

COLD SINTERING PROCESS: SUSTAINABILITY MEETS ADVANCED CERAMICS PROCESSING

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ABSTRACT

Ceramic sintering is essential for obtaining dense and functional bodies, but it typically requires high temperatures, long dwell times and high energy consumption, limiting the integration of thermally sensitive phases. The Cold Sintering Process (CSP) has emerged as a sustainable low-temperature alternative based on the combined action of pressure, moderate heating and a Transient Liquid Phase (TLP). This work presents CSP as an advanced ceramic processing methodology, from the densification mechanisms and key processing variables to its application in solid battery materials. Special attention is given to the processing of NASICON-type solid-state electrolytes (SSEs) and composite architectures developed in our laboratory. In LATP-based systems, CSP enabled the fabrication of dense ceramic and polymer-in-ceramic composites by tuning particle size, TLP content and polymer-salt additives. This approach was also extended to Na-based NASICON electrolytes, where sodium salt doping further improved densification and electrochemical performance. Finally, CSP was used to fabricate integrated LFP|LATP-based composite electrolyte bilayers, demonstrating the potential of this route for producing electrode-electrolyte architectures at only 150 °C. Overall, these results highlight CSP not only as a low-energy sintering technique, but as a versatile tool for designing advanced ceramic and hybrid materials for next-generation energy technologies.

1. INTRODUCTION

Ceramic manufacturing is intrinsically linked to high-temperature processing. Conventional solid-state sintering remains the most established route to obtain dense ceramic components, but it typically requires temperatures above 1000 °C, long dwell times and significant energy input. These conditions make ceramic sintering an energy-intensive step, contributing to high production costs and greenhouse gas emissions. In addition, high-temperature treatments may promote excessive grain growth, volatilization of selected elements, undesired phase transformations and chemical degradation, ultimately limiting the microstructural control and functional performance of advanced ceramics.

These limitations become even more critical when ceramic processing involves thermally sensitive or chemically incompatible phases. Polymers, salts, hydrated phases, metastable compounds or low-melting functional additives cannot generally withstand conventional firing temperatures. As a result, the fabrication of dense ceramic-based composites, hybrid materials or multi-functional architectures often requires alternative processing strategies capable of reducing the thermally associated costs while preserving phase stability and interfacial compatibility.

In this context, the Cold Sintering Process (CSP) has emerged as a promising low-temperature route for the densification of ceramics and ceramic-based composites. CSP combines uniaxial pressure, moderate-to-low temperatures and a transient liquid phase (TLP) to activate mass transport through dissolution–precipitation and pressure-assisted particle rearrangement mechanisms. Unlike conventional sintering, where densification is mainly driven by high-temperature solid-state diffusion, CSP enables consolidation at temperatures typically below 300 °C. This opens new opportunities for processing thermally sensitive systems and integrating inorganic phases with polymers, salts or other functional components that would otherwise degrade during conventional firing.

The potential of CSP as a sustainable processing route is illustrated in **Figure 1**, where the energy consumption required to obtain highly dense ZnO ceramics is compared for conventional solid-state sintering (CSS), spark plasma sintering (SPS), CSP and a hybrid CSP-SPS route. While all methods achieved relative densities close to 99%, CSP reduced the energy consumption by almost a 95-fold reduction in comparison to CSS.^[1] This comparison highlights one of the central advantages of CSP: the possibility of achieving high densification with drastically reduced energy demand, while also limiting microstructural coarsening. Therefore, CSP should not be regarded only as a low-temperature alternative to conventional sintering, but as an enabling ceramic processing technique for sustainable manufacturing, microstructural design and the development of advanced functional materials.

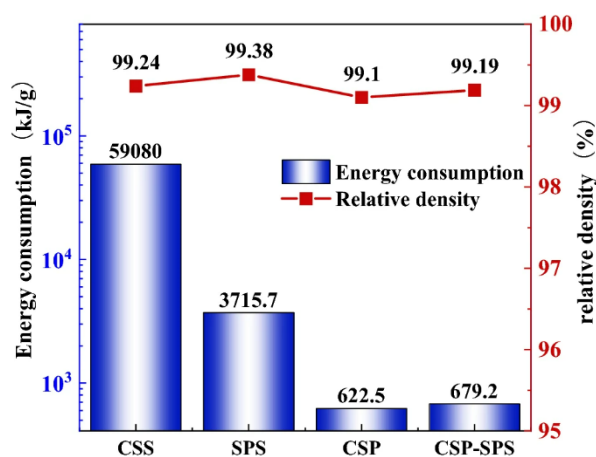


Figure 1. Energy consumption of different sintering techniques for the case of ZnO densification ^[1].
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2. FUNDAMENTALS OF THE COLD SINTERING PROCESS

2.1. Densification mechanisms

CSP is a low-temperature, pressure-assisted sintering route in which a ceramic powder is mixed with the required amount of TLP and consolidated under uniaxial pressure and moderate heating. Its densification mechanism is commonly associated with *pressure solution creep*, a liquid-mediated process involving dissolution, mass transport and reprecipitation on the grain boundaries of the particles. Under applied pressure, highly stressed particle–particle contacts partially dissolve into the confined liquid. The dissolved species are then transported through the TLP and reprecipitated in regions with lower pressure, promoting neck formation, pore filling and progressive densification. Although this dissolution–precipitation process is generally considered the main mechanism, particle rearrangement, plastic deformation and local chemical reactions may also contribute depending on the material–solvent system.^[2,3]

2.2. Key processing variables

The efficiency of CSP is governed by the coupled effect of four main groups of variables: pressure, temperature, TLP and material characteristics. Pressure promotes particle rearrangement, increases the contact area and generates the local stress gradients required for pressure solution creep. Temperature controls the kinetics of dissolution–precipitation, TLP evaporation and microstructural evolution, with relevant parameters including maximum temperature, heating rate and dwell time. The TLP determines the chemical pathway for densification through its amount, composition, pH, polarity, boiling point, viscosity and reactivity with the ceramic surface. Finally, the material itself defines the tendency to be sintered of the system through particle size, particle size distribution, morphology, solubility, crystal structure and defect chemistry. A summary of the critical variables is shown in **Figure 2**.

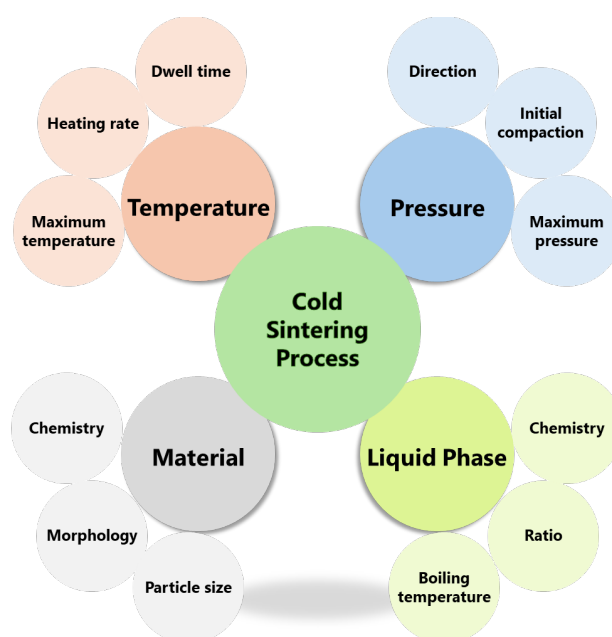


Figure 2. Summary of the main variables controlling CSP.

2.3. Typical processing cycle

ZnO is one of the most widely used model systems to illustrate CSP. An example of a reported cycle involves mixing ZnO powder with a 20 wt.% acetic acid aqueous solution (1m), loading the wetted powder into a metallic die, applying uniaxial pressures up to 750 MPa and heating to approximately 170–250 °C for 1 h. Whereas conventional solid-state sintering required 1300 °C for 5 h to reach comparable densification.^[4] A visual comparative of the differences in the sintering cycles for this case is shown in **Figure 3**.

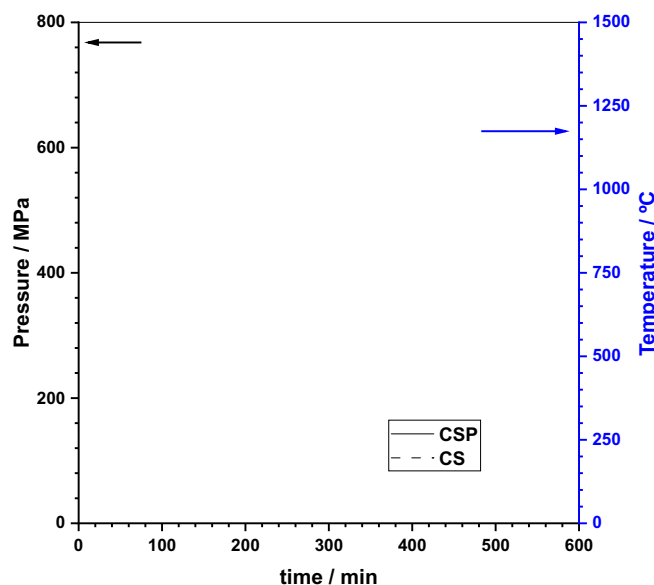


Figure 3. Pressure and temperature cycles for sintering of ZnO bodies by CS and CSP.

2.4. Experimental setup and scalability

At laboratory scale, CSP is typically carried out using a simple uniaxial press equipped with a stainless-steel die and mold, resistive heating elements and temperature control by thermocouples. The powder is manually mixed with the TLP, loaded into the mold and simultaneously subjected to pressure and moderate heating. This open and relatively simple setup makes CSP highly attractive for laboratory screening, since it avoids complex equipment.

Despite this simplicity, scale-up remains one of the main challenges. Most studies are still performed on small pellets, while larger demonstrations remain limited. C. Randall, *et al.* reported ZnO samples with a record size of 25 cm² processed by CSP at reduced pressures, although applied pressure, microstructural homogeneity and solvent evaporation gradients remain critical issues in the scale up.^[5] That is, producing a 1 m² body at 700 MPa would require about 71,000 tonnes-force, which is unrealistic for direct batch processing. Therefore, industrial implementation will likely require reduced-pressure formulations, smaller batch modules, or continuous roller-based concepts, where pressure is applied locally over smaller contact areas with controlled temperature and residence time.

3. STATE OF THE ART IN COLD-SINTERED CERAMICS

The current state of the art shows that CSP is no longer limited to proof-of-concept demonstrations. A wide variety of ceramic systems have already been processed, including model oxides, dielectric ceramics, semiconductors, and ceramic-polymer composites. These examples demonstrate the potential of CSP to process materials and interfaces with lower temperatures and costs compared to conventional high-temperature routes. **Table 1** summarizes selected examples that illustrate sintering conditions, densification level and technological relevance of some cold-sintered ceramics.

Material	P (MPa)	T (°C)	Time (min)	Relative density (%)	Ref.
ZnO	200	150 / 200	30 + 30	97.36 / 99.43	[6]
ZnO	1500 / 300	250	10 + 45	97.5	[7]
BaTiO ₃	520	300	720	98	[8]
BaTiO ₃	500	220	90	93.5	[9]
LATP	510 / 600	140 / 280	60	90 / 98	[10]

Table 1. Examples of highly reported materials processed by CSP.

3.1. Solid-state batteries as an emerging opportunity

The rapid expansion of electric mobility is accelerating the demand for advanced battery technologies. According to the Global EV Outlook 2026, electric car sales exceeded 20 M units in 2025, representing 25% of new car sales worldwide, and are expected to reach around 23 M units in 2026. In parallel, global EV battery deployment is projected to increase from approximately 1.2 TWh in 2025 to almost 3 TWh by 2030, highlighting the scale of the industrial opportunity for battery materials and manufacturing technologies.^[11]

Within this context, all-solid-state batteries (ASSBs) are regarded as one of the most promising next-generation battery concepts. In ASSBs, the toxic and flammable liquid electrolyte used in conventional Li-ion batteries is replaced by a solid-state electrolyte (SSE) which can improve safety and potentially enable high-energy-density lithium-metal anodes.^[12] Ceramic SSEs, including NASICON, LISICON, perovskite and garnet-type materials, are particularly important due to their high ionic conductivity, mechanical robustness and electrochemical stability. These properties are essential for replacing the current organic liquid electrolyte.

However, careful processing is still required to control the density, porosity, grain boundaries and solid-solid interfaces of these ceramic components. While conventional sintering can achieve high densification, it requires high temperatures that can cause excessive grain growth, lithium volatilization, secondary reactions and incompatibility with polymers, salts and electrode components. CSP is therefore an attractive option for ASSBs manufacturing, as it enables low-temperature densification and co-processing of ceramic, polymeric and salt-based phases, which would be difficult to integrate using conventional firing methods.

4. CASE STUDY: CSP APPLIED TO SOLID-STATE ELECTROLYTES

Beyond the general state of the art, our research group ChEMTECh at Universitat Jaume I, led by A. Barba-Juan and N. Vicente-Agut, has focused on the detailed study of CSP as a low-temperature processing route for ceramic SSEs. The work has mainly addressed lithium and sodium NASICON-type SSEs based on $\text{Li}_{1.3}\text{Al}_{0.3}\text{Ti}_{1.7}(\text{PO}_4)_3$ (LATP) and $\text{Na}_3\text{Zr}_2\text{Si}_2\text{PO}_{12}$ (NZSP), respectively, and related polymer-in-ceramic composites and multilayer architectures. These studies have explored how CSP parameters, ceramic particle size and distribution, TLP, sintering aids and composite formulation affect densification, microstructure, ionic transport and cyclability in simple cell concepts.

4.1. In situ EIS monitoring of the CSP

A key aspect of this work is the development of a CSP setup coupled with *in situ* Electrochemical Impedance Spectroscopy (EIS). The system is based on the commonly reported CSP setup but takes advantage of an inner insulation and a conductive die connected to a potentiostat, allowing the isolated electrical response of the material to be followed during the CSP cycle (see **Figure 4**).

This approach uses EIS not only as a conventional tool to evaluate the final ionic transport properties of the solid electrolyte, but also as an *in situ* probe of the sintering process itself. By monitoring the impedance evolution during heating, dwell and cooling, it becomes possible to correlate changes in the electrical response with particle rearrangement, liquid-assisted densification, grain-boundary formation and microstructural evolution. In this way, CSP is no longer treated as a black-box processing step, but as a dynamic process that can be electrically monitored. ^[13]

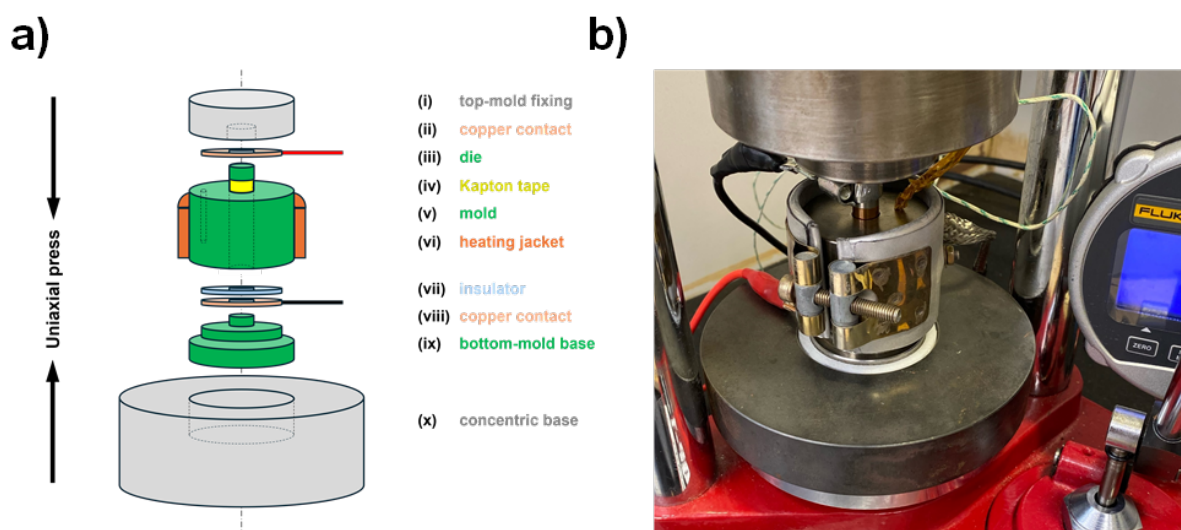


Figure 4. a) Scheme of the experimental parts. b) Actual image of the CSP setup working.

4.2. CSP of Li and Na SSEs, composites and bilayers

The CSP studies carried out in our group followed a processing–microstructure–performance approach, where the main variables investigated were the particle size and distribution, TLP amount and chemistry, applied pressure, polymer–salt additives and multilayer configurations. The main results are summarized in **Table 2**, showing how these parameters directly affect relative density and ionic conductivity.

Material	Additives	TLP	d ₅₀ (μm)	P (MPa)	T (°C)	t (min)	φ (%)	σ (S/cm)
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	-	HAc 3m (15 wt.%)	0.875	700	150	90	79.81	1.25·10 ⁻⁴
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	-	HAc 3m (15wt.%)	0.645	700	150	90	80.81	8.21·10 ⁻⁵
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	-	HAc 3m (15wt.%)	0.415	700	150	90	81.2	6.90·10 ⁻⁵
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	-	HAc 3m (15wt.%)	0.376	700	150	90	81.17	6.40·10 ⁻⁵
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	-	HAc 3m (15wt.%)	0.268	700	150	90	81.38	5.17·10 ⁻⁵
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	-	HAc 3m (15wt.%)	0.190	700	150	90	81.47	3.15·10 ⁻⁵
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (5 wt.%)	0.190	700	150	90	96.2	5.77·10 ⁻⁵
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (10 wt.%)	0.190	700	150	90	97.7	6.94·10 ⁻⁵
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (15 wt.%)	0.190	700	150	90	98.9	1.32·10 ⁻⁴
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (15 wt.%)	1.208	700	150	90	88.4 6	2.95·10 ⁻⁴
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (15 wt.%)	0.875	700	150	90	89.9 4	2.13·10 ⁻⁴
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (15 wt.%)	0.415	700	150	90	91.22	1.69·10 ⁻⁴
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (15 wt.%)	0.415	300	150	90	87.5 6	1.69·10 ⁻⁴
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (15 wt.%)	0.415	500	150	90	88.0 3	3.76·10 ⁻⁴
Li _{1.3} Al _{0.3} Ti _{1.7} (PO ₄) ₃	PEO ₂ - LiTFSI (10 wt.%)	HAc 3m (15 wt.%)	0.415	720	150	90	90.5 0	5.00·10 ⁻⁴

$\text{Na}_{3.4}\text{Zr}_{1.9}\text{Zn}_{0.1}\text{Si}_{2.2}\text{P}_{0.8}\text{O}_{12}$	NaTFSI (20 wt.%)	HAc 3m (15 wt.%)	1.42	720	150	90	87.7	$3.10 \cdot 10^{-4}$
$\text{Na}_{3.4}\text{Zr}_{1.9}\text{Zn}_{0.1}\text{Si}_{2.2}\text{P}_{0.8}\text{O}_{12}$	NaTFSI (20 wt.%)	HAc 3m (15 wt.%)	0.65	720	150	90	87.7	$2.60 \cdot 10^{-4}$
$\text{Na}_{3.4}\text{Zr}_{1.9}\text{Zn}_{0.1}\text{Si}_{2.2}\text{P}_{0.8}\text{O}_{12}$	NaPF₆ (20 wt.%)	HAc 3m (15 wt.%)	1.42	720	150	90	94.3	$8.10 \cdot 10^{-4}$
$\text{Na}_{3.4}\text{Zr}_{1.9}\text{Zn}_{0.1}\text{Si}_{2.2}\text{P}_{0.8}\text{O}_{12}$	NaPF₆ (20 wt.%)	HAc 3m (15 wt.%)	0.65	720	150	90	91.1	$5.10 \cdot 10^{-4}$

d_{50} : Particle size; P : Uniaxial pressure; T : Temperature; t : Sintering time; φ : Relative density; σ_{tot} : Total ionic conductivity.

Table 2. Summary of some of the main results obtained in our laboratory with CSP. Bold numbers highlight the studied variables.

In initial studies for LATP-based ceramic electrolytes, the combination of 15 wt.% 3m acetic acid as TLP, low temperature (150 °C) and high uniaxial pressure (720 MPa) allowed the preparation of SSEs without thermal post-treatments. *In operando* EIS showed that the impedance evolution during the dwell step can be used to follow the evaporation of the TLP and the progress of densification (**Figure 5 a**). In Bi_2O_3 -doped LATP, optimized CSP conditions led to samples with porosity lower than 15% and ionic conductivities of $4.5 \cdot 10^{-5} \text{ S cm}^{-1}$.^[13]

The role of LATP particle size was then studied systematically. Smaller particles improved densification because of the larger contact area and enhanced pressure-solution mechanisms, but they also increased the total grain boundary surface, reducing the total ionic conductivity. Therefore, the optimum point was not simply the finest powder, but a compromise between densification and ionic transport. In pure LATP, a mean particle size of around 0.415 μm provided the best balance, with a relative density of 81.2% and ionic conductivities close to $7 \cdot 10^{-5} \text{ S cm}^{-1}$.^[14]

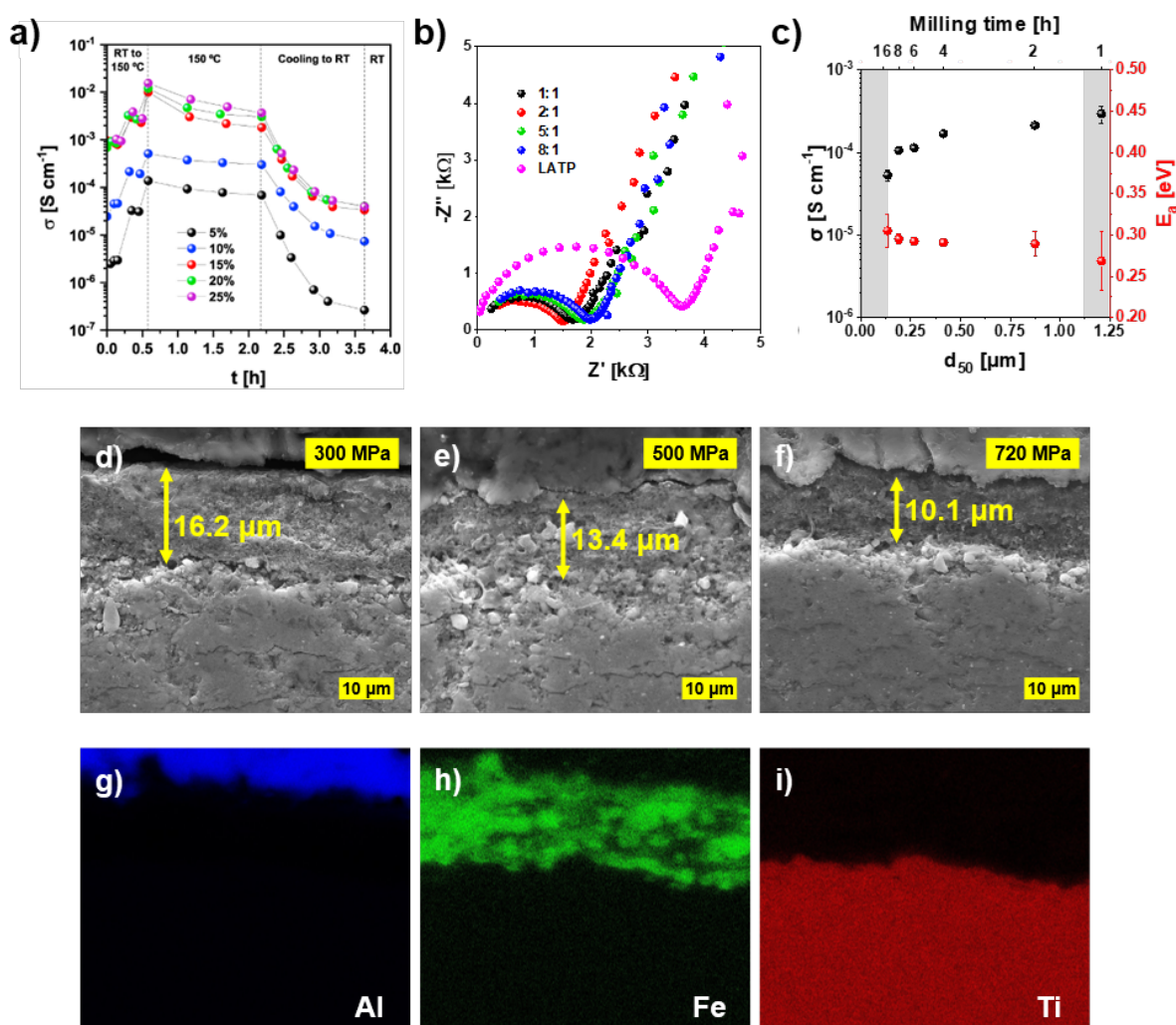
A major improvement was achieved by moving from purely ceramic LATP electrolytes to polymer-in-ceramic composite solid electrolytes (CSEs), using 90 wt.% LATP and 10 wt.% $\text{PEO}_n\text{-LiTFSI}$. The low processing temperature of CSP enabled the incorporation of the polymer-salt phase, which would not be possible in conventional ceramic sintering. With 15 wt.% TLP and an $\text{EO}:\text{Li}^+$ ratio of 2:1, CSEs reached porosities lower than 10%, ionic conductivities higher than of $1 \cdot 10^{-4} \text{ S cm}^{-1}$ and a reduced activation energy of 0.298 eV (**Figure 5 b**). Further microstructural optimization of these LATP- $\text{PEO}_2\text{-LiTFSI}$ composites confirmed again the key role of particle size, with $d_{50} = 0.415 \mu\text{m}$ providing competitive properties: around 91% relative density, $1.7 \cdot 10^{-4} \text{ S cm}^{-1}$ of ionic conductivity and stable operation for more than 500h under current densities of 0.1 mA cm^{-2} in Li symmetric cells (**Figure 5 c**).^[15]

CSP was also used to process integrated LFP|LATP-based CSE bilayers, demonstrating its potential beyond single electrolyte pellets. By co-sintering the LFP cathode and the LATP- $\text{PEO}_2\text{-LiTFSI}$ CSEs at 150 °C, intimate electrode-electrolyte contact was achieved (**Figure 5 d-i**). The applied pressure was critical: bilayers processed at 300 MPa showed higher ionic conductivity, around $5 \cdot 10^{-4} \text{ S cm}^{-1}$, and good electrochemical performance, with capacities of 160 mAh $\text{g}_{\text{LFP}}^{-1}$ close to the theoretical value (170 mAh $\text{g}_{\text{LFP}}^{-1}$) at C/10 (10 hours of charge and discharge) and

stable cycling at higher rates. In contrast, excessive pressure, 720 MPa, induced structural distortion of the LFP crystal phase and the subsequent capacity fading. Follow-up GITT, *operando* XRD and XPS analyses confirmed that high-pressure processing can generate irreversible phase changes and Fe^{3+} -rich inactive domains, highlighting the need to optimize CSP pressure in multilayer battery architectures.^[16,17]

Finally, the same CSP strategy was extended to Na-based NASICON solid electrolytes, specifically $\text{Na}_{3.4}\text{Zr}_{1.9}\text{Zn}_{0.1}\text{Si}_{2.2}\text{P}_{0.8}\text{O}_{12}$. In this case, the chemistry of the TLP was especially relevant: 15 wt.% of 3M acetic acid preserved the NASICON structure more effectively than NaOH and led to higher ionic conductivity. The addition of sodium salts at the grain boundaries further improved the performance, with 20 wt.% NaPF_6 -doped samples reaching 94.3% relative density and $8 \cdot 10^{-4} \text{ S cm}^{-1}$. These electrolytes also showed promising electrochemical behavior, with stable Na symmetric cell cycling for more than 500 h and Na|NVP half-cell capacities above 100 mAh $\text{g}_{\text{NVP}}^{-1}$ at C/10 with good stability at higher rates (**Figure 5 j**).^[18]

Overall, these results demonstrate that CSP is not only a low-temperature densification method for solid electrolytes, but also a versatile processing platform for engineering ceramic, composite and multilayered components.



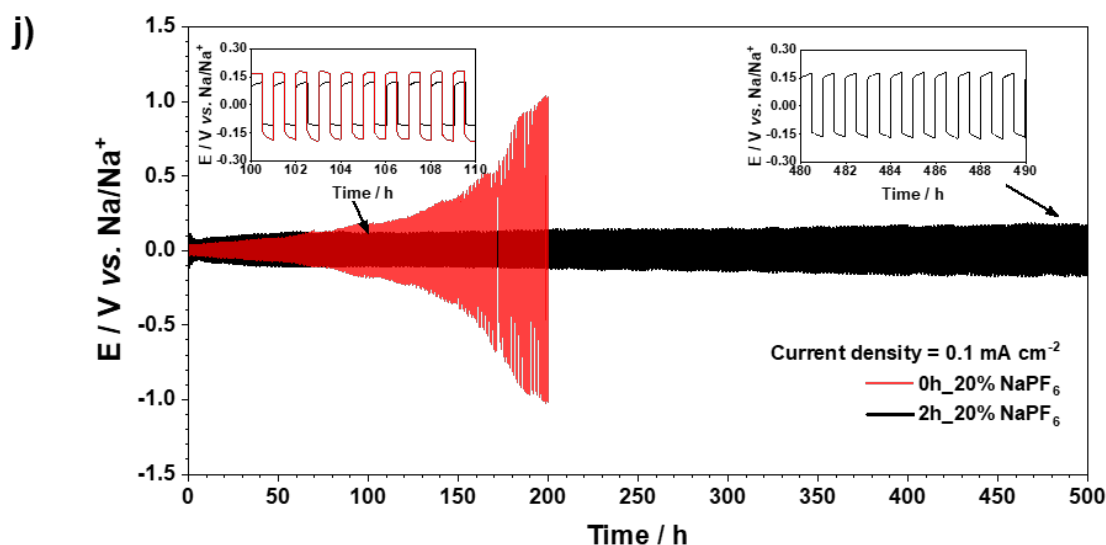


Figure 5. a) Evolution of the measured ionic conductivity of pure LATP SSEs during CSP as a function of the TLP content. b) Nyquist plot of the evolution of CSEs impedance depending on the polymer composition.

c) Effect of the LATP mean particle size on the ionic conductivity and activation energy of the CSEs. d) – f) Cross-section SEM images of the LFP|LATP interface in the bilayers. g) – i) EDX mapping of the characteristic elements of each layer. j) Cycling behavior of the 80%NZSP–20%NaPF₆ CSEs with different particle sizes in Na symmetric cells.

5. CONCLUSION

The Cold Sintering Process offers a promising route toward more sustainable ceramic manufacturing by drastically reducing the thermal requirements for densification. Its combination of pressure, moderate temperature and TLP phase enables ceramics, composites and multilayer architectures, which could be difficult to obtain by conventional sintering, to be processed. In solid-state batteries, CSP is particularly attractive because it allows the densification of ceramic electrolytes, the incorporation of polymeric or salt-based conductive phases and the fabrication of integrated electrode-electrolyte bilayers at temperatures compatible with thermally sensitive components. The results summarized here show that processing variables such as particle size, TLP chemistry, additives and pressure must be carefully optimized to balance densification, ionic transport and electrochemical stability. Therefore, CSP should be considered not only as a sustainable alternative to high-temperature sintering, but also as an advanced ceramic processing tool for the design of next-generation energy materials.

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