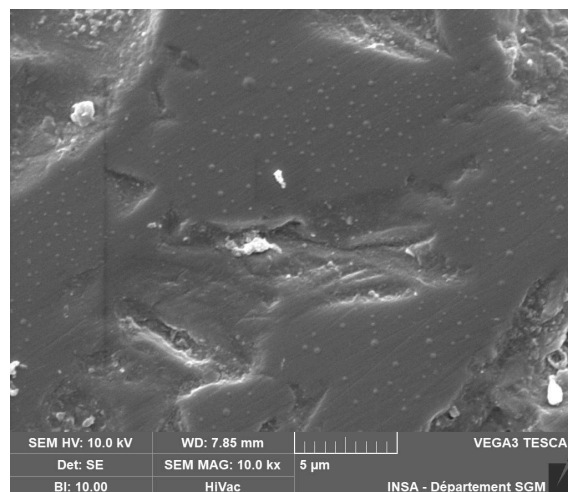


Figure 92. SEM images of the 1400°C tantalum pellets with current

Indeed, porosity appears significantly reduced, with more compact particles and less visible empty space. Therefore, it can be concluded that microstructure evolves significantly with temperature. This is consistent with the principle that increasing temperature during sintering enhances material densification. Additionally, the use of graphite or tantalum does not produce major differences in the final microstructure.

Table 8. Pore size measurement of samples obtained from SEM images

Temperature (°C)	Without current	Average	With current	Average
1000	299,39 nm	289,93 nm	241,48 nm	233,72 nm
	264,50 nm		233,72 nm	
	289,93 nm		221,70 nm	
1100	283,29 nm	241,50 nm		
	241,50 nm			
	232,53 nm			
1200	442,73 nm	424,30 nm	1380 nm	830 nm
	424,30 nm		660 nm	
	356,40 nm		830 nm	
1300	393,39 nm	393,39 nm	10040 nm	6390 nm
	419,54 nm		6390 nm	
	388,90 nm		5100 nm	
1400	543,78 nm	493,75 nm	600 nm	600 nm
	447,96 nm		650 nm	
	489,52 nm		520 nm	

In general, the application of an electric field during the reactive sintering of AlON should promote the formation of denser samples, with a significant reduction in the porosity between grains. Additionally, increasing the temperature should also contribute to this effect.

However, the results obtained do not follow this expected trend. Except for the sample treated at 100 °C, the samples show larger pore sizes when an electric field is applied. Similarly, samples treated at higher temperatures exhibit larger pore sizes than those sintered at lower temperatures. This discrepancy prevents drawing definitive conclusions, as it may be attributed to factors such as the quality of the SEM images or the specific areas selected for analysis. It is important to note that porosity can vary significantly within a single sample, so taking measurements from multiple regions is essential to obtain a more accurate estimate of the actual porosity across the entire surface of the pellets.

Additionally, more detailed measurements of grain and pore sizes were carried out, where it was observed that at 1400 °C without current, the grains appeared smaller than at the same temperature with current. UV-VIS spectrometry was also used to measure the transmittance of the pellets, in order to better evaluate the impact of the electric field on this property and to more clearly identify the compounds present in the samples.

Dureza

The hardness tests were carried out using the Vickers method, which measures a material's resistance to permanent plastic deformation by applying a load with a diamond indenter. The results obtained can be seen below (see Table 9).

Table 9. *Experimental measurements of the samples using the Vickers hardness test*

Sample	Measurement	Value (HV 0.1)
1400 °C avC Graphite	1	773.5
	2	2428.7
	3	97.4
	4	1009.1
	5	197.9
	6	2376.0
	7	2181.5
	8	1754.8
	9	1617.3
	10	87.9
1400 °C avC Tantale	11	1894.2
	12	150.2
	13	845.6
	14	1952.9
	15	1797.1

The results obtained show low homogeneity, as there are areas with higher hardness and others with lower values. Values between 1000–2000 HV 0.1 are considered high, which is

typical for ceramic materials. On the other hand, values below 1000 HV 0.1 are considered low for the type of material being studied. The large variation in the measured values may be attributed to intrinsic factors (such as differences in grain structure and porosity, the presence of different compounds in various regions of the sample, variations in crystallinity, or internal residual stresses) as well as extrinsic factors (such as poor surface polishing or experimental measurement errors).

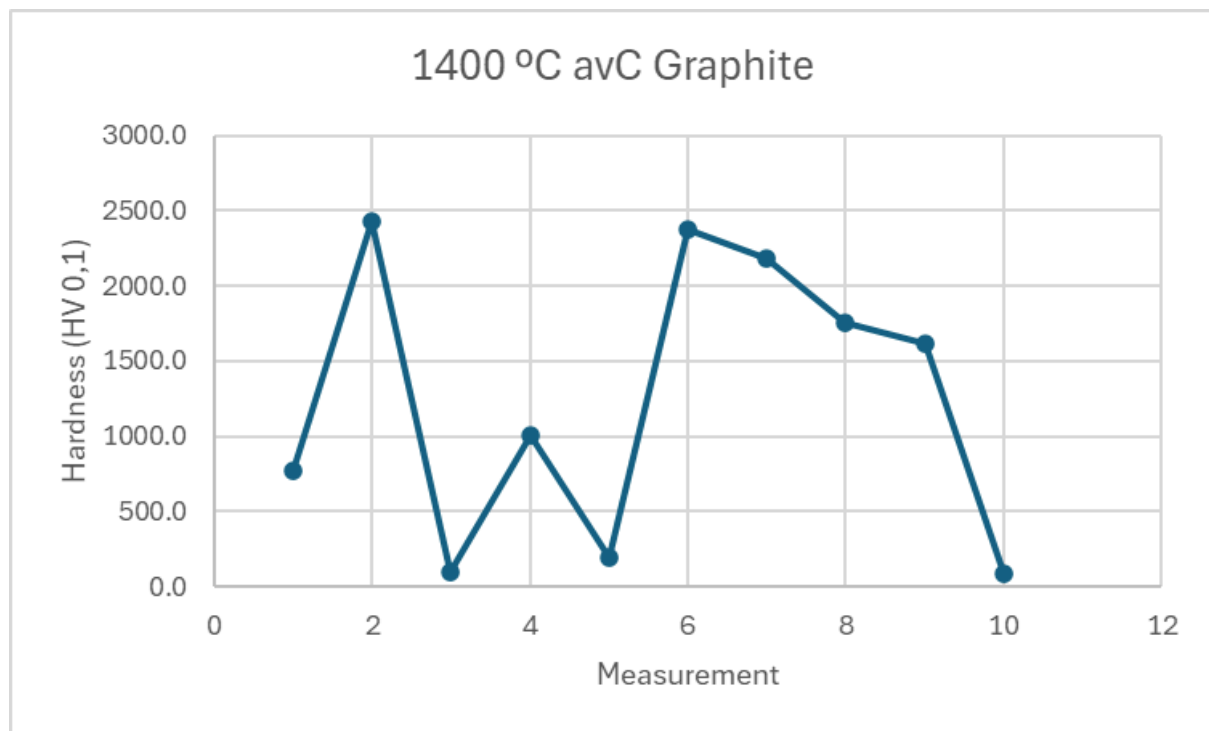


Figure 93. Hardness representation for 1400 °C with current Graphite

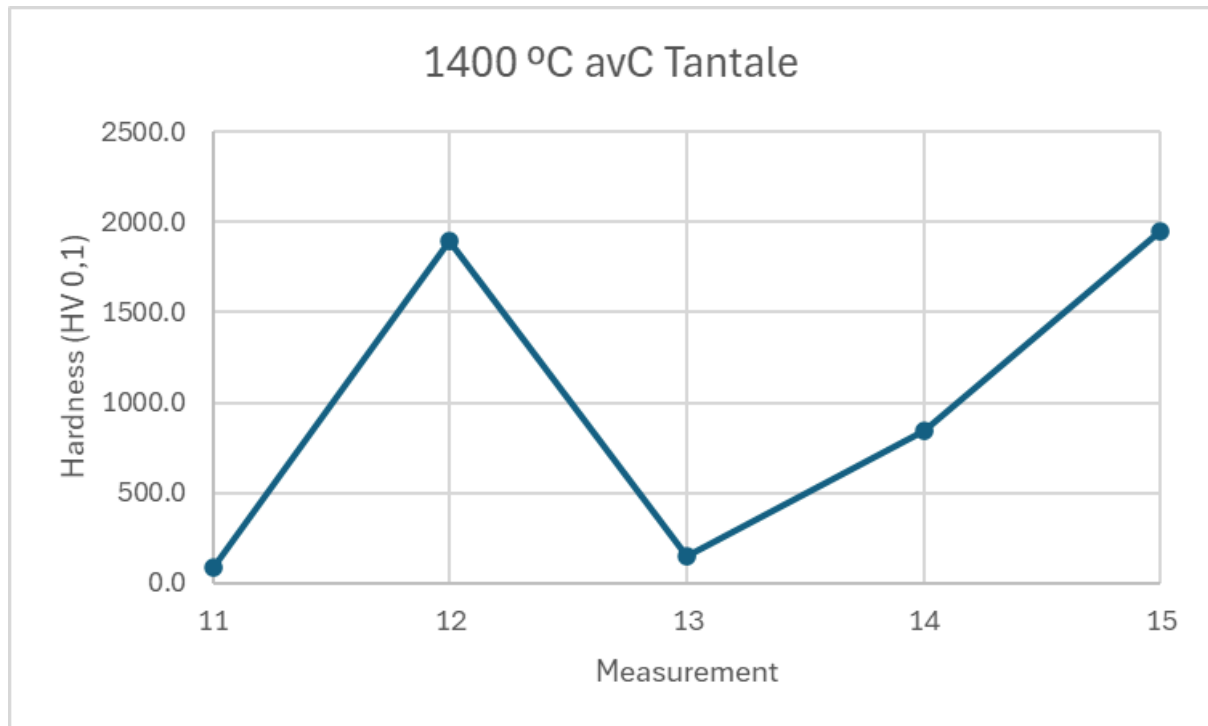


Figure 94. Hardness representation for 1400 °C with current Tantalum

In the sample using graphite, higher hardness values were reached compared to the sample with tantalum; however, overall, the values are fairly similar. Therefore, the use of graphite or tantalum does not significantly influence the hardness of the resulting material (see **Figure 93 and 94**).

III.2. Perspectives and future research objectives

Transparent aluminum continues to show great potential as research advances in refining its properties and developing methods to improve its feasibility for large-scale production [35]. Some of the future research objectives include:

- 1) Improvement of optical transparency: Achieving near-100% transparency in the visible and near-infrared ranges through optimization of parameters such as microstructure, control of secondary phase formation, and elimination of porosity.
- 2) More efficient sintering processes to reduce production costs and processing time, including the use of more effective sintering additives that do not compromise transparency or purity.

- 3) Industrial-scale production, enabling the adaptation of laboratory techniques to large-scale manufacturing without compromising quality and property stability.
- 4) Broader integration of the material across additional fields and sectors.
- 5) Sustainability and environmental impact reduction, by minimizing the use of harmful materials and reducing energy consumption.
- 6) Analysis and optimization of advanced properties, such as the material's behavior under extreme conditions. Additionally, exploring new compositions and dopants may impart other functional properties.

III.3. Conclusion

After conducting a thorough analysis of the results obtained from the microstructural observations, it was evident that the different parameters do have a significant influence on the final product.

The resistivity values obtained were inconsistent, as ceramic materials typically exhibit high resistivity, behaving as non-conductive, insulating materials.

Regarding transparency, it begins to appear at 1300 °C when applying electric current.

Furthermore, density tests show that it increases as temperature increases and an electric field is used.

As for the influence of the electric field, it was observed that the pellets sintered without current showed no signs of AlON formation, regardless of the temperature used—except at the highest temperature, 1400 °C, where AlON was detected. X-ray diffraction analysis confirmed that these samples only contained the initial powder mixture components (AlN and alumina).

In contrast, pellets sintered under an electric field began to show traces of AlON at 1200 °C. From 1300 °C onward, most of the initial powder mixture began transforming into AlON. The identified AlON compositions include $\text{Al}_{2.78}\text{O}_{3.65}\text{N}_{0.35}$, $\text{Al}_{2.767}\text{O}_{3.701}\text{N}_{0.299}$, and

$\text{Al}_{2.81}\text{O}_{3.56}\text{N}_{0.44}$. At 1400 °C, nearly complete formation of AlON was observed, with minor residual AlN.

From a microstructural standpoint, both the application of electric current and the increase in temperature should have an impact on the densification level of the samples; however, this was not evident in the reduction of porosity, which would be expected to decrease with higher temperature and the application of an electric field during sintering. The crystal defects in AlON are oxygen vacancies that occur when oxygen atoms are replaced by nitrogen atoms [21].

Finally, conclusive hardness values could not be determined, as the results varied significantly between different areas of the samples. This confirms that the homogeneity within the specimens is relatively low.

Final Conclusion

In summary, the flash sintering process of AlON represents a complex area of research, where various physical, chemical, and mechanical phenomena are closely interconnected. To achieve the highest possible transparency and density, it is essential to precisely control several sintering parameters, such as porosity, sintering temperature, the intensity of the applied electric field, the atmosphere, and the sintering duration. It has been demonstrated that experiments conducted at higher temperatures and with the application of an electric field yield improved results, both physically and microstructurally.

More in-depth investigations and the acquisition of additional experimental data could provide valuable, yet undiscovered insights. This opens up multiple opportunities for the development of new hypotheses and research directions, establishing this field as a promising area for future studies.

SDGs (Sustainable Development Goals)

SDG 9: Industry, Innovation and Infrastructure

This project is directly aligned with SDG 9, as it promotes technological innovation within the industrial sector of advanced materials. The main goal of the work is to improve the ceramic sintering process, specifically in the production of AlON (aluminum oxynitride)—a high-performance transparent ceramic material used in applications such as optical armor,

sensors, and aerospace components.

Through the application of flash sintering assisted by an electric field, the project proposes an alternative to conventional sintering that offers multiple industrial benefits, such as a significant reduction in time and temperature, and optimization of the material's microstructure.

SDG 12: Responsible Consumption and Production

This project directly contributes to a more sustainable and efficient production model. By implementing flash sintering assisted by an electric field, a notable reduction is achieved in both the time and temperature required for the sintering of ceramic materials like AlON. This leads to lower consumption of energy resources and a reduction in the use of polluting materials such as graphite.

SDG 7: Affordable and Clean Energy

As a secondary objective, the project promotes greater energy efficiency in industrial processes. It does so by reducing the thermal demands of conventional sintering through the application of an electric field, which enables lower energy usage during material fabrication.

SDG 4: Quality Education

Also as a secondary contribution, the project is an academic research initiative that fosters advanced scientific and technical training, contributing to high-quality higher education, particularly in STEM fields (science, technology, engineering, and mathematics).

SDG 13: Climate Action

Also as a secondary contribution, industrial processes are optimized to use less energy and generate less waste, contributing to the reduction of emissions associated with material manufacturing (greenhouse gases), which helps mitigate environmental impact.

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Annexes

1) Conversion from mole fraction to mass fraction

First, the mass fraction equation will be defined as follows (see equation 12, 13):

$$W_1 = \frac{m_1}{m_1 + m_2} \quad (12) \quad W_2 = \frac{m_2}{m_1 + m_2} \quad (13)$$

being m_1 , m_2 the masses of AlN and Al_2O_3 respectively

To obtain the masses of both components (see equation 14, 15):

$$m_1 = x_1 \cdot M_1 \quad (14)$$

$$m_2 = x_2 \cdot M_2 = (1 - x_1) \cdot M_2 \quad (15)$$

being x_1 , x_2 the mole fractions of AlN and Al_2O_3

Substituting equation (14)(15) in equation (12)(13), we have (see equation 16, 17):

$$W_1 = \frac{x_1 \cdot M_1}{x_1 \cdot M_1 + (1 - x_1) \cdot M_2} = \frac{x_1 \cdot M_1}{x_1 \cdot (M_1 - M_2) + M_2} \quad (16)$$

$$W_2 = \frac{(1 - x_1) \cdot M_2}{x_1 \cdot M_1 + (1 - x_1) \cdot M_2} = \frac{(1 - x_1) \cdot M_2}{x_1 \cdot (M_1 - M_2) + M_2} \quad (17)$$

$$M_1 = 40,9882 \text{ g/mol}$$

$$M_2 = 101,96 \text{ g/mol}$$

$$x_1 = [27 - 40]\%$$

Finally, the corresponding mass fraction values are:

$$W_1 = \frac{0,27 \cdot M_1}{0,27 \cdot (M_1 - M_2) + M_2} = 12,94\%$$

$$W_1 = \frac{0,4 \cdot M_1}{0,4 \cdot (M_1 - M_2) + M_2} = 21,13\%$$

2) Conversion of units of force

$$1 \text{ meT} = 1000 \text{ kgf}$$

$$1 \text{ kgf} = 9,80665 \text{ N}$$

$$1 \text{ meT} = 9806,65 \text{ N}$$