



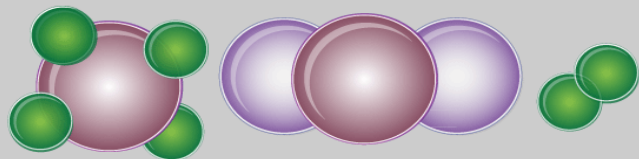
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Mechanical properties of $\text{La}_2\text{NiO}_{4+\delta}$ membranes for gas separation

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Outline

- **Introduction**
- **Material**
- **Water based tape casting**
- **Mechanical properties**
- **Summary**

Introduction / *oxygen production*

Cryogenic Distillation: Proven Oxygen Supply Technology

- Mature, reliable technology
- Energy consumption intensive
- complicated process

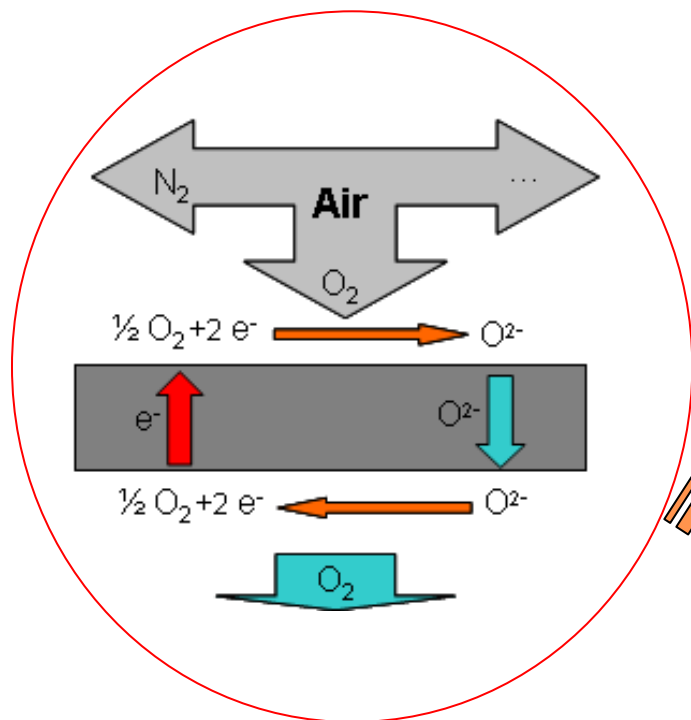


Pressure swing adsorption: Convenient Oxygen Supply Technology

- Mature, flexible technology
- Low oxygen purity



Introduction / MIEC membranes



Gas sensor

Cathode for SOFC

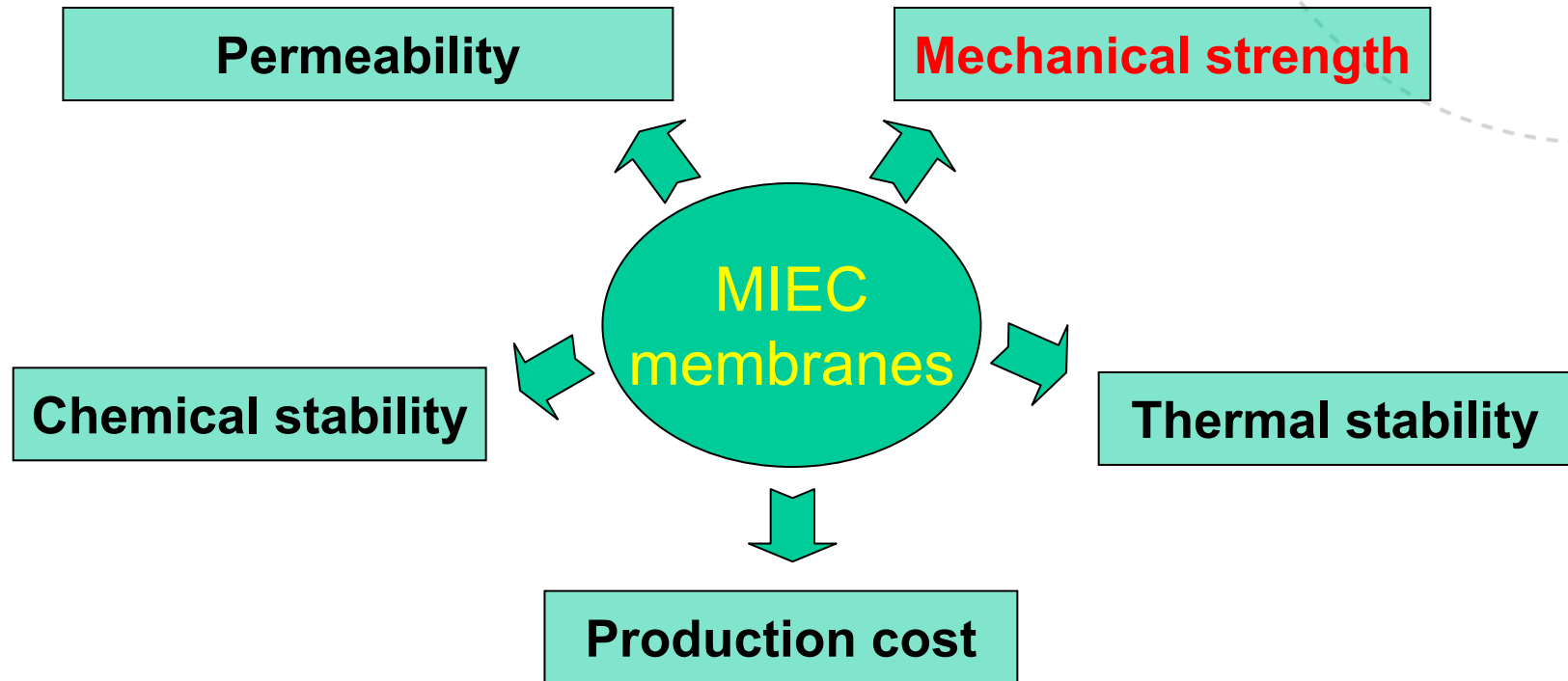
Oxygen separation membrane

Combined reactor for syngas production or natural gas conversion

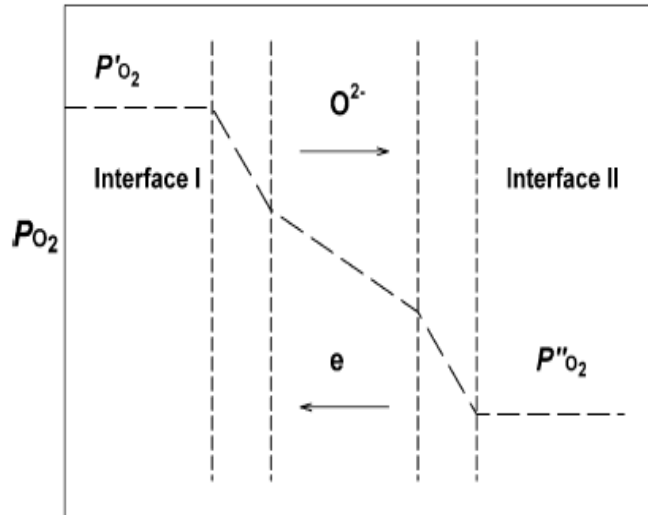
**Mixed ion and electron
conducting (MIEC) materials**

Introduction / *requirements*

Requirements for high temperature ceramic oxygen permeation membranes



6 Introduction / *model*

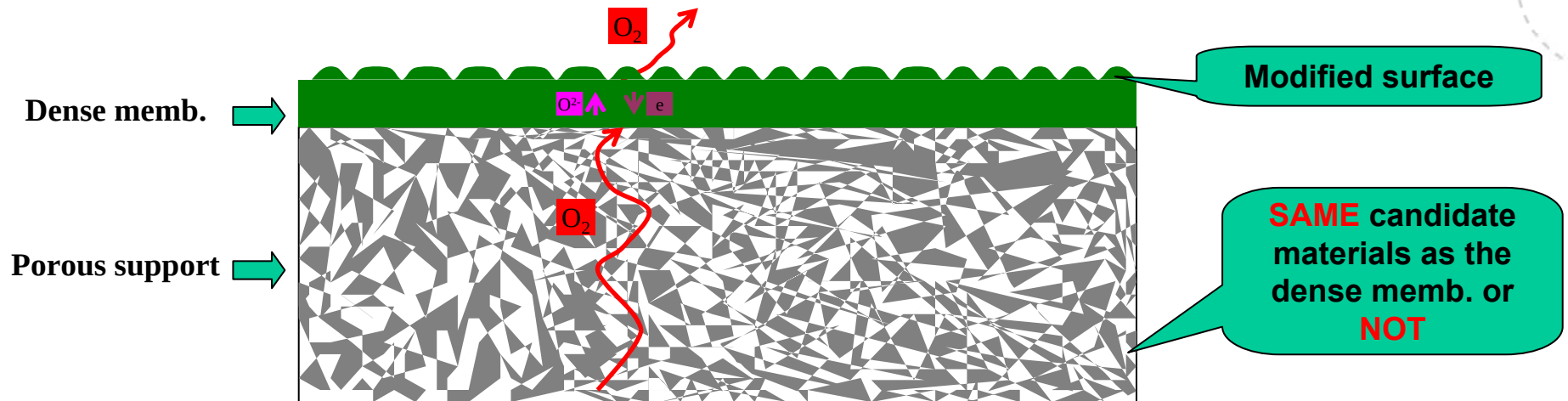


Decreasing the thickness of dense membrane

Increasing the flux

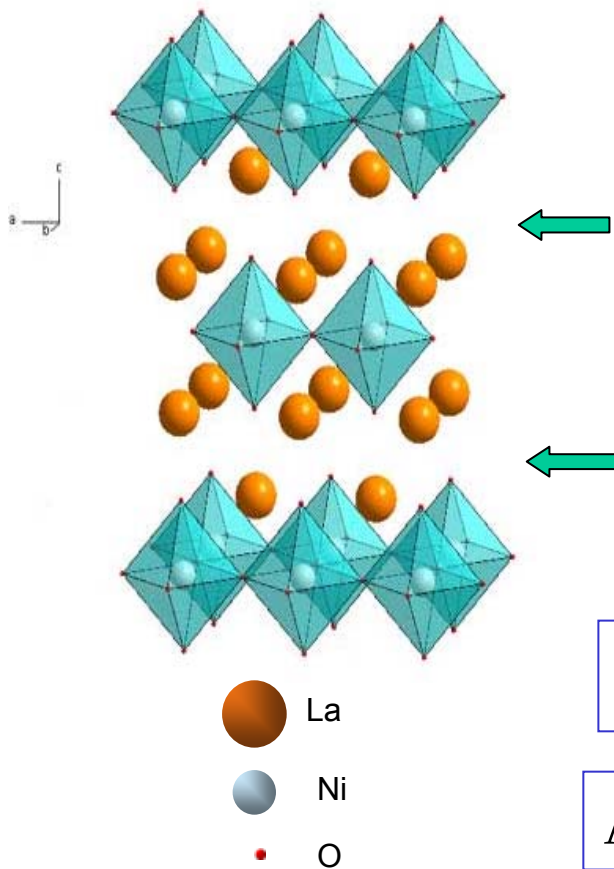
Appropriate porous support

Providing main mechanical strength

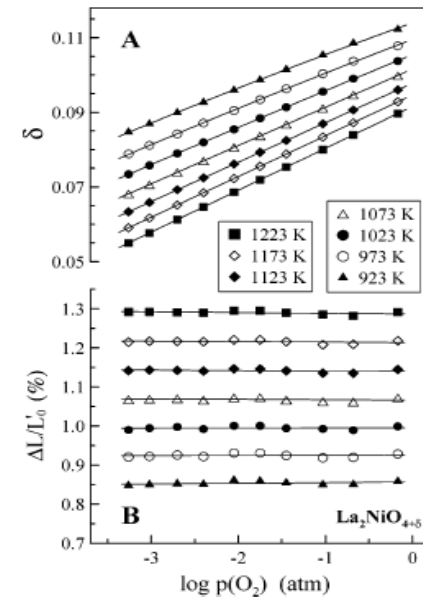
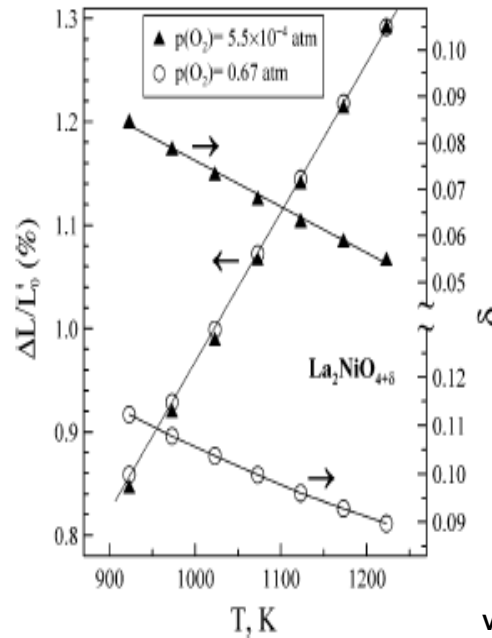


7 Material / $\text{La}_2\text{NiO}_{4+\sigma}$

Candidate material for dense thin membrane



Ideal structure of $\text{La}_2\text{NiO}_{4+\sigma}$



Vladislav V. Kharton, et.al. *Chem. Mater.* 2007, 19, 2027-2033

Chemical expansion of $\text{La}_2\text{NiO}_{4+\sigma}$ is almost independent of oxygen content variation due to anisotropic crystal structure

Additionally

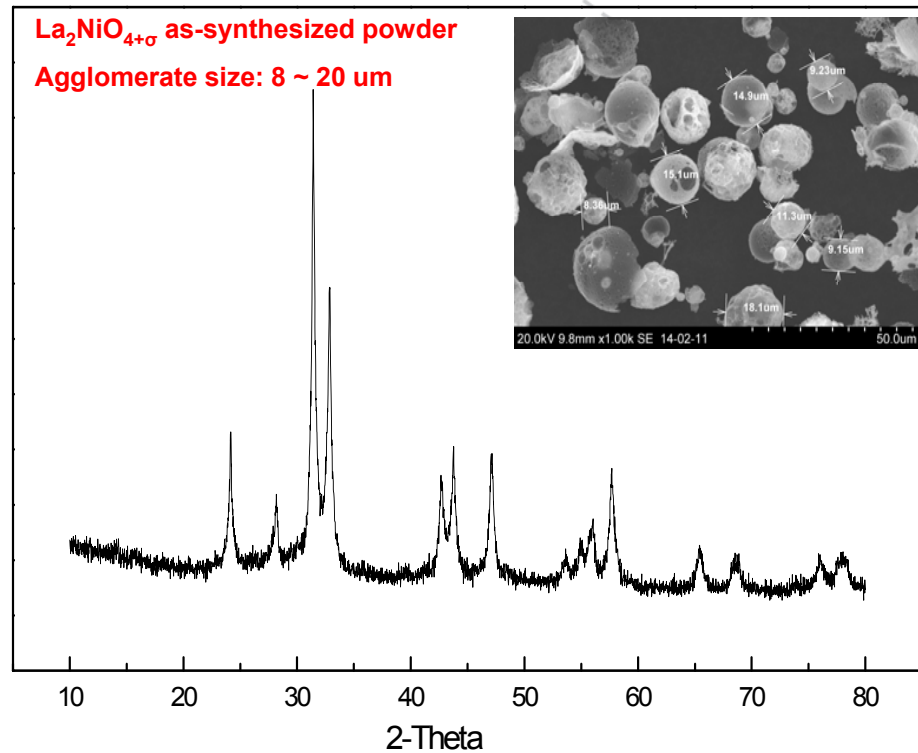
- Sufficient permeation flux
- High catalytic activity
- Anisotropic diffusion

8 Material / $\text{La}_2\text{NiO}_{4+\sigma}$

Synthesized by spray pyrolysis



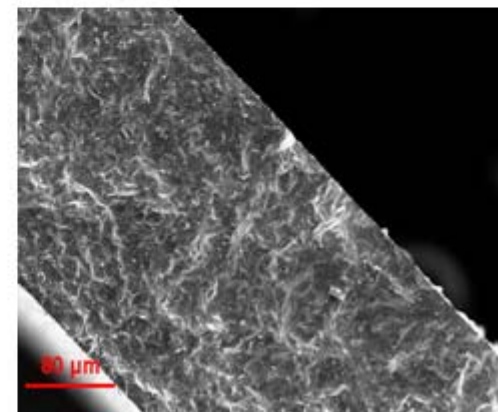
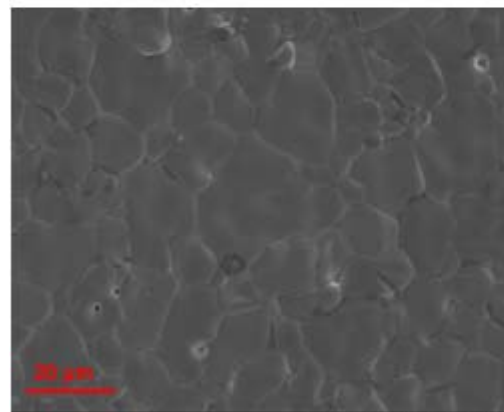
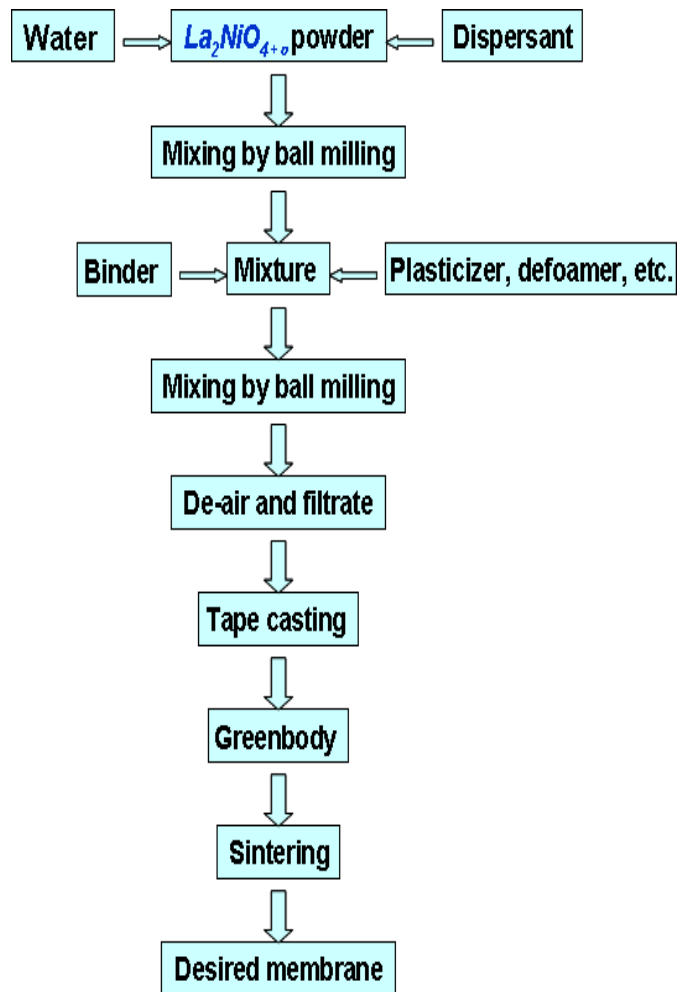
$\text{La}_2\text{NiO}_{4+\sigma}$ as-synthesized powder
Agglomerate size: 8 ~ 20 μm



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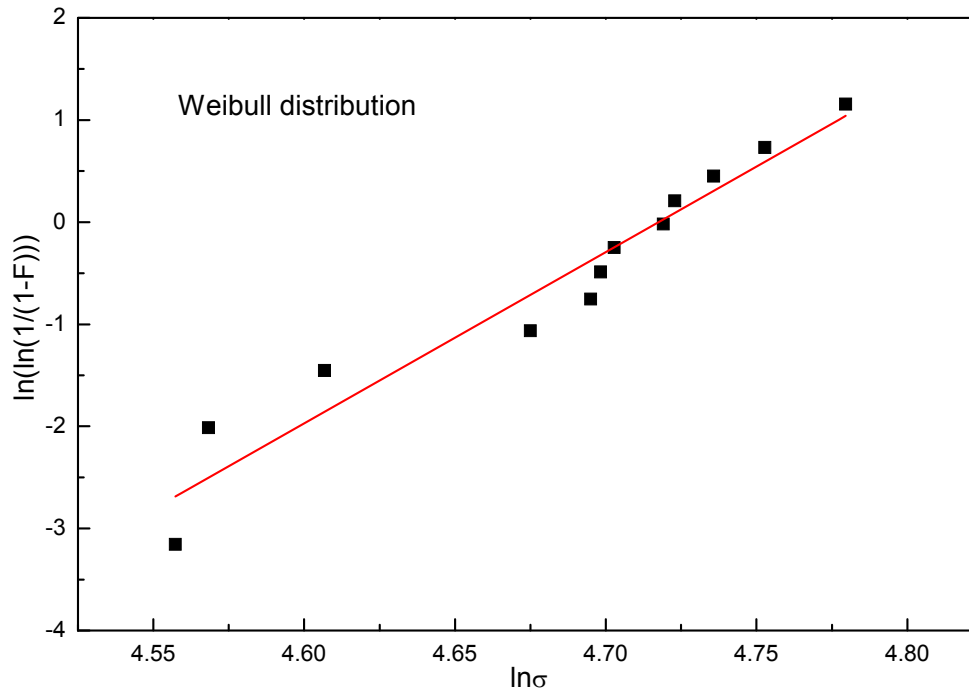
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Water based tape casting / $\text{La}_2\text{NiO}_{4+\sigma}$ membranes



Mechanical properties / fracture strength

Ball on ring test of $\text{La}_2\text{NiO}_{4+\sigma}$ membranes



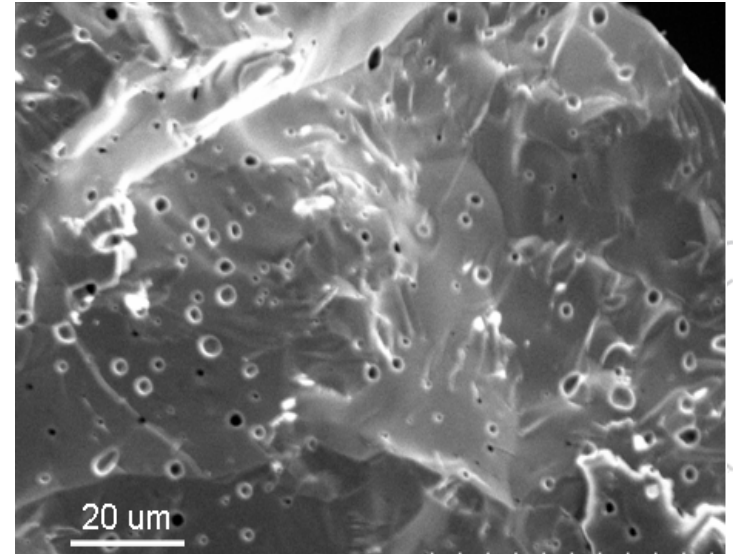
Average biaxial fracture strength: 109 MPa

Weibull modulus : 17

Relative density: 96 %

Fracture strength value (porosity ~ 52 %) in literature is less than 25 MPa at R.T

B.X.Huang, et.al, Thermo-mechanical properties of La_2NiO_4 .
J.Mater Sci (2011) 46:4937 - 4941

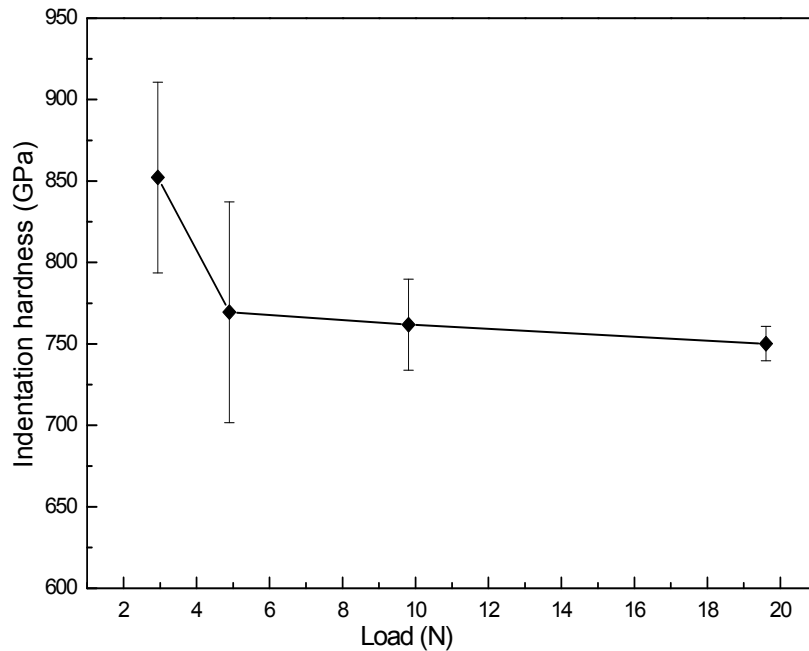


Fracture surface

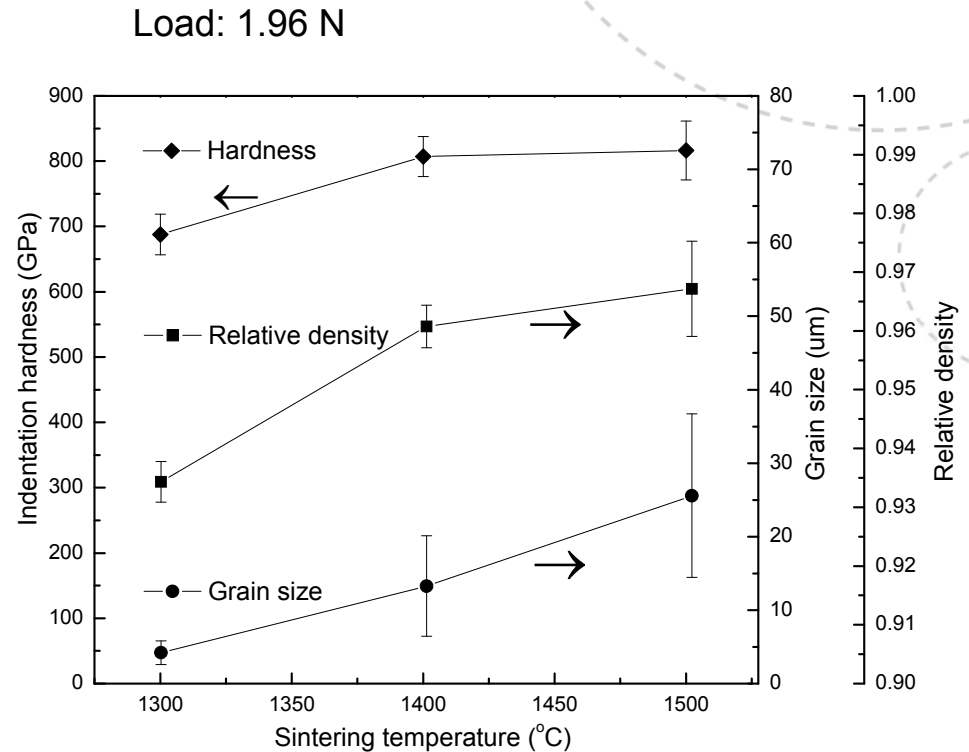
Transgranular fracture mode is expected

Mechanical properties / *hardness*

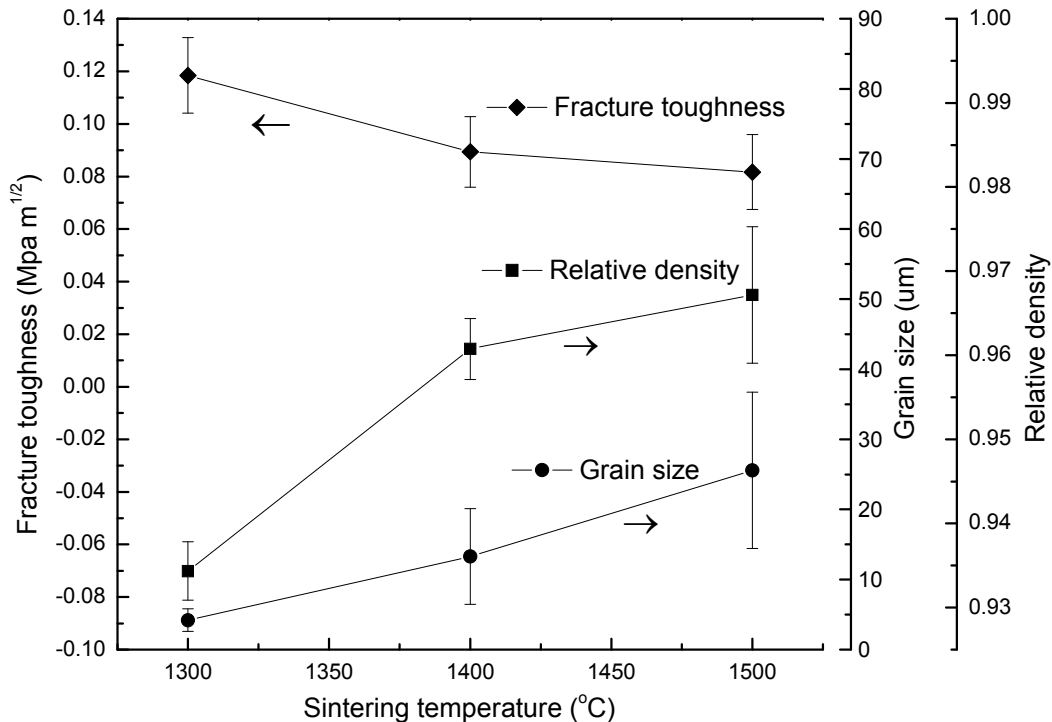
Indentation hardness of $\text{La}_2\text{NiO}_{4+\sigma}$ pellet



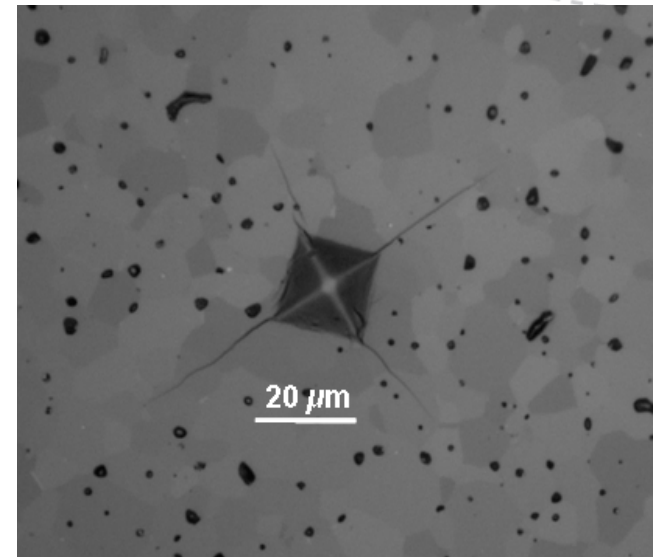
Indentation size effect can be found



Indentation fracture toughness of $\text{La}_2\text{NiO}_{4+\sigma}$ pellet

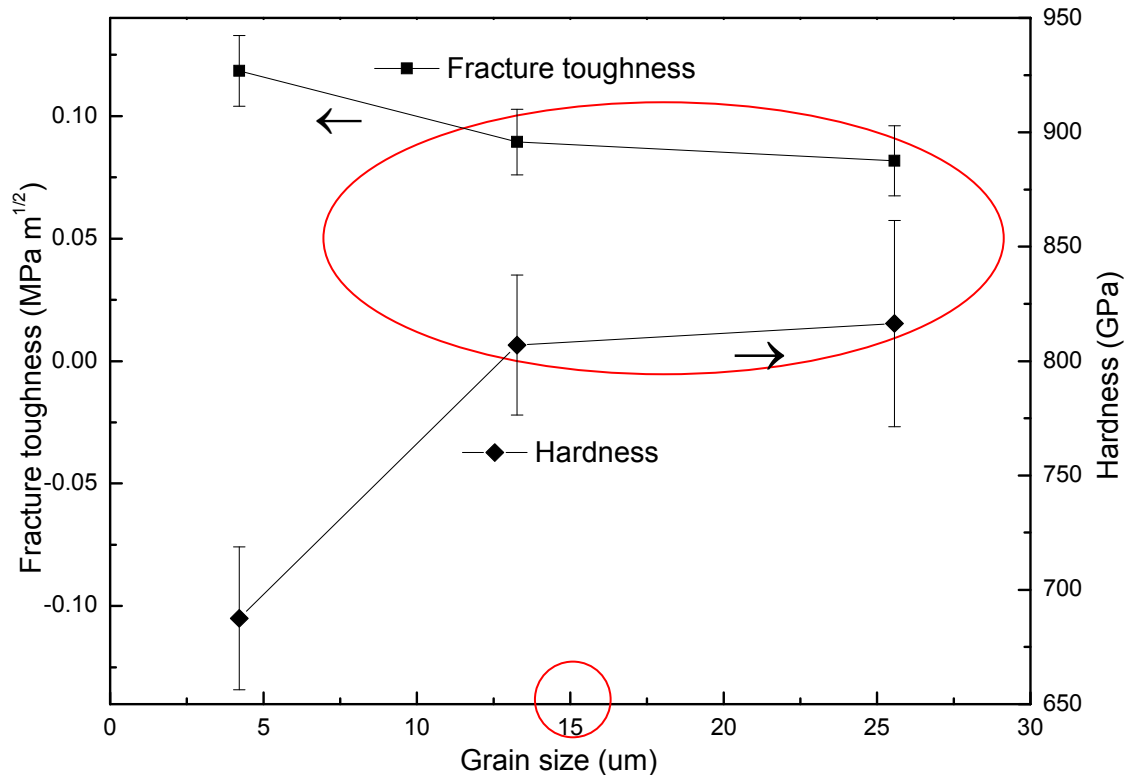


Load: 1.96 N



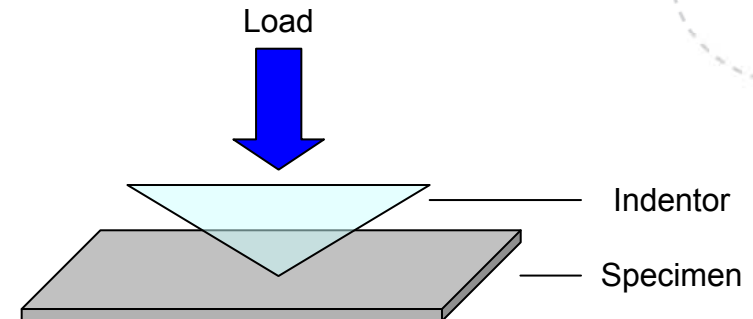
Optical microscopy image of indent

The fracture toughness value is much lower than the typical value of ordinary polycrystalline ceramics ($2\text{--}6 \text{ MPa m}^{1/2}$)



Load: 1.96 N

Indentation size: ~15 μm



Grain boundary effect would be less and less when the grain size is larger than indentation size

Summary

- ▶ Developed a successful method for $\text{La}_2\text{NiO}_{4+\sigma}$ water based tape casting, membranes with a density of above 96 % of the theoretical density were prepared by this method.
- ▶ $\text{La}_2\text{NiO}_{4+\sigma}$ has very good chemical stability, but a poor mechanical properties, fracture strength: $\sim 109 \text{ MPa}$; hardness: $\sim 780 \text{ GPa}$; fracture toughness: $\sim 0.1 \text{ MPa m}^{1/2}$
- ▶ Asymmetric membranes with $\text{La}_2\text{NiO}_{4+\sigma}$ as support are expected to have poor mechanical performance in long term operation condition, therefore alternative materials as the porous support are necessary.

Acknowledgement

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GDF SUEZ



Thanks for your attention