

Analysis of the luminescent emission during the sharp shrinkage of ceramic compacts in electric-field-assisted sintering

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Introdução

Experimental

Resultados e Discussão

Conclusões

Agradecimentos

Sintering

several routes for sintering a ceramic green body

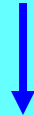
1. conventional sintering: heating up to T_s for a time t_s and cooling down to RT, $\uparrow\downarrow \Delta T / \Delta t$.

(1 to 10 degree/min)

2. fast firing: $\uparrow\downarrow \Delta T / \Delta t$ are relatively very large

(10^3 to 10^4 degree/min)

3. two-step sintering: heating to $\sim 70-80\%TD$ + heating at a lower temperature for longer times.



high density without grain growth

I. Wei Chen, X-H. Wang, Nature 404 (2000) 168.

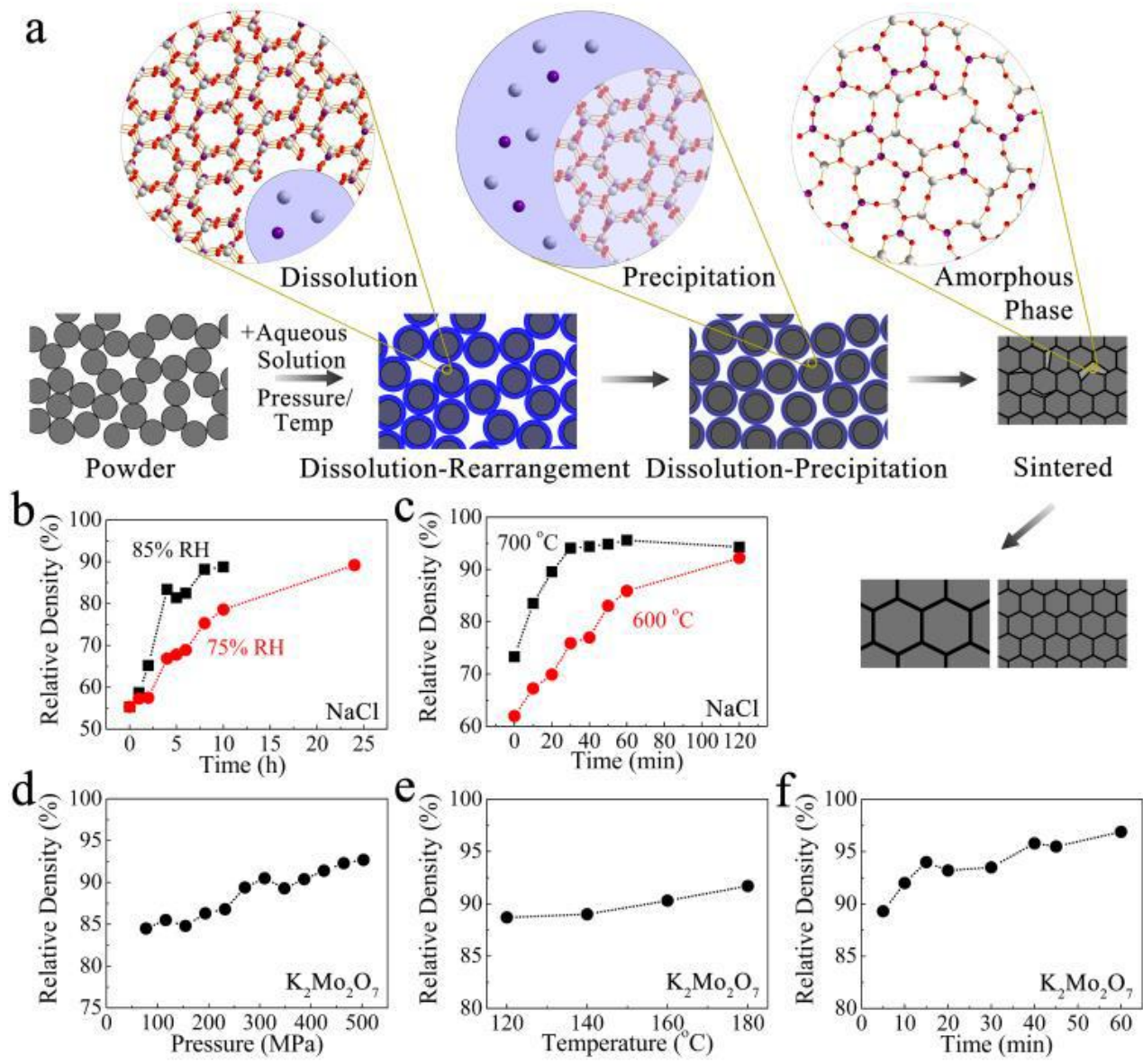
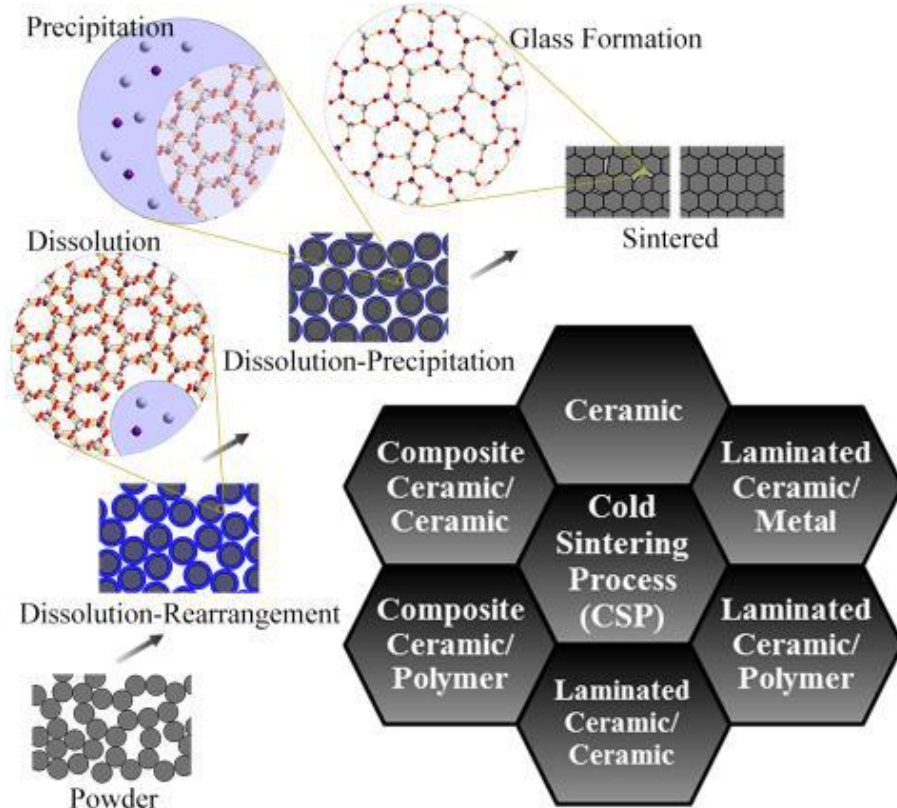
Introdução

Sinterização – **novos métodos**

- assistida por campo elétrico (*electric field assisted – flash sintering*) - **2010**
- a frio (*cold sintering*) - **2016**
- com luz negra (*black light sintering*) - **2020**
- aquecimento ultra rápido (*ultra-fast high temperature sintering*) - **2022**

Introdução

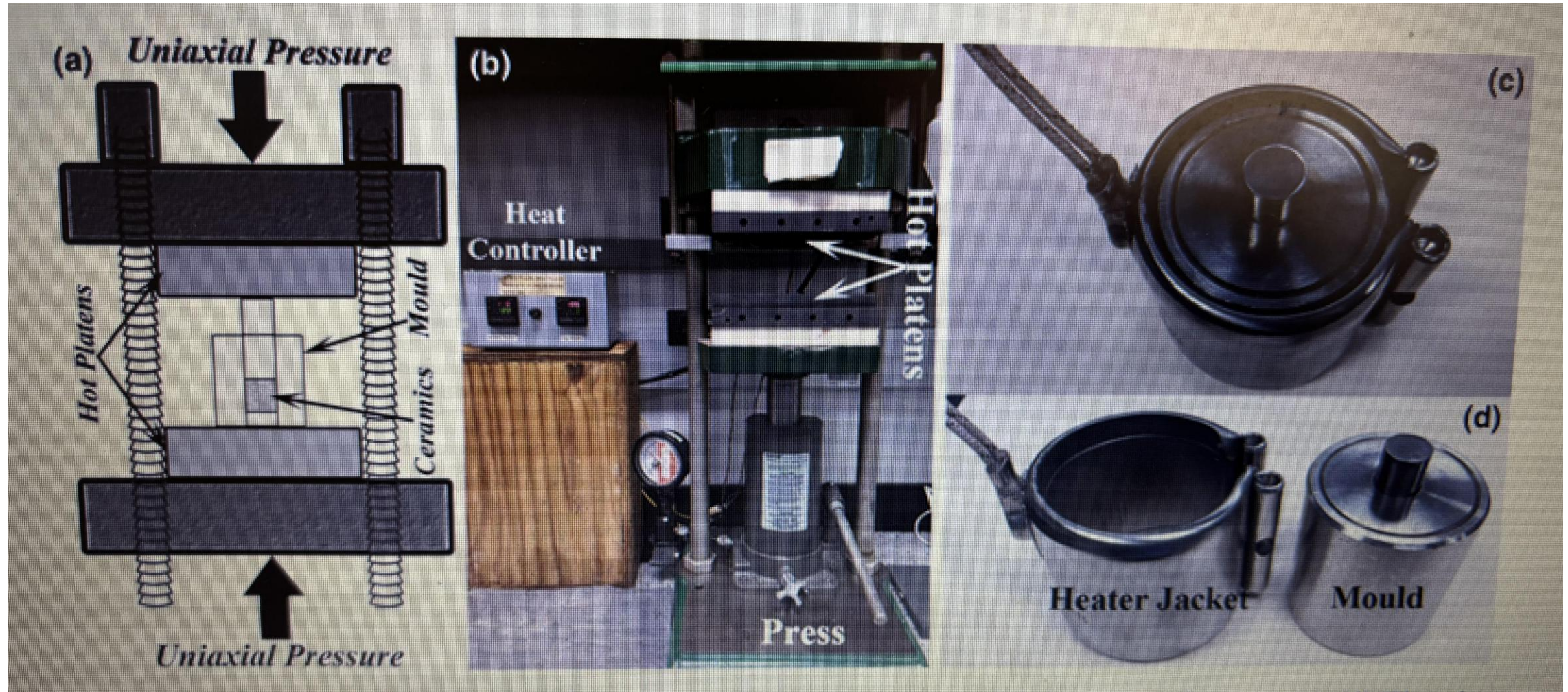
Sinterização a frio (*cold sintering*)



J. Guo, H. Guo, A.L. Baker, M.I. Lanagan, E.R. Kupp, G.L. Messing, C.A. Randall, Cold Sintering: A Paradigm Shift for Processing and Integration of Ceramics

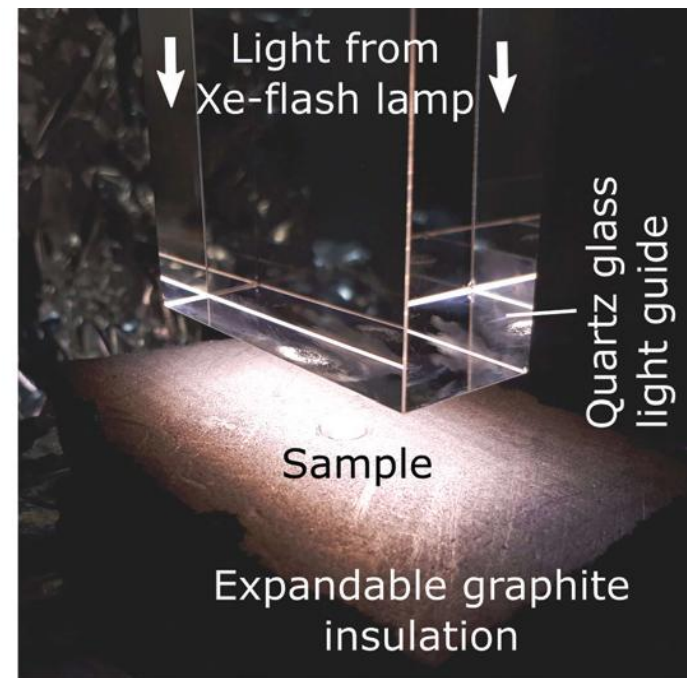
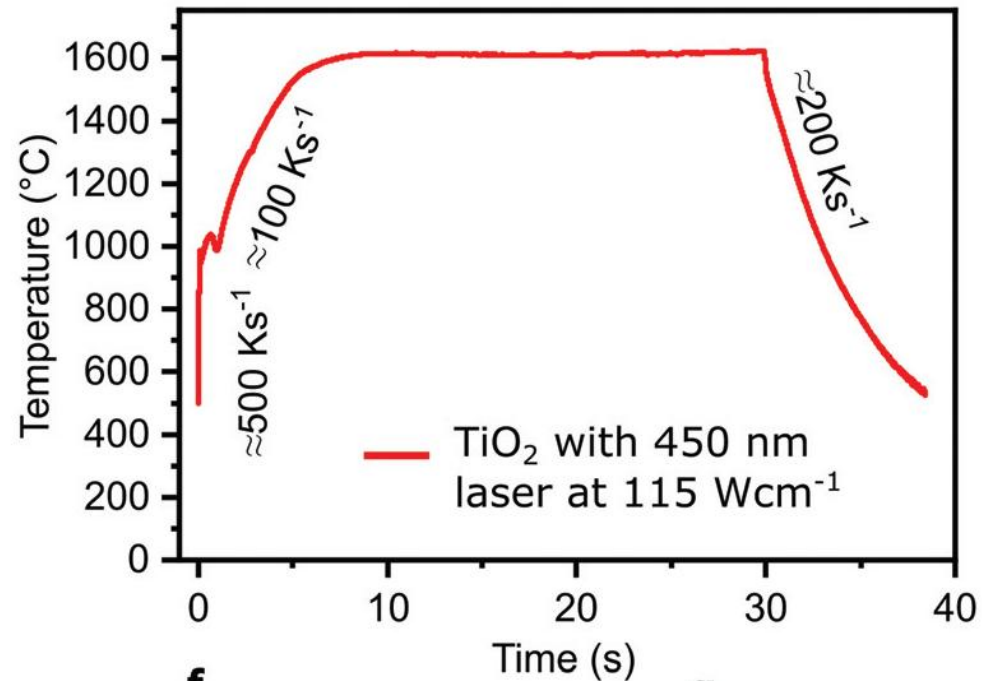
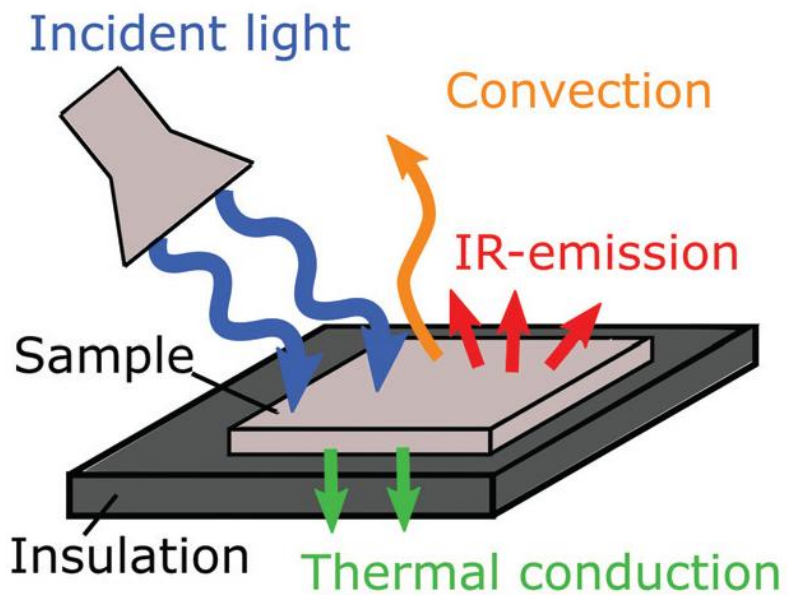
Introdução

a frio
(cold sintering)



H. Guo, A. Baker, J. Guo, C.A. Randall, "Cold Sintering Process: A Novel Technique for Low-Temperature Ceramic Processing of Ferroelectrics", J. Am. Ceram. Soc. 1–4 (2016) DOI: 10.1111/jace.14593

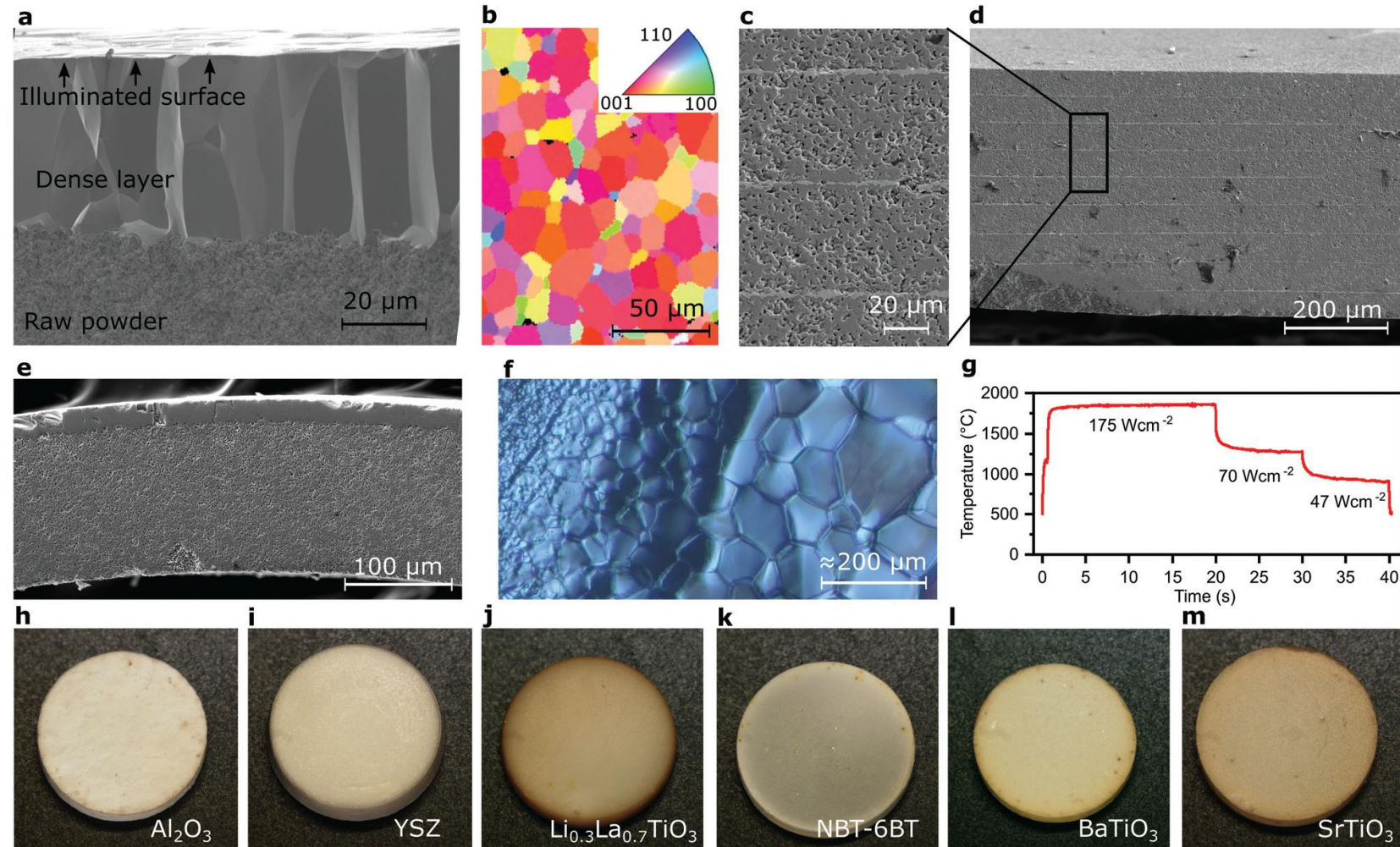
com luz negra - (*black light sintering*)



Blacklight sintering of ceramics, Mater. Horiz., 2022, 9, 1717–1726

Introdução

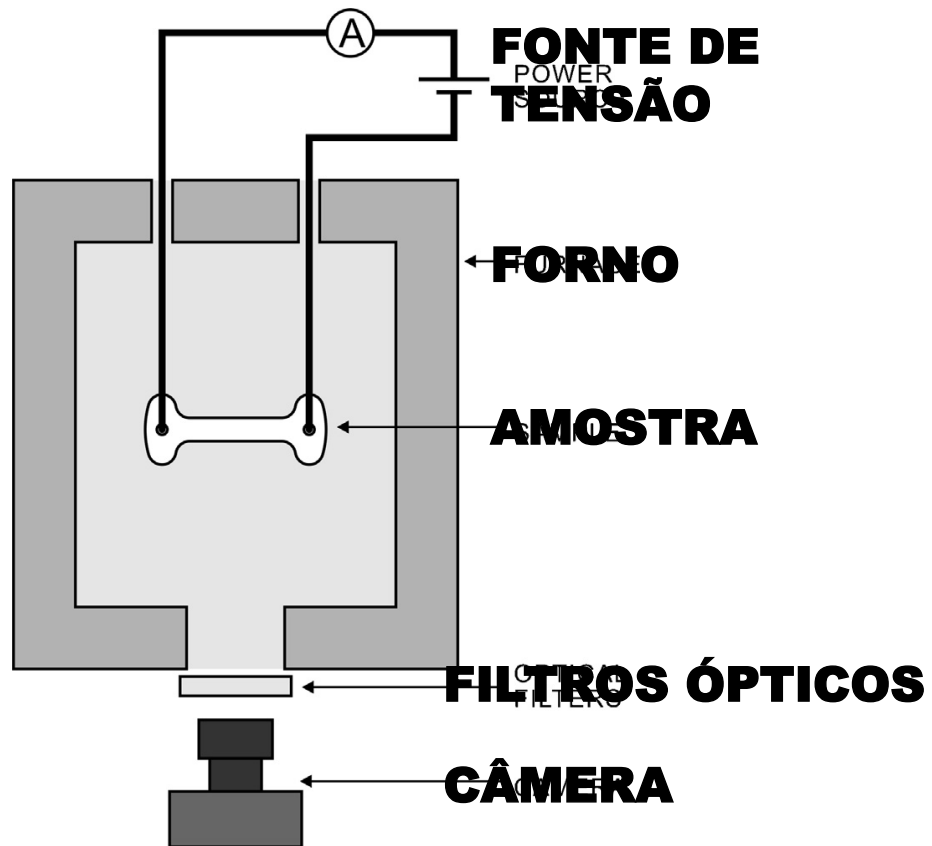
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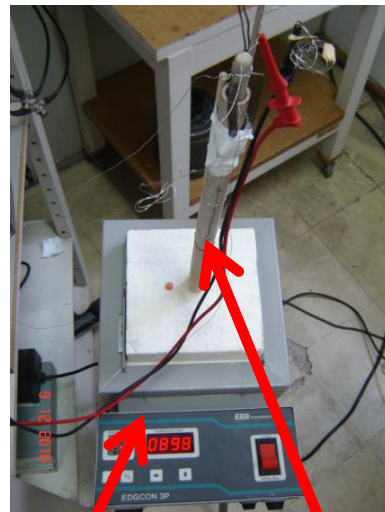
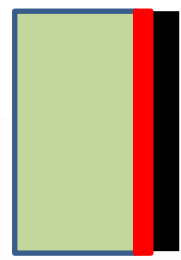
Introdução

assistida por campo elétrico (*electric field assisted - flash-sintering*)

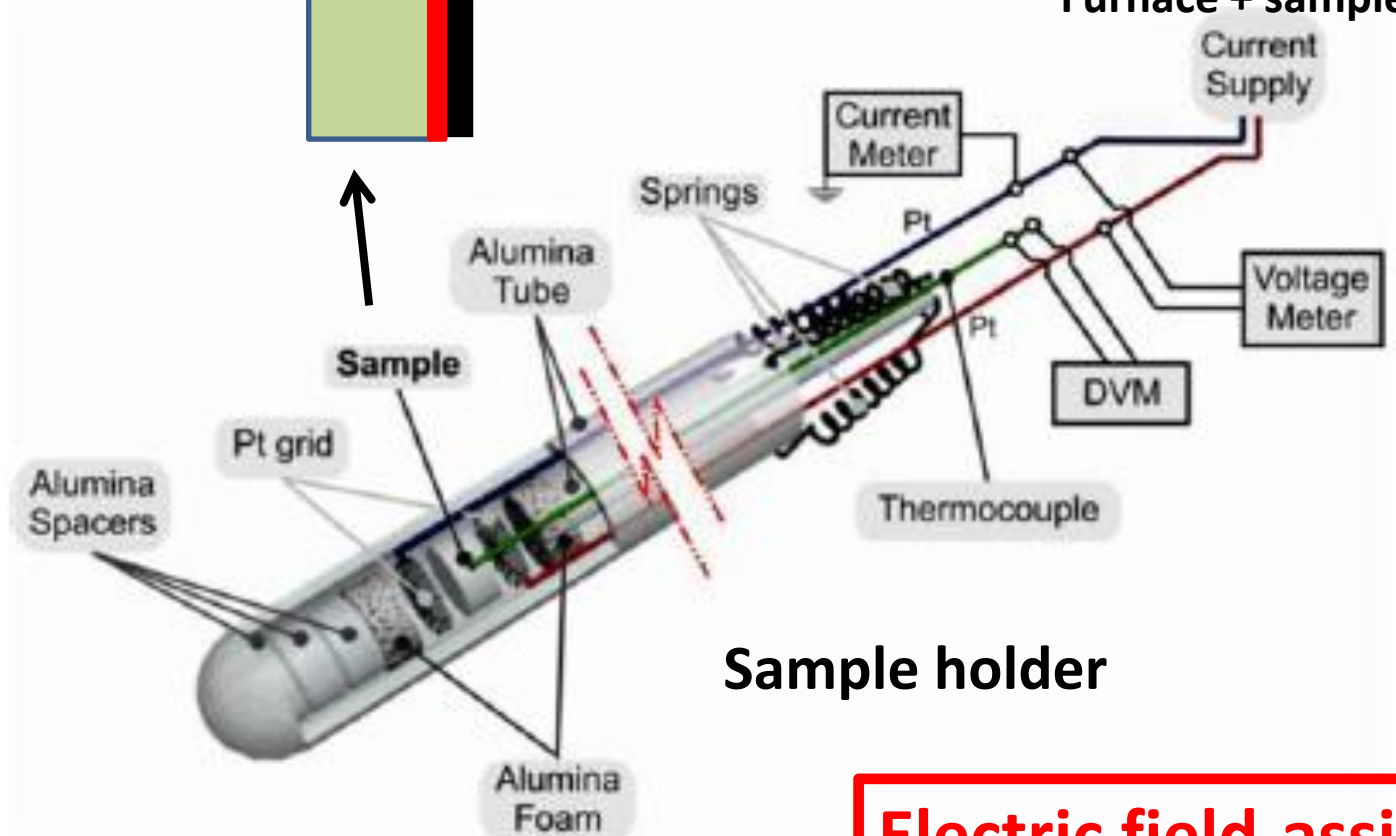


Introdução

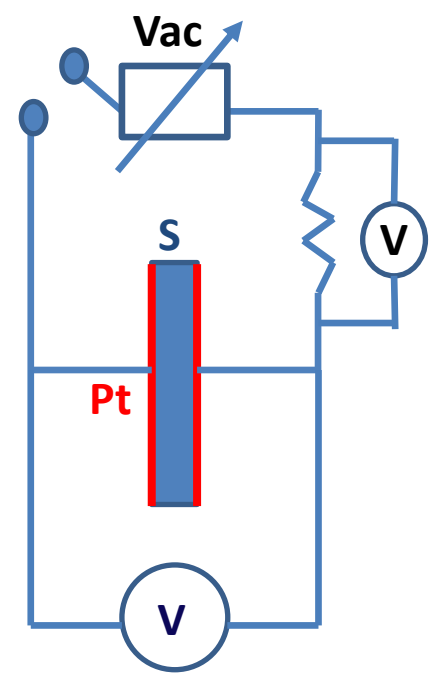
8YSZ+NiO 8YSZ LSM



Furnace + sample holder + ac power supply

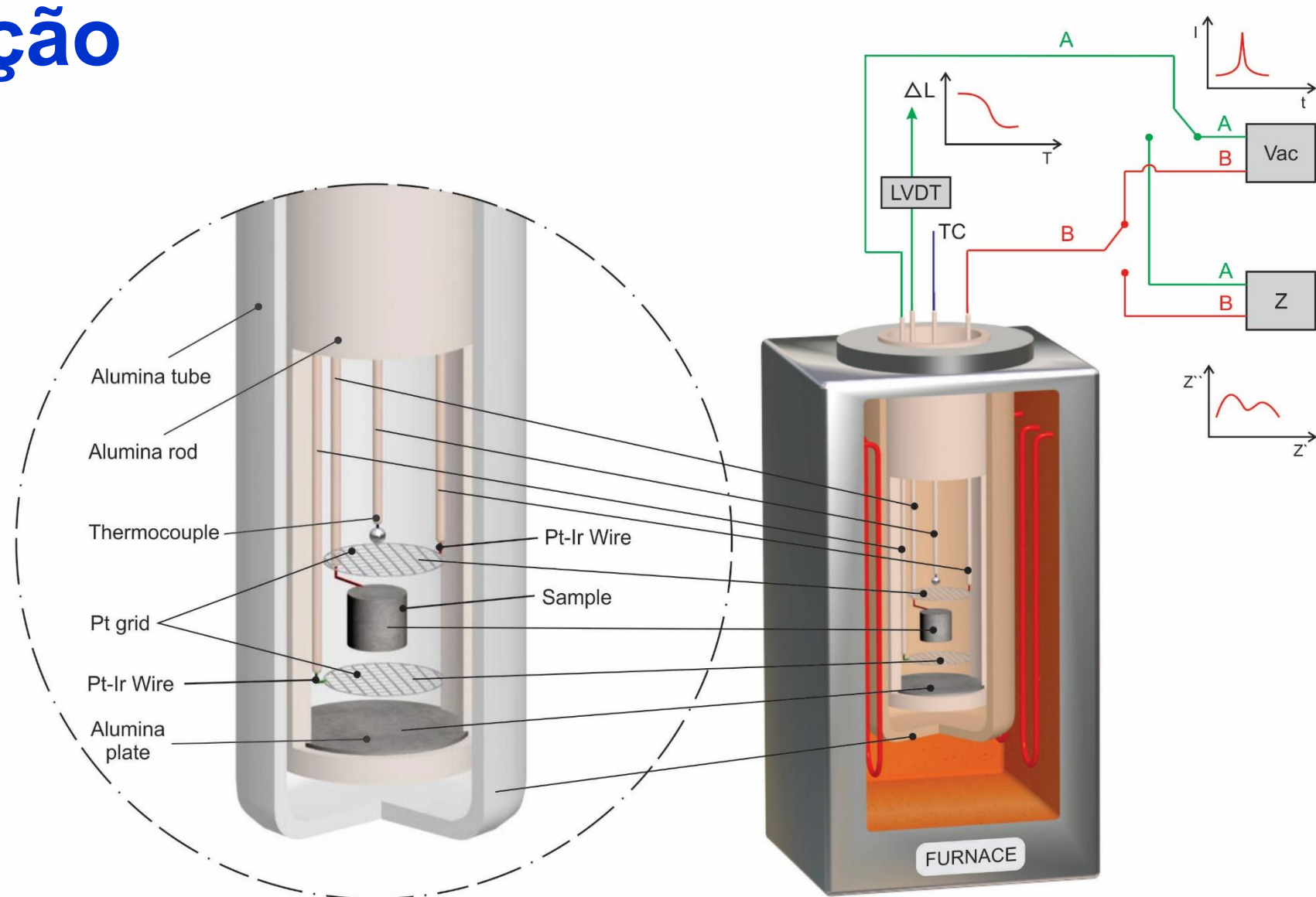


Sample holder



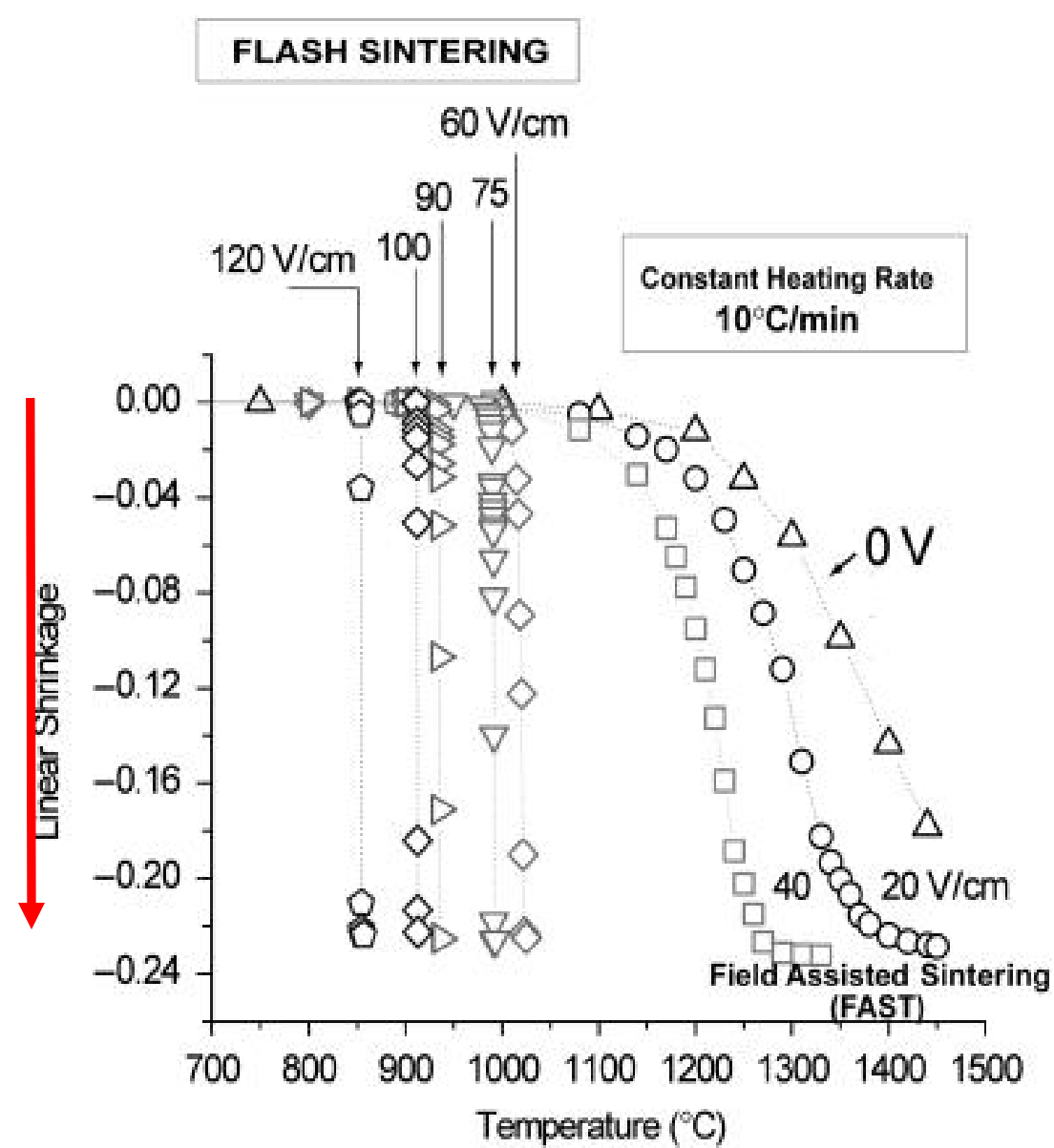
Electric field-assisted sintering

Introdução



Scheme of the sample positioning inside the dilatometer furnace

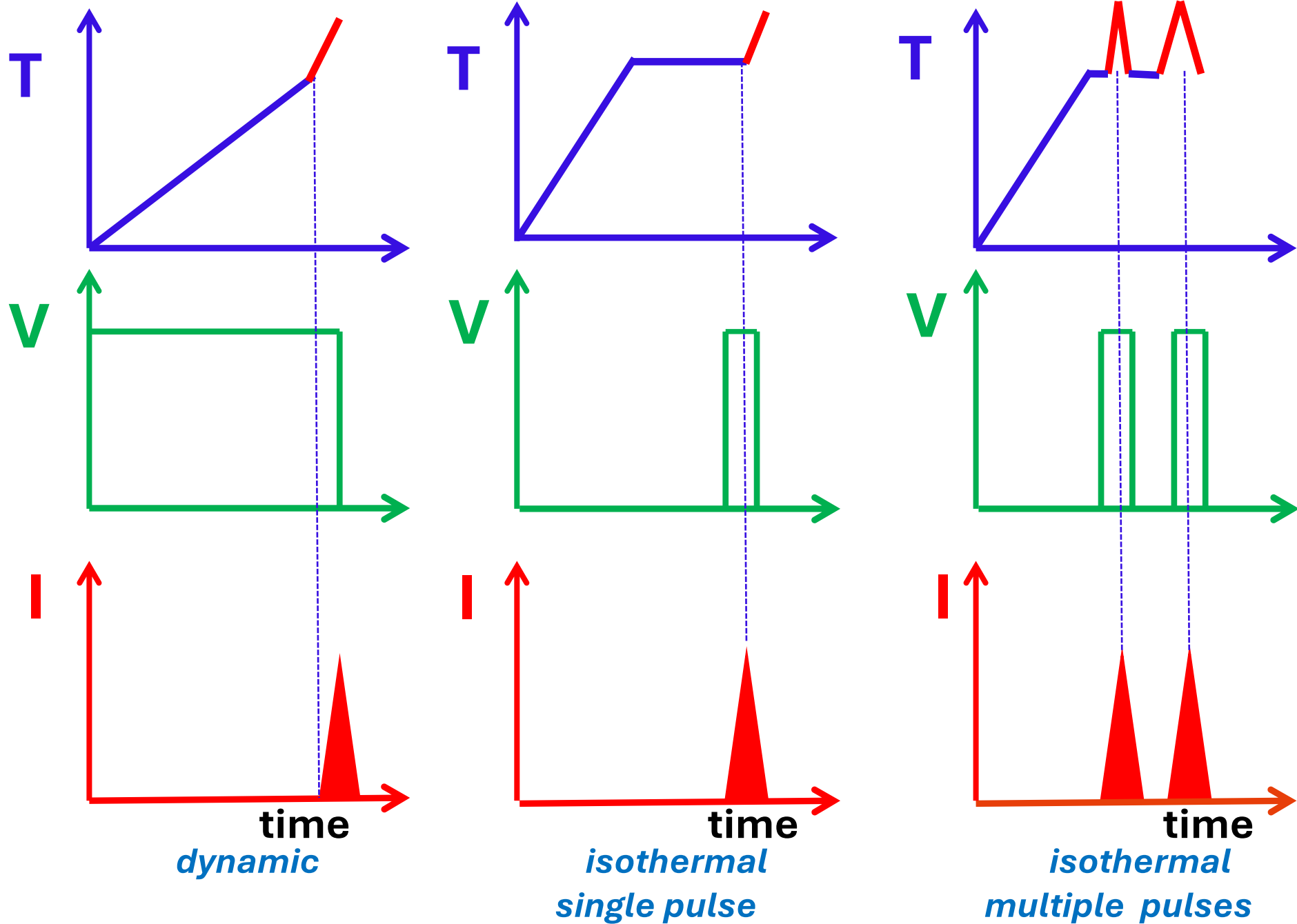
Introdução



S. Ghosh, A. H. Chokshi, P. Lee, R. Raj, A huge effect of weak dc electrical fields on grain growth in zirconia, J. Am. Ceram. Soc. 92 (2009) 1856-9.

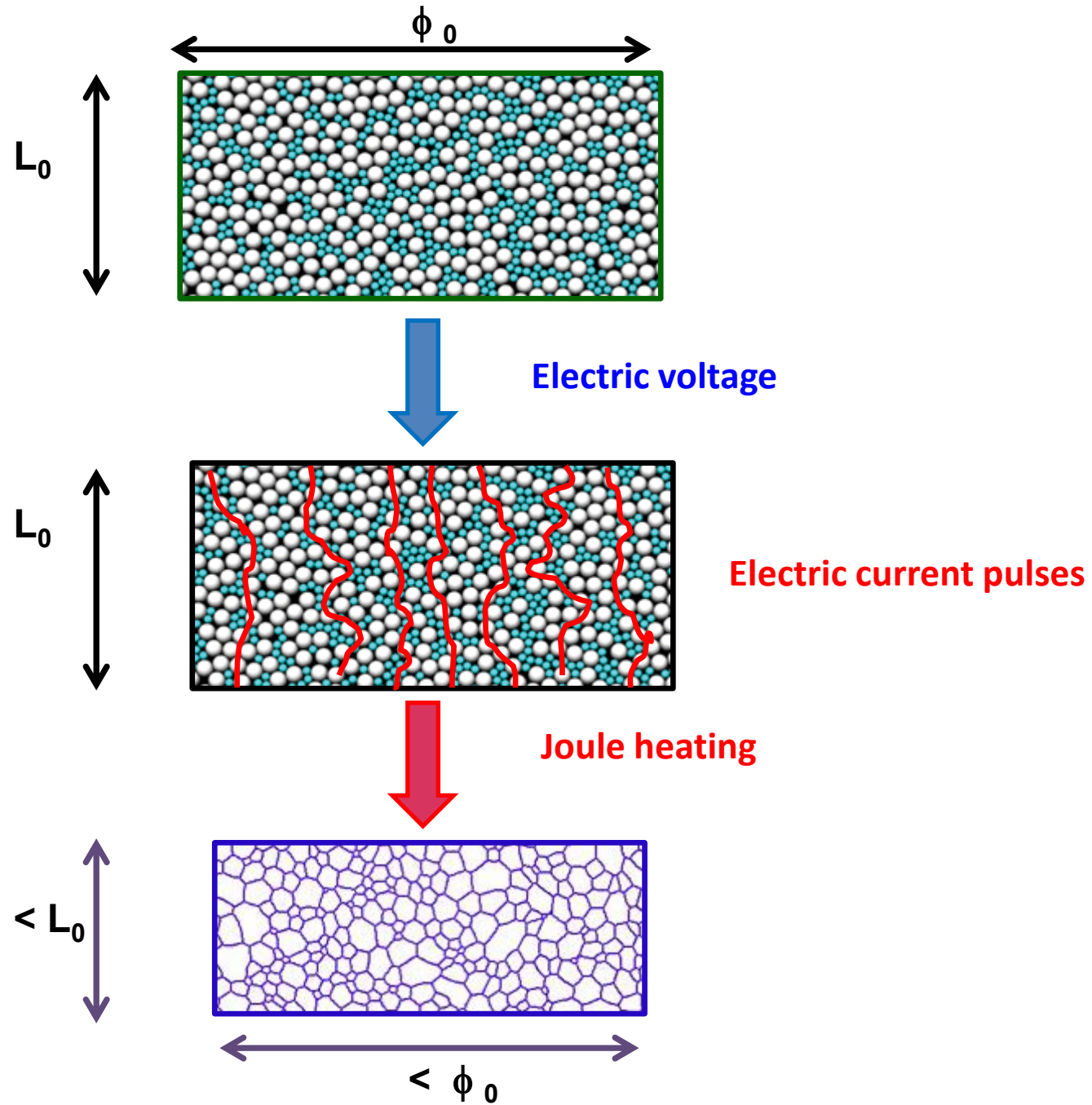
M. Cologna, B. Rashkova, R. Raj, Flash Sintering of Nanograin Zirconia in < 5 s at 850 degrees C, J. Am. Ceram. Soc. 93 (2010) 3556-3559.

electric field-assisted sintering

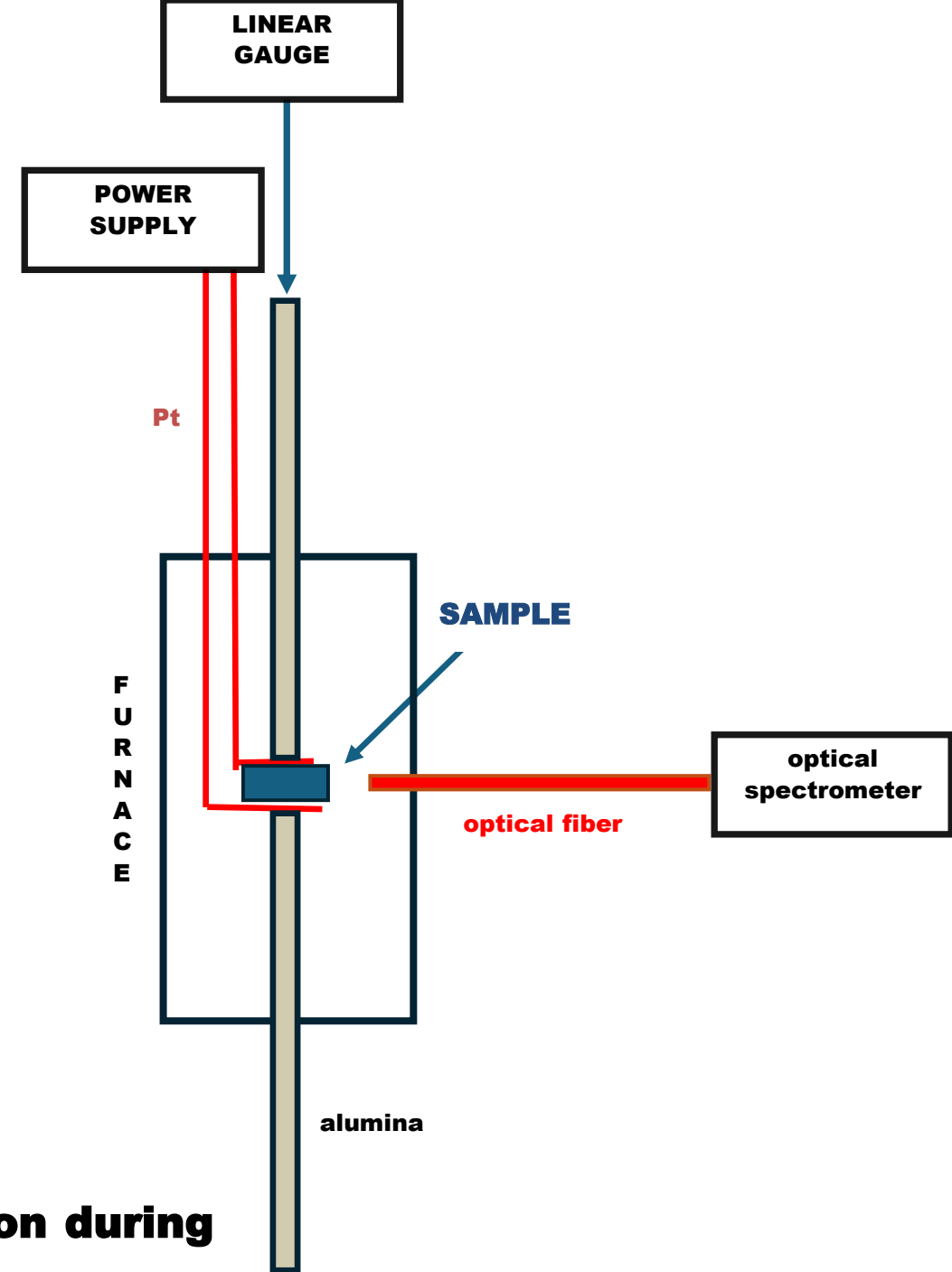


Introdução

electric field-assisted sintering



Experimental

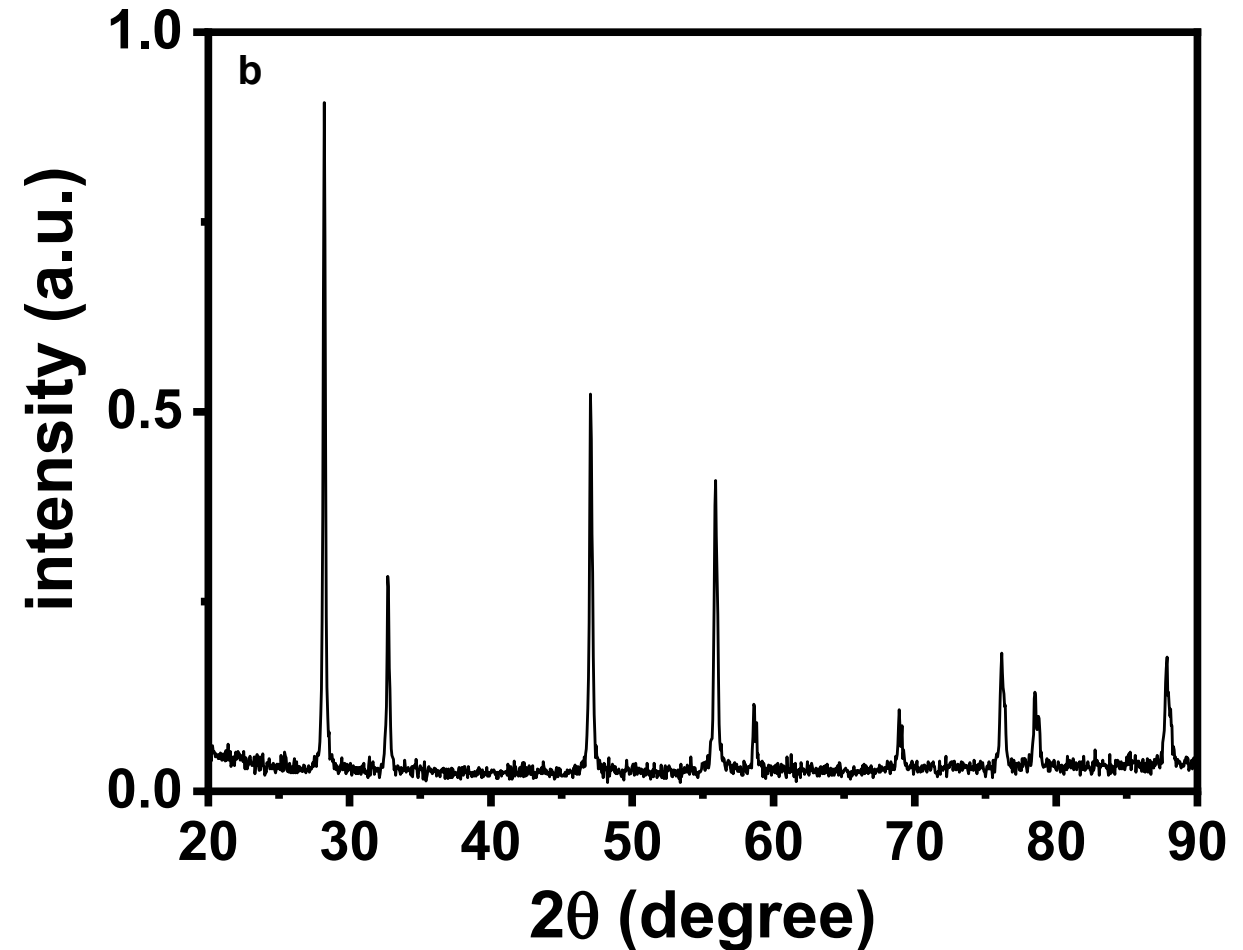
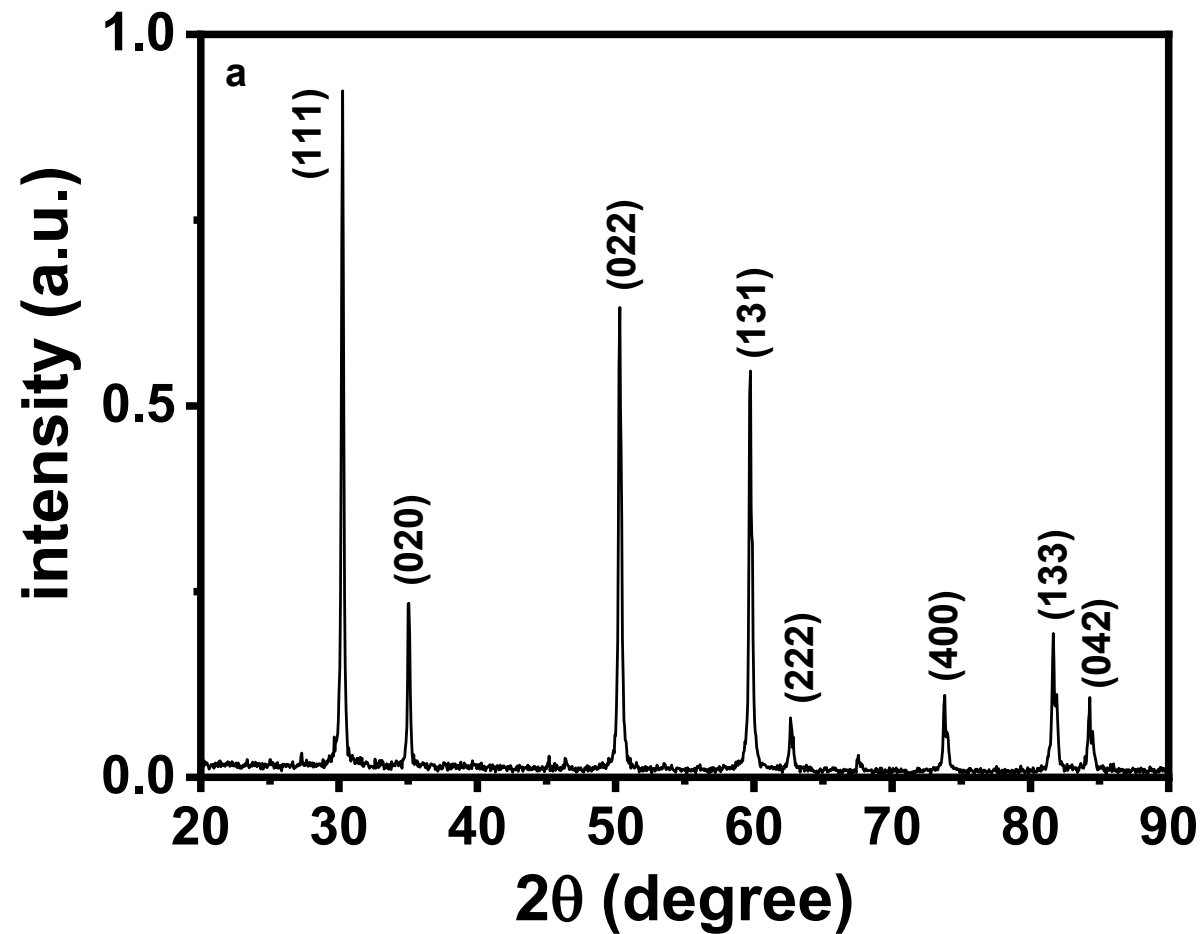


Experimental setup for measuring luminescent emission during electric field-assisted sintering.

Experimental



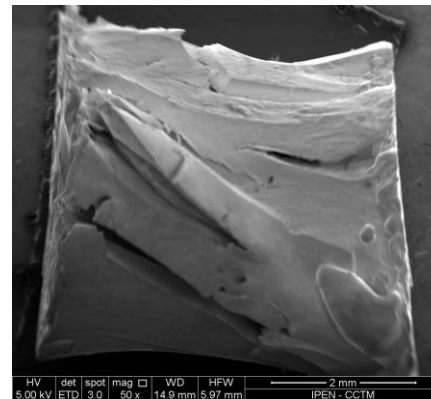
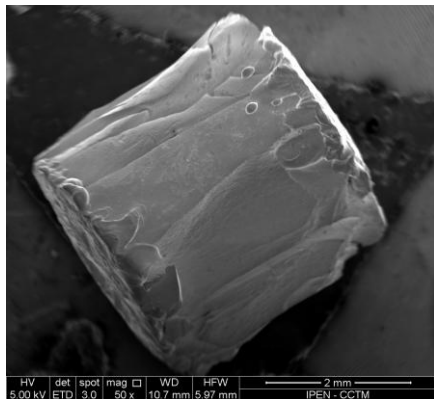
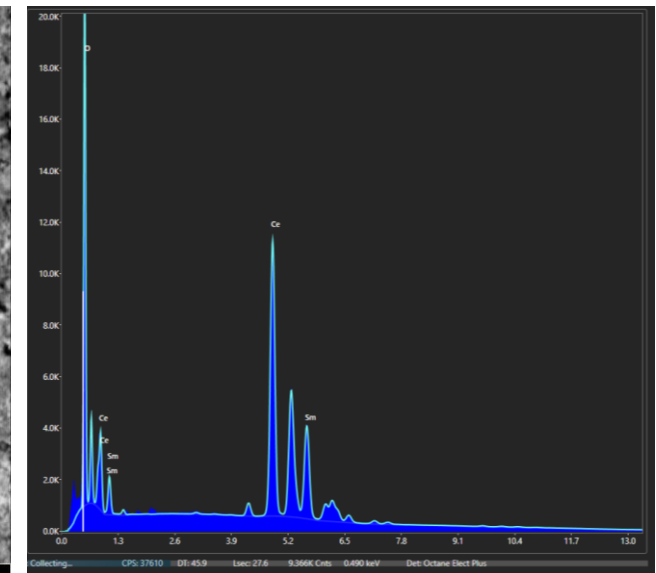
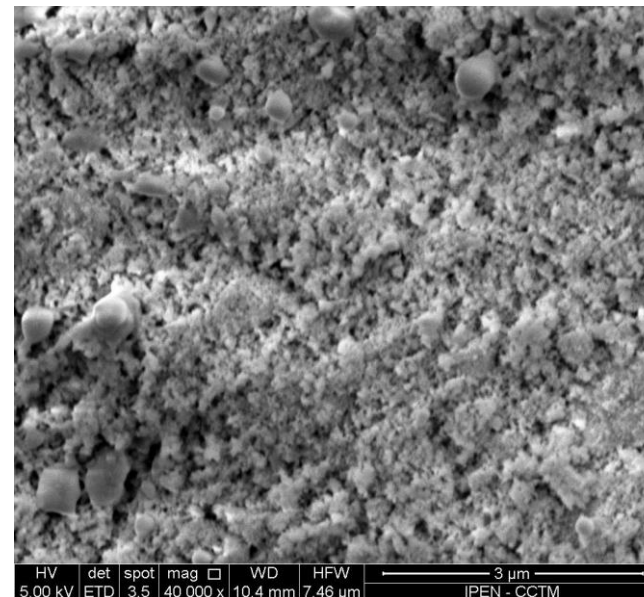
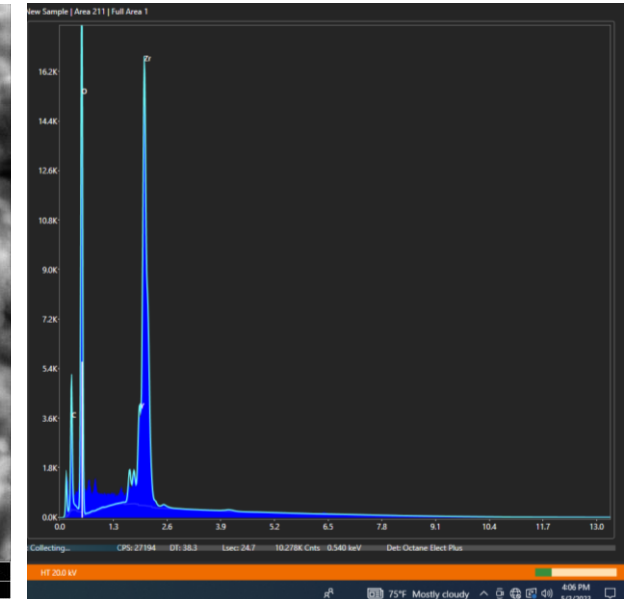
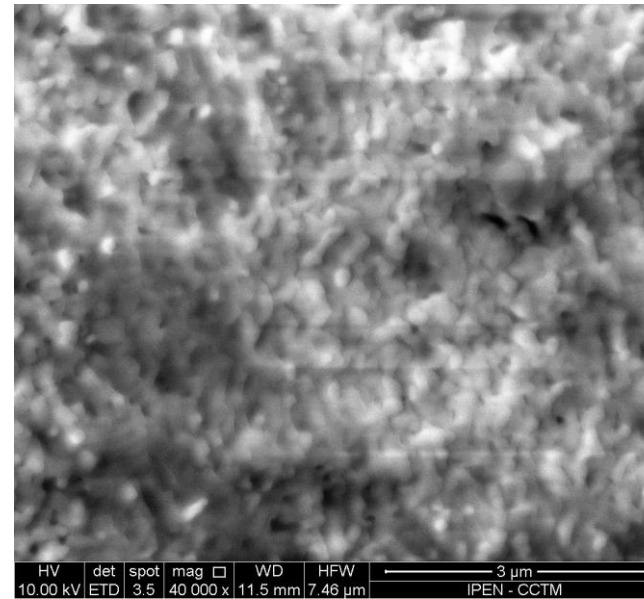
Resultados



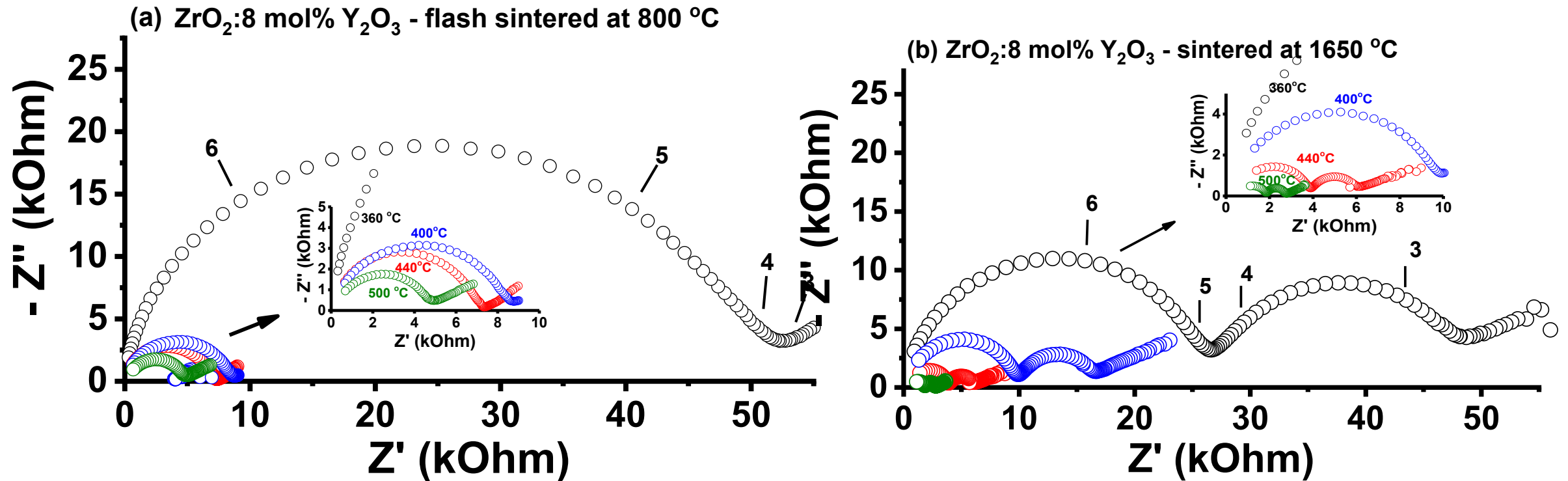
X-ray diffraction patterns of (a) ZrO₂: 8 mol% Y₂O₃ (8YSZ) and (b) CeO₂: 20 mol% Sm₂O₃ (20SDC) flash sintered ceramic pellets.

Resultados

Scanning electron microscopy micrographs and EDS plots of ZrO_2 : 8 mol% Y_2O_3 (top) and CeO_2 : 20 mol% Sm_2O_3 (bottom) flash sintered ceramic pellets

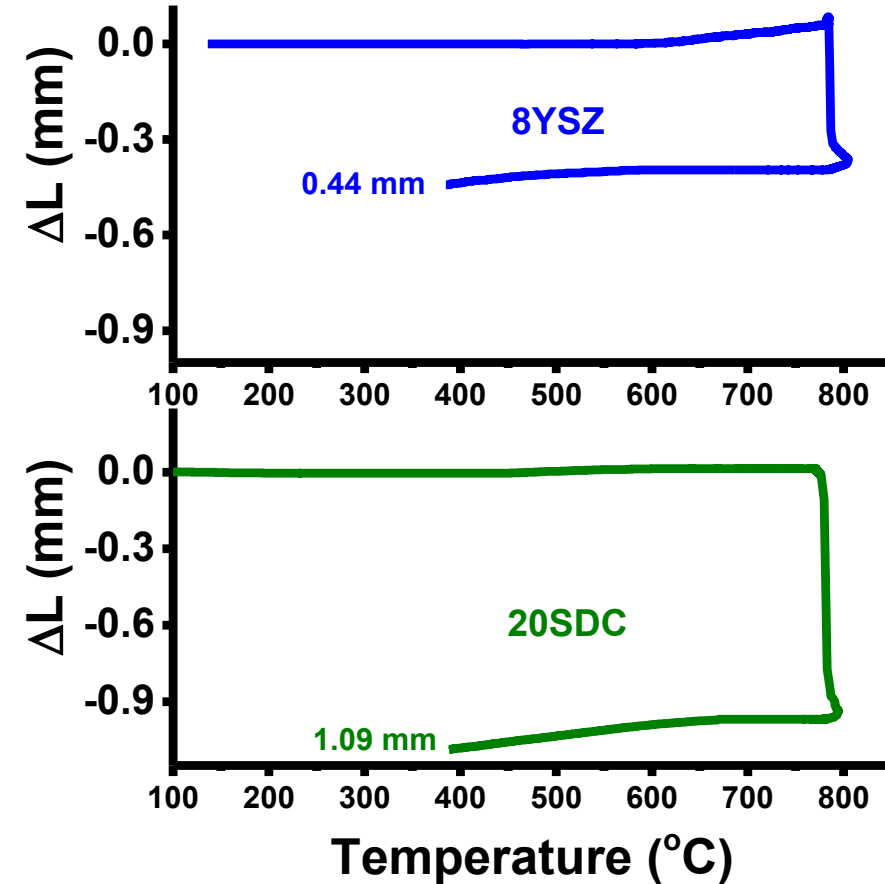
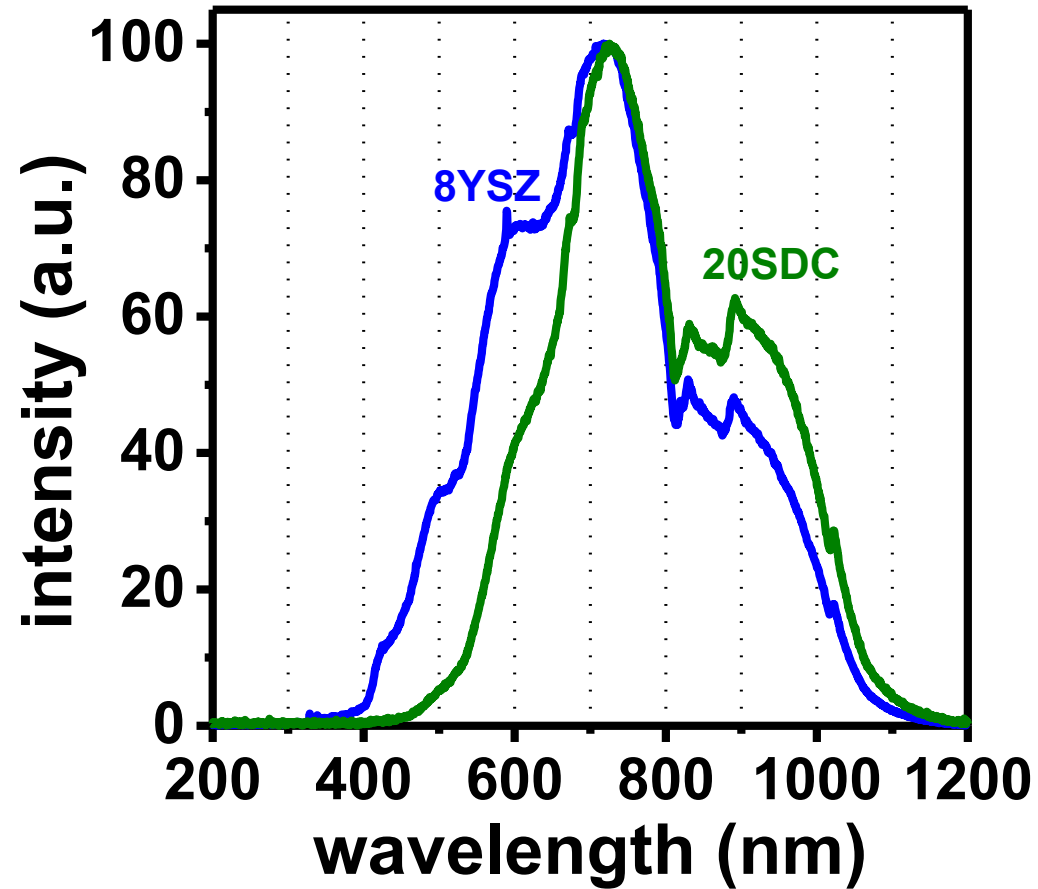


Resultados



Impedance spectroscopy diagrams of (a) $\text{ZrO}_2: 8 \text{ mol\% Y}_2\text{O}_3$ (8YSZ) ceramic pellets flash sintered at 800 °C and (b) sintered at 1650 °C/4 h. Inset: exploded view of diagrams measured at high temperatures; numbers stand for log f (f: Hz).

Resultados



Left: spectrometric optical data in the 200 - 1200 nm range of the luminescent emission detected at 800 $^{\circ}\text{C}$ during flash sintering ZrO_2 : 8 mol% Y_2O_3 (8YSZ) and CeO_2 : 20 mol% Sm_2O_3 ; (20SDC).

Right: thickness monitoring of both ceramic pellets with the digital linear gauge.

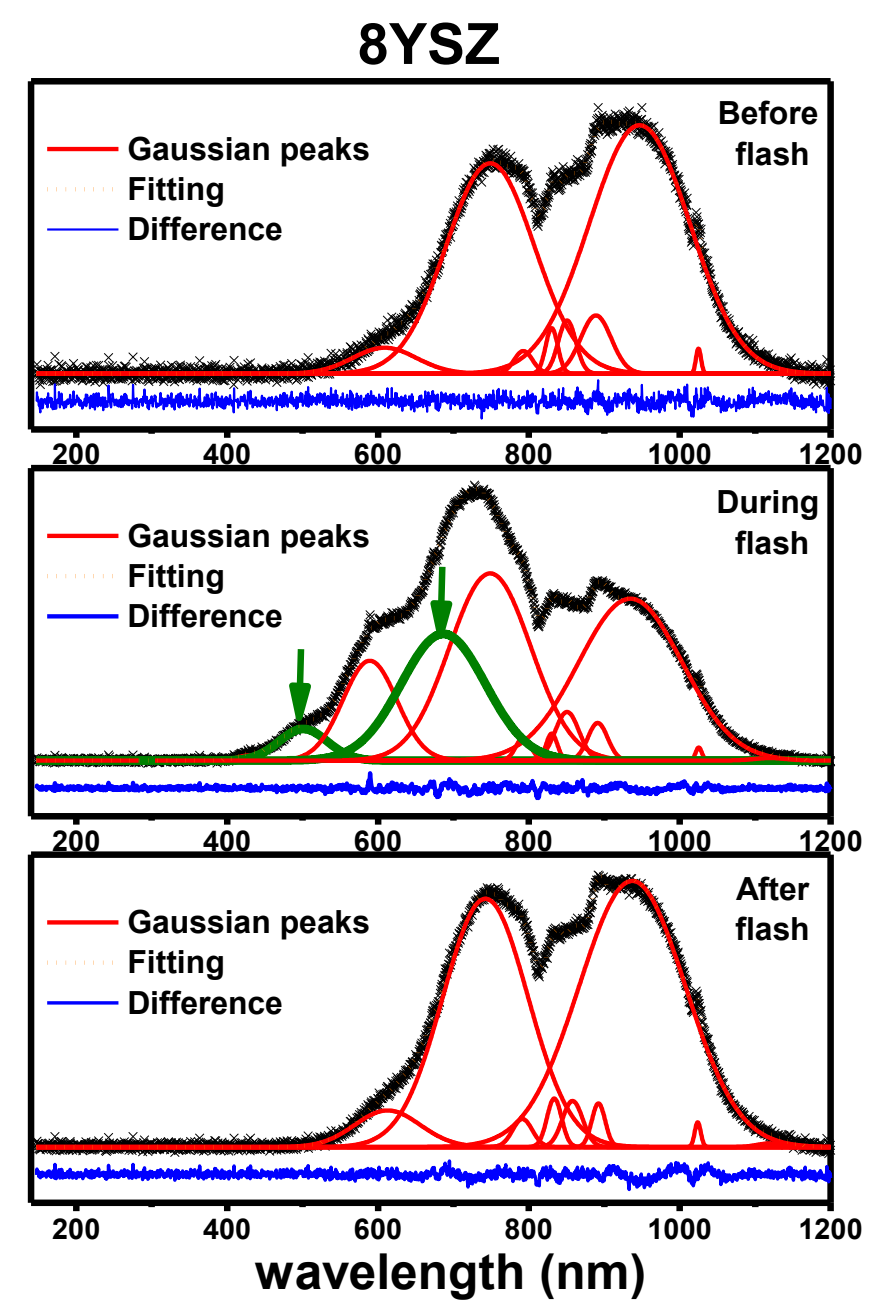
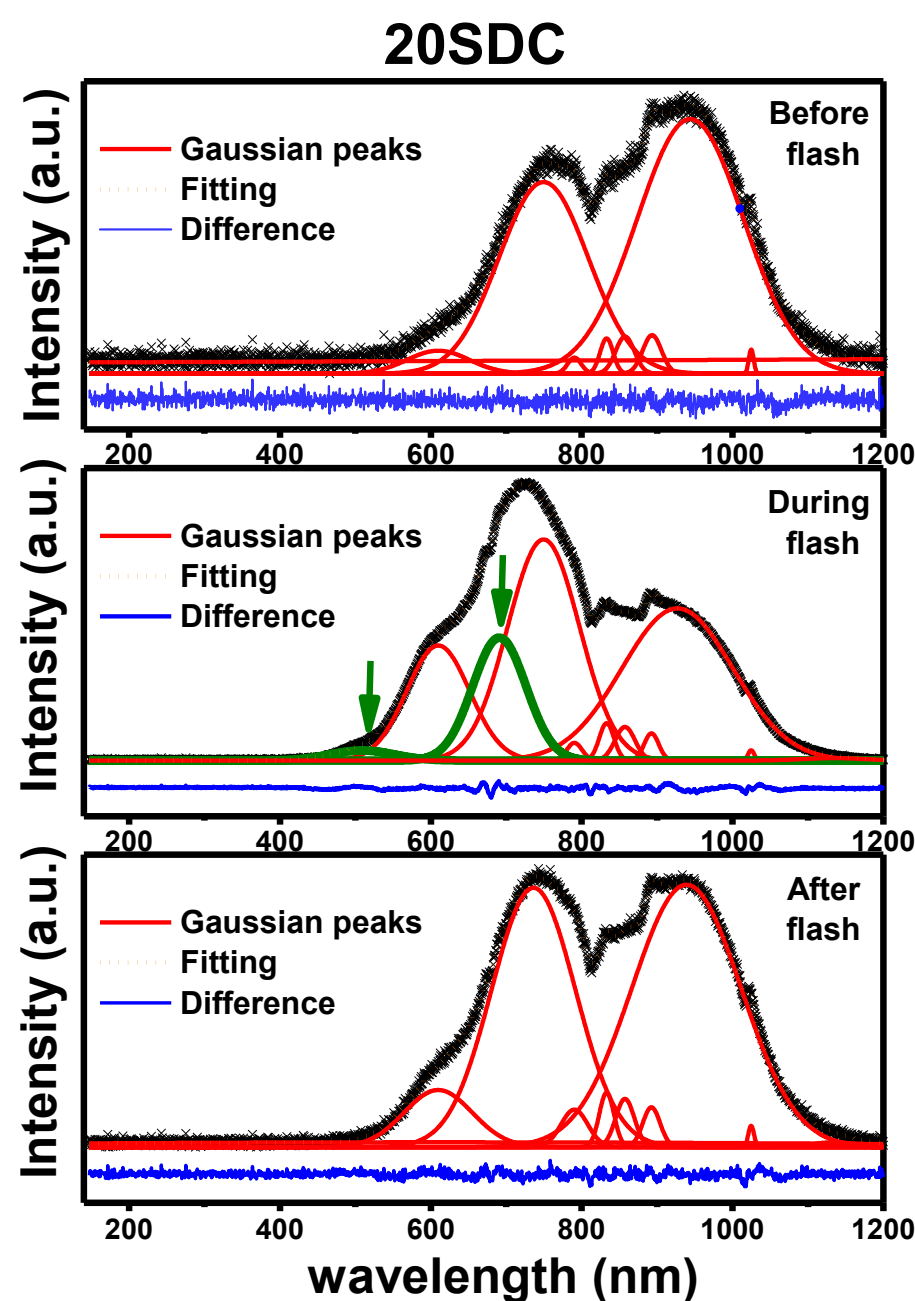
Resultados

Optical spectrometric data of the luminescent emission detected at 800 °C before (top), during (center) and after (bottom) shrinkage surge under flash sintering CeO_2 : 20 mol% Sm_2O_3 (20SDC) and ZrO_2 : 8 mol% Y_2O_3 (8YSZ).

Red lines: deconvolution bands.

Blue lines: deviation of the sum of the deconvoluted and the experimental spectra.

Green lines: extra bands.



Resultados

Values of wavelengths at the peak amplitude of the Gaussian bands obtained by deconvolution of the luminescent spectra collected before, during, and after the occurrence of the flash event during flash sintering CeO_2 : 20 mol% Sm_2O_3 and ZrO_2 : 8 mol% Y_2O_3 ; bold red types denote extra band maxima.

Sample, λ_{max} (nm)
20SDC before flash 610, 750, 940
20SDC during flash 517, 610, 690, 750, 940
20SDC after flash, 610, 750, 940
8YSZ before flash, 610, 750, 940
8YSZ during flash 500, 610, 680, 750, 940
8YSZ after flash, 610, 750, 940

Conclusões

1- Durante o processo de sinterização assistida por campo elétrico em cerâmicas de ZrO_2 : 8 mol% Y_2O_3 e CeO_2 : 20 mol% Sm_2O_3 é detectada luminescência pelo aquecimento Joule nos contornos de grão.

2- O espectro de emissão luminescente permitiu detectar bandas ópticas de emissão devidas exclusivamente às amostras cerâmicas.

3. Um mecanismo é proposto para a ocorrência das bandas atribuídas às cerâmicas: interação dos portadores de carga da corrente elétrica que percola os contornos de grão com espécies químicas ali localizadas.

Agradecimentos

FAPESP 

CAPES 

CDMF 


Conselho Nacional de Desenvolvimento Científico e Tecnológico

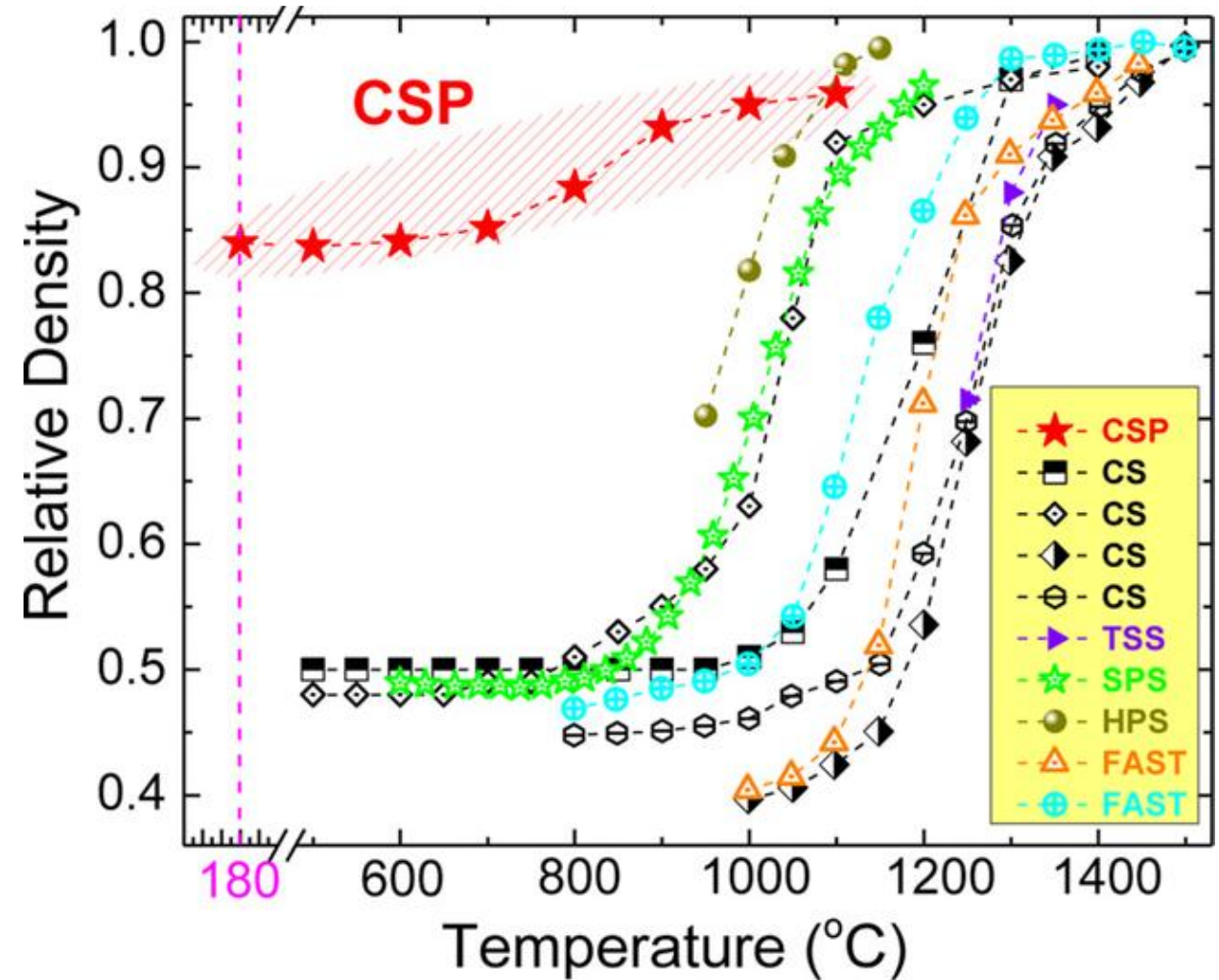
CNPq


Comissão Nacional de Energia Nuclear

CNEN

Introdução

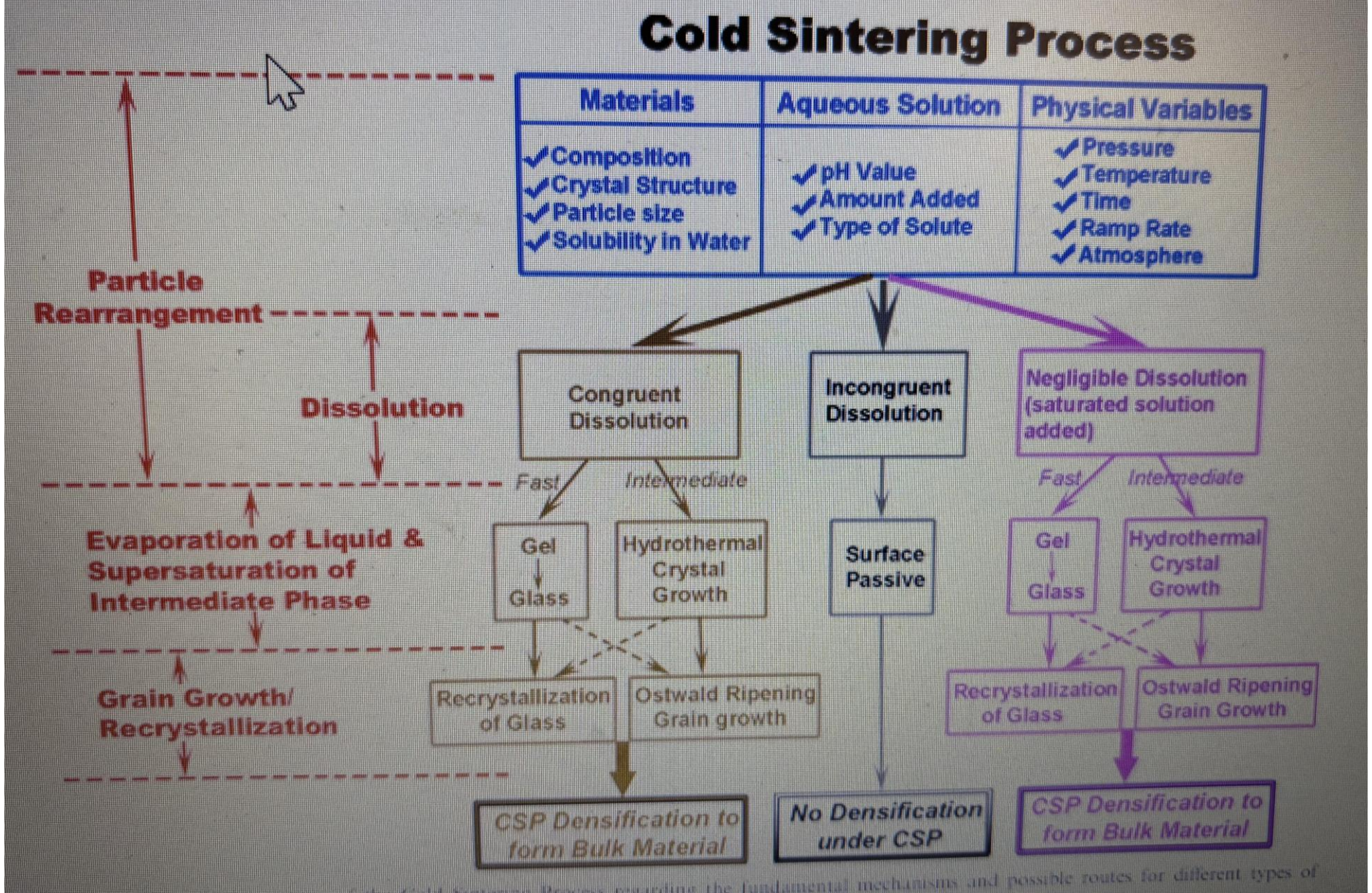
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