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Study of a glass-ceramic seal: porosity and sealing

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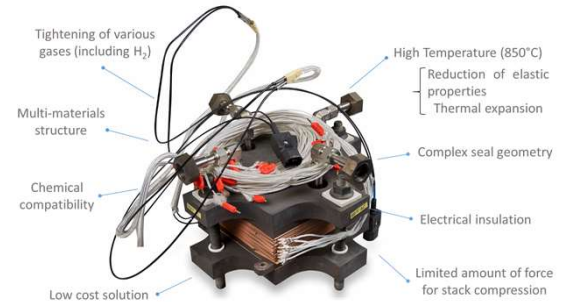
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CONTEXT & OBJECTIVES

Ensuring the sealing of complex structures such as Solid Oxide Cells stack (ceramic/metallic multilayer) is technically very challenging. Sealing is obtained by heat treatment of a suspension composed of glass powder, solvents and adjuvants, leading to a glass-ceramic.

Objective of this work :

Improving the global sealing performances by controlling the microstructure of the glass-ceramic.



SCIENTIFIC APPROACH

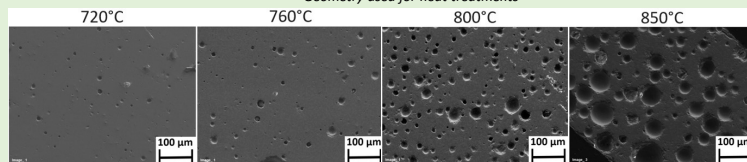
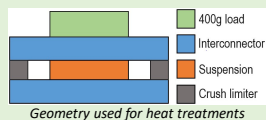
Material characterization & Sealing measurements

- 1 Understand the effect of heat treatment on the porosity of the glass-ceramic
- 2 Characterize the evolution of the porosity
- 3 Determine the sealing performance

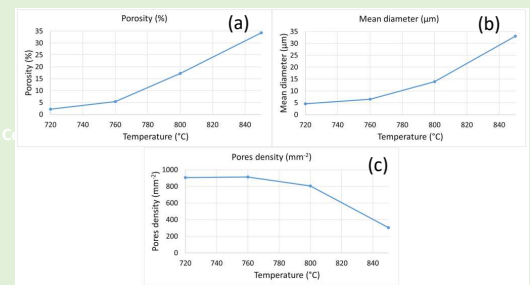
The reduction of the material **porosity** is a first parameter that can be used to improve the **sealing** material properties. In fact, large and interconnected porosity can create a permanent leakage path. In addition, a porous material is less mechanically resistant than a dense material [1, 2]. Therefore, cracks can appear and create preferential leak paths.

STUDY OF THE POROSITY

Various heat treatments are realized on the suspension to determine its effect on the glass-ceramic porosity. Gas analysis have shown that gases present in the porosity are composed of 60 % vol. of CO_{2(g)} which comes from the decomposition of organic solvents and 40 % vol. of N_{2(g)} or Ar_(g) coming from the furnace atmosphere. Surface porosity quantification is realized by SEM pictures analysis using thresholding method on polished section of glass-ceramic.



SEM images of glass-ceramic seals formed by a 10h treatment at different temperatures between two plates of interconnects. Polished sections of the core of the sample.



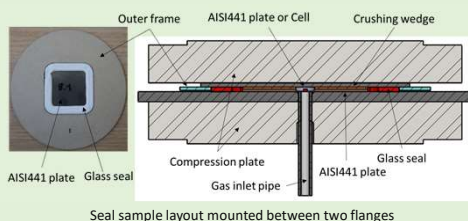
Detailed characterization of the glass-ceramic porosity formed by a 10h heat treatment at different temperatures. % Surf. porosity (a), pores mean diameter (b), pores density (c)

➤ The porosity significantly increases with temperature. Mean diameter increases because of two combined phenomena. Gas molar volume increases and glass viscosity decreases with temperature.

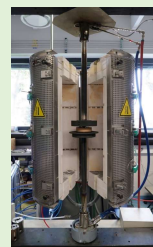
➤ Coalescence is observed at high temperature, so mean diameter increases and pores number decreases.

SEALING MEASUREMENTS

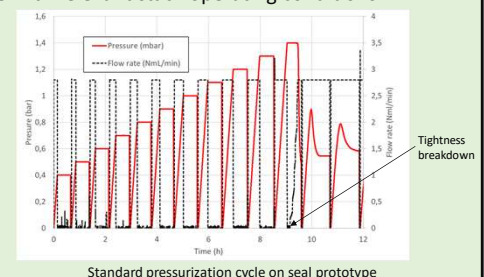
Technical qualification of sealing solutions requires the definition of a **standard test protocol** compatible with relevant stack operating conditions.



Seal sample layout mounted between two flanges



Sample mounted on a press



Standard pressurization cycle on seal prototype

This protocol, with the application of cyclic increasing loads on standard seals is rather discriminant. This test allows also quantitative analysis of the seals stiffness related to their geometries (width and height).

PERSPECTIVES

Combining a program of analysis on the glass-ceramic and standardized mechanical test protocol, a R&D program is under way to optimize the stack fabrication process and performances. Evolution of porosity with time and temperature will be further characterized and modelled. The influence of the glass-ceramic seal microstructure on the sealing properties will be determined.

[1] D. Liu, B. Savija, G. E. Smith, P. E. J. Flewitt, T. Lowe, and E. Schlengen., *Int. J. Fract.*, vol. 205, 57-72 (2017).

[2] A. Perkins, W. Yang, Y. Liu, L. Chen, and C. Yenusah., *ASME International Mechanical Engineering Congress and Exposition* (2020)

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