DTU Energy
Department of Energy Conversion and Storage

Stockholm University

Development of Porous $LaNi_{0.6}Fe_{0.4}O_3$ Electrodes with tailored microstructure for High Temperature and Pressure Alkaline Electrolysis Cells

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Outline

- Motivation
- Stabilization of $LaNi_{0.6}Fe_{0.4}O_3$ (LNF) in aqueous media
- Rheological characterization of LNF suspensions
- Microstructural characterization of sintered LNF layers
- Conclusion and outlook

Motivation

- Bimodal porosity distribution with pore sizes around 50-100 nm and 2-10 μm respectively
- 3D interconnected open porous network
- Conventional, easily scalable processing methods; e.g. screen printing, dip coating or tape casting

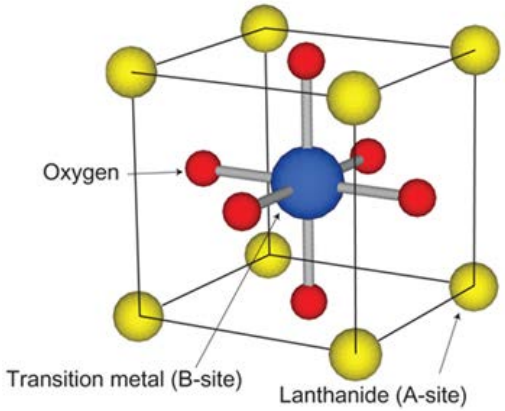
Suspensions

$LaNi_{0.6}Fe_{0.4}O_3$ powder (SSA_{BET}=12.6 m^2/g)

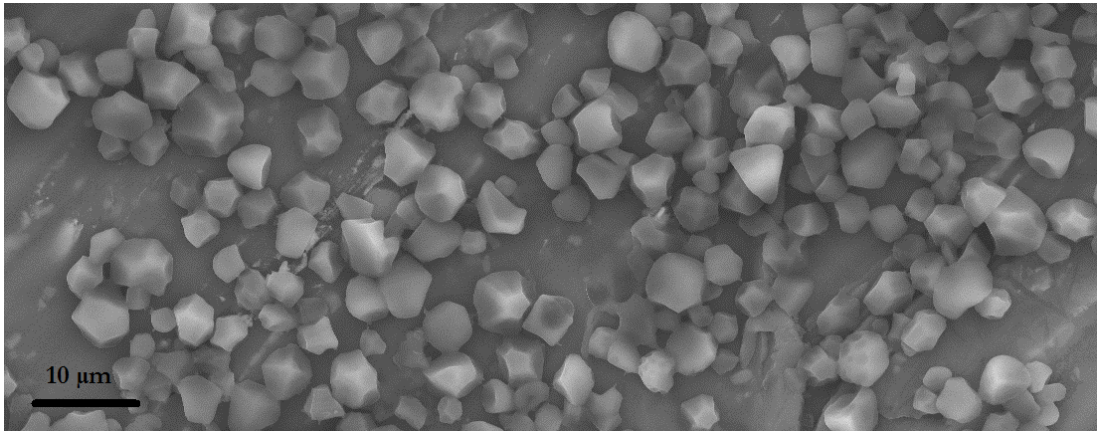
Water

Dispersants
PVP k15 (10,000 g/mol)
PMAA-NH_4 (15,000 g/mol)
PAA (5,000 g/mol)

Pore former
Rice starch
Size = 4.3 ± 1.3 μm



J. Suntivich et al., Nature Chem. 3 (546-550), 2011



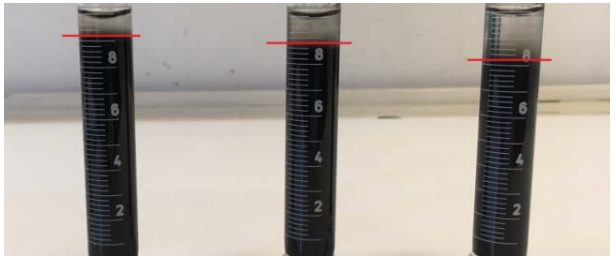
Sedimentation analysis

1.5 vol% LNF suspensions
ball milled 84 h

PVP, 14 days

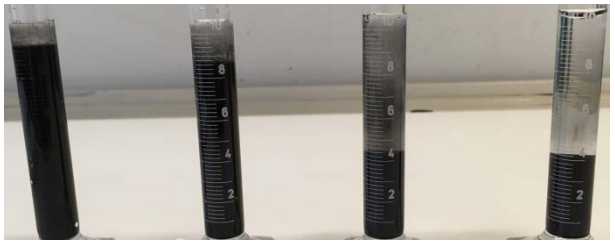
0.25 mg/m² | 0.79 mg/m² | 2.5 mg/m² | 8.0 mg/m²

PAA, 6 days



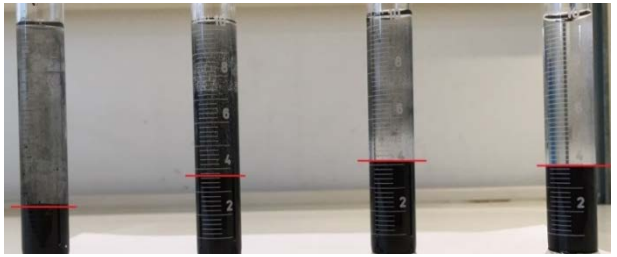
0.5 mg/m² | 0.75 mg/m² | 1.0 mg/m²

PMAA, 4 h



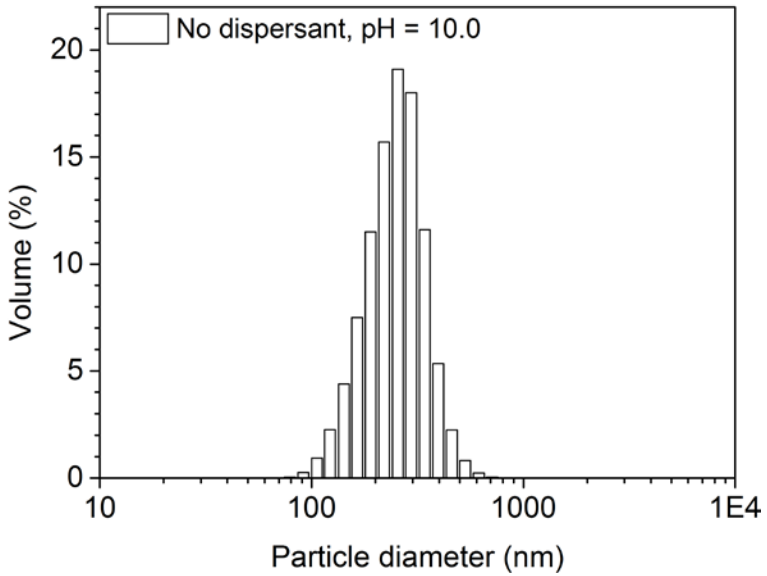
0.25 mg/m² | 0.5 mg/m² | 0.75 mg/m² | 1.0 mg/m²

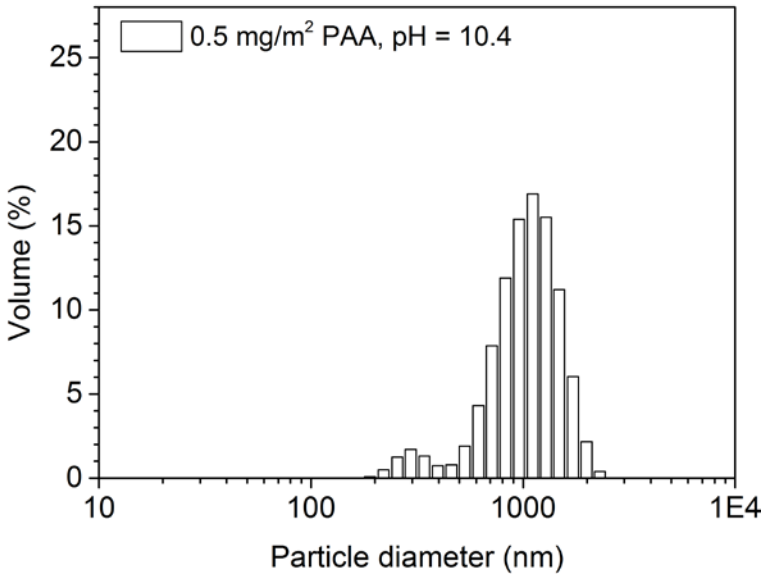
PMAA, 48 h

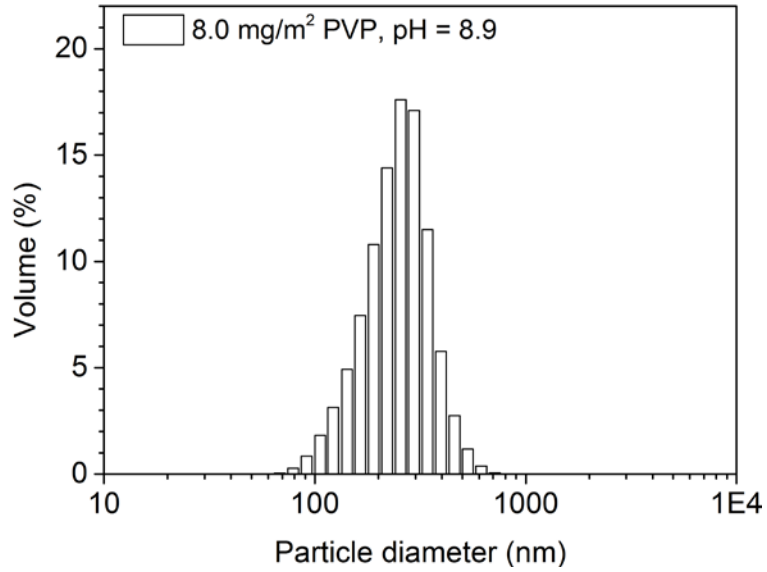


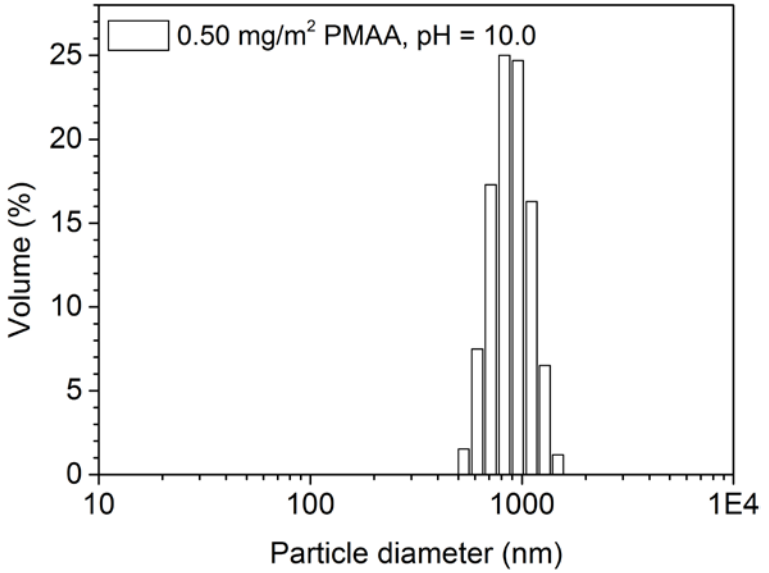
0.25 mg/m² | 0.5 mg/m² | 0.75 mg/m² | 1.0 mg/m²

Particle size measurements

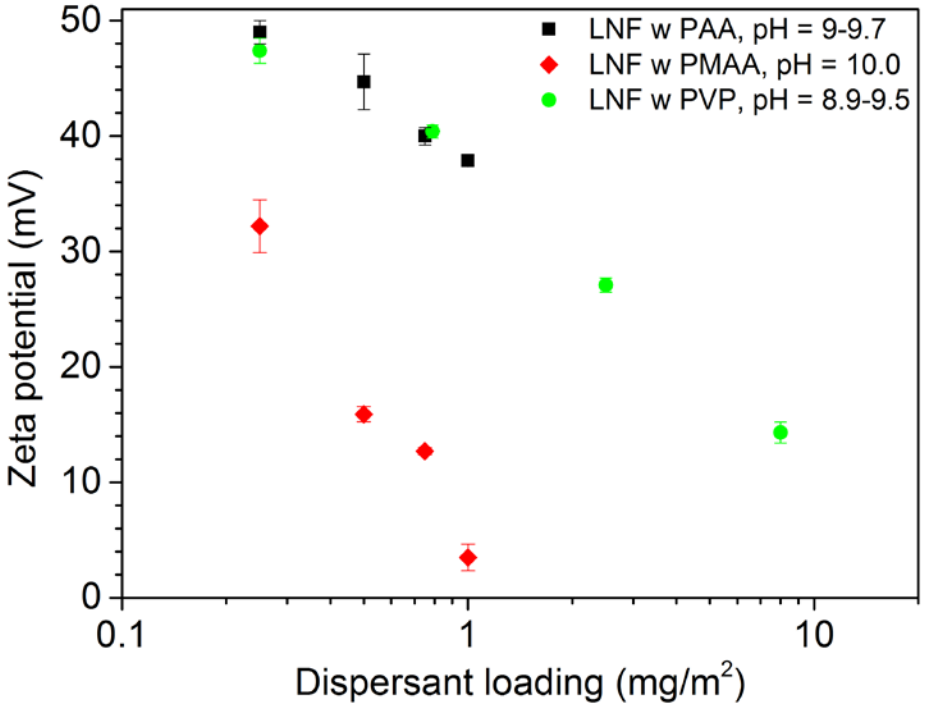


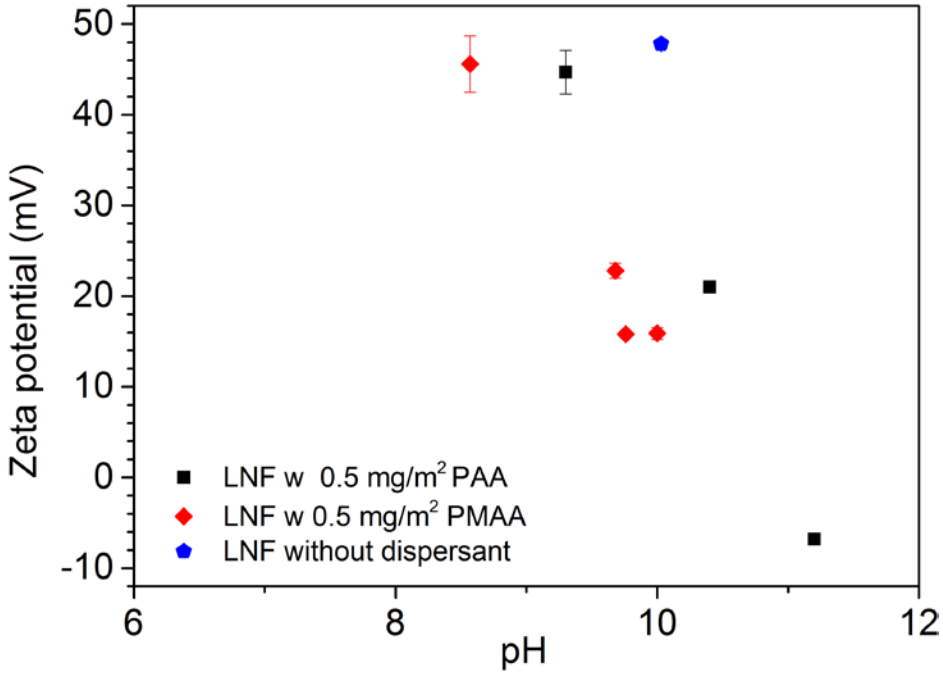






Zeta potential measurements





Rheological characterization

A pre-rotational shear (10 s^{-1} for 2 min) was performed before of the followsing step:

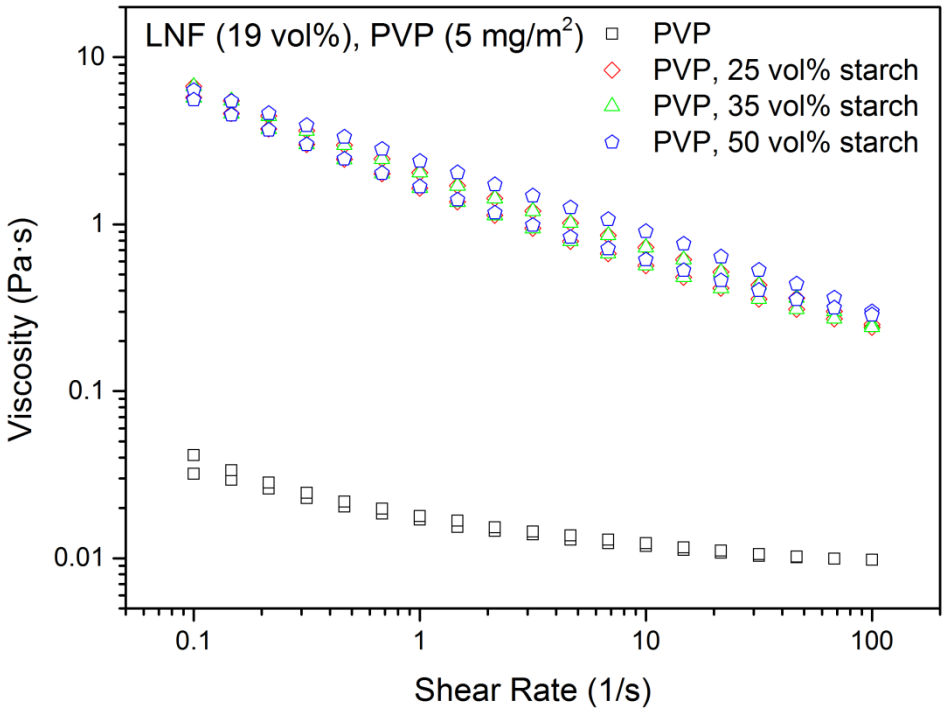
i. Pre-oscillatory shear (10 rad·s^{-1}, 0.1%)
Amplitude sweep at 10 rad·s^{-1}

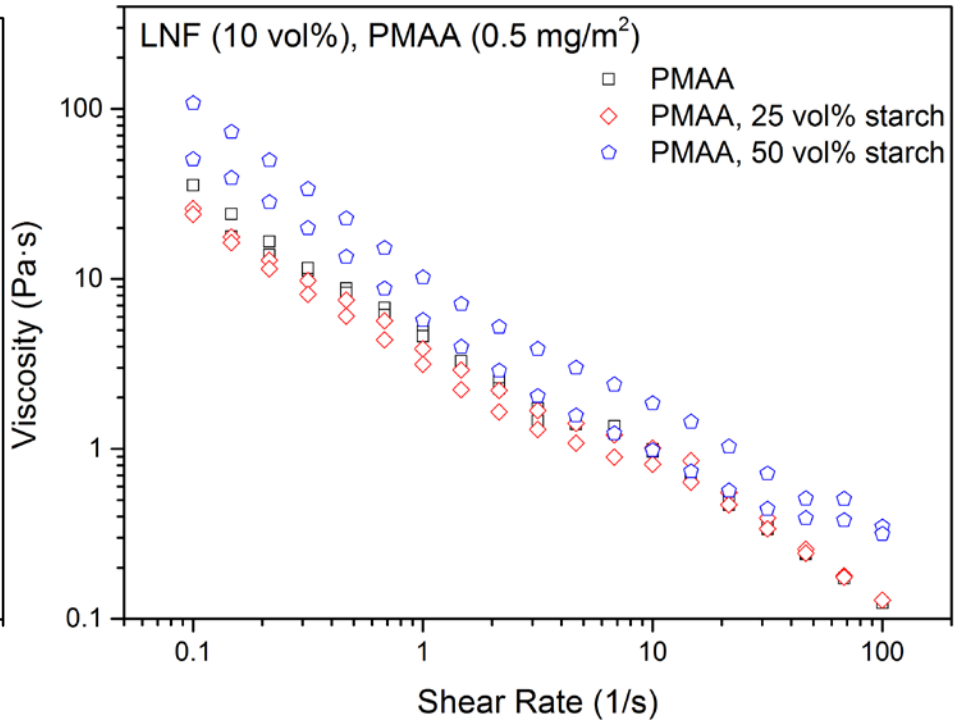
ii. Pre-oscillatory shear (10 rad·s^{-1}, 0.1%)
Frequency sweep in LVE

iii. Flow curve (0.1-100-0.1 s^{-1})
Rotational shear at 10 s^{-1} for 2 min

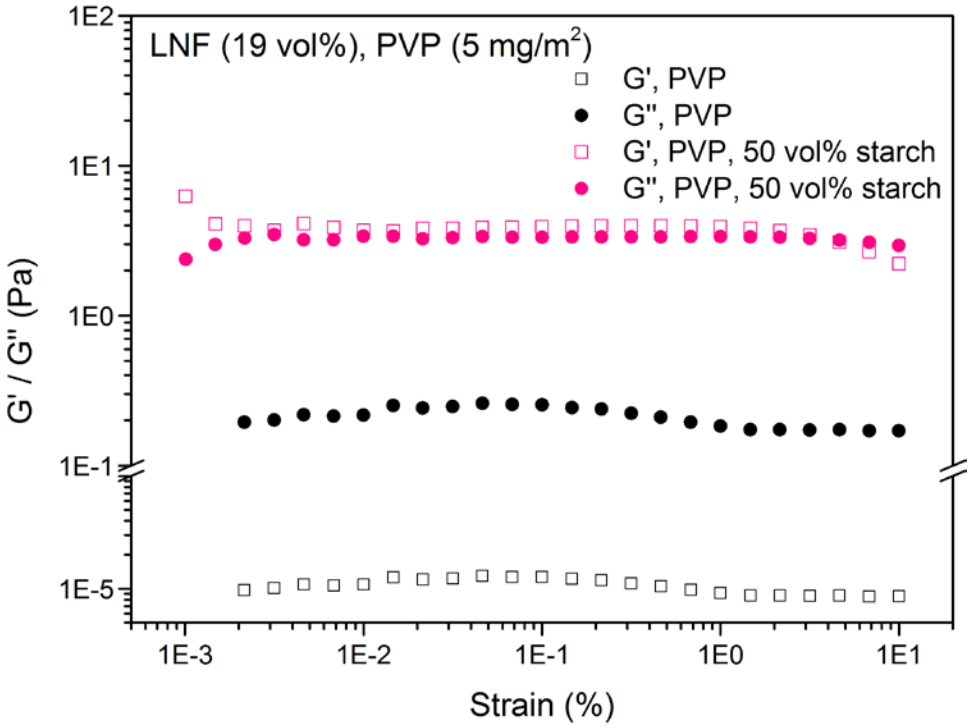
Concentric cup and cylinder (CC27) measuring system from Anton Paar GmbH

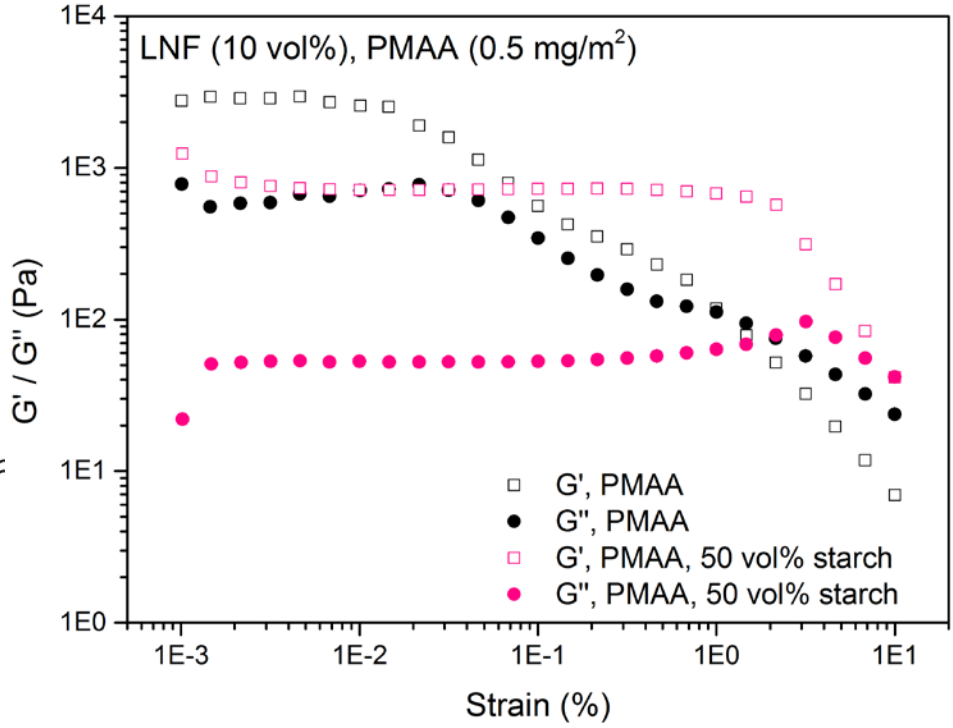
Rheological characterization – flow curves



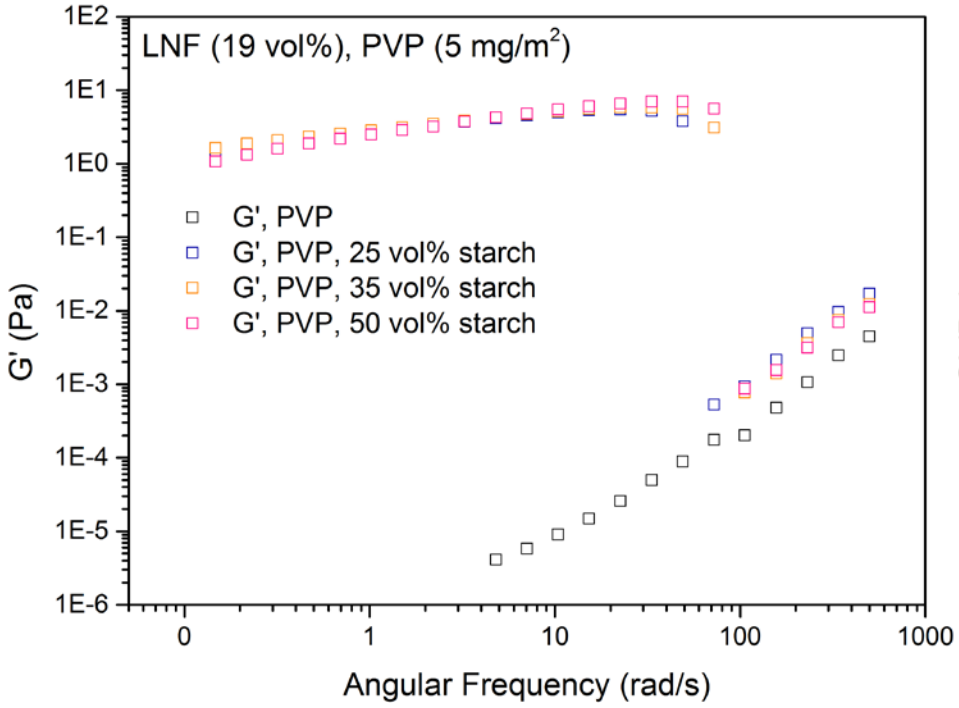


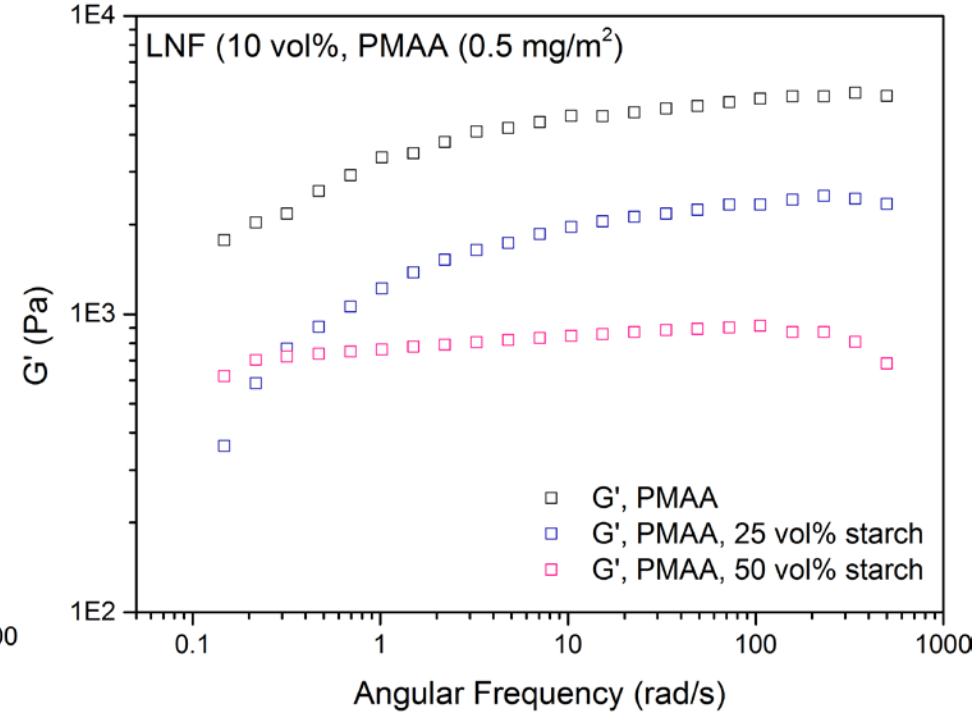
Rheological characterization – Amplitude sweeps





Rheological characterization – Frequency sweeps





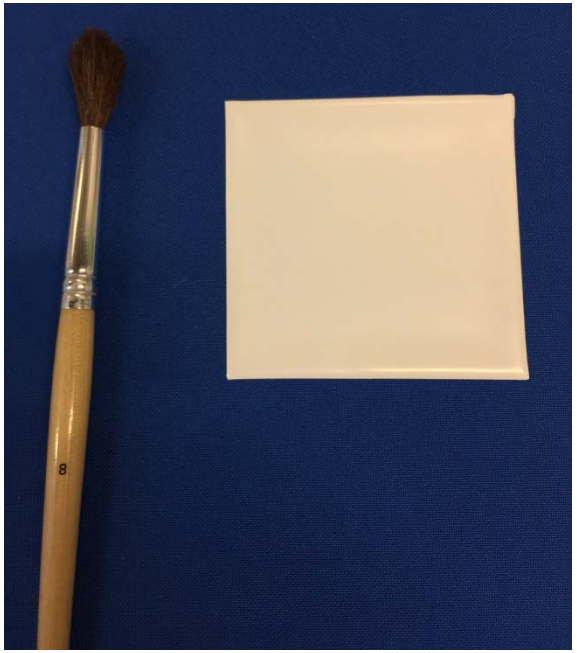
Deposition and sintering

Deposition by brush painting on sintered, meso-porous YSZ substrates

Drying at 80 °C for 30 min to form a starch consolidation casted [1] layer

Sintering

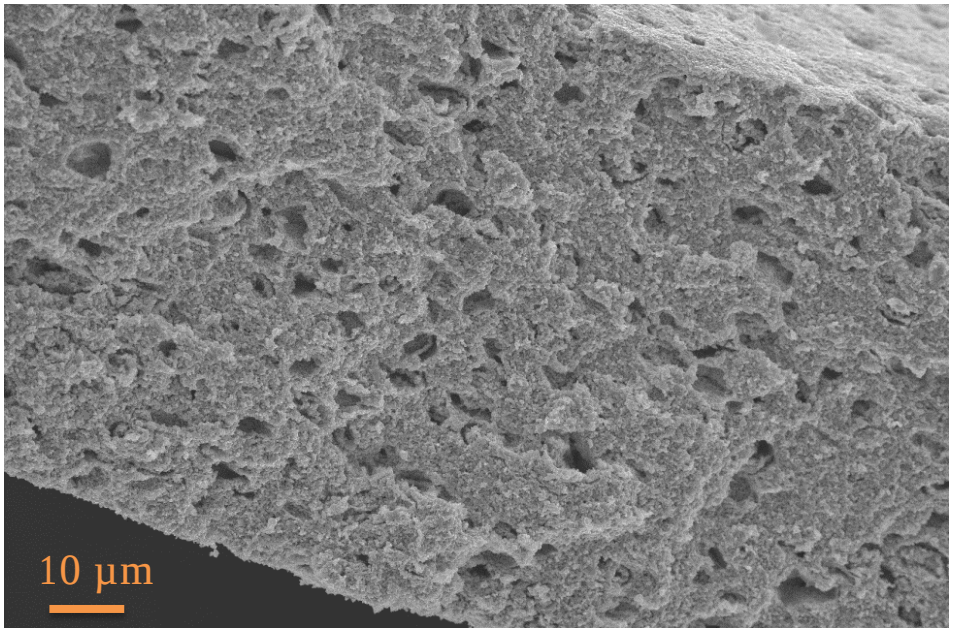
ramp rate 1°C/min from 20-250°C /1 h
holding 250°C / 1 h
ramp rate 1°C/min from 250-500°C
ramp rate 2°C/min from 500-1100 °C
holding 1100°C / 1 h
ramp rate 2°C/min 1100-20 °C



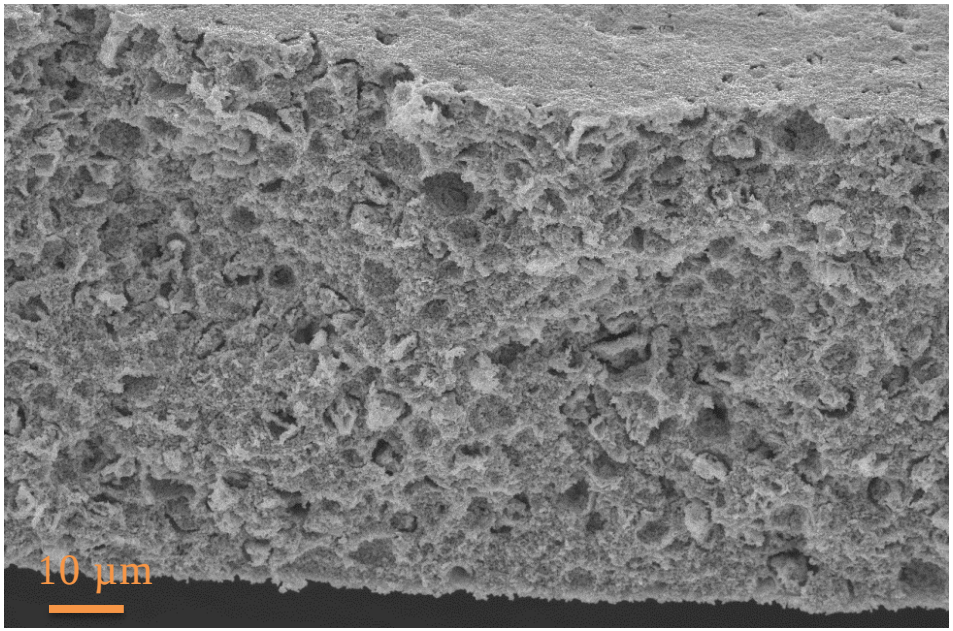
[1] Lyckfeldt, O. and Ferreira, J. M. F., J. Eur. Ceramic Soc., 1998, 18, 131-140

Microstructural characterization of sintered porous LNF layers

LNF, PVP and 25 vol% starch
Cracked cross section

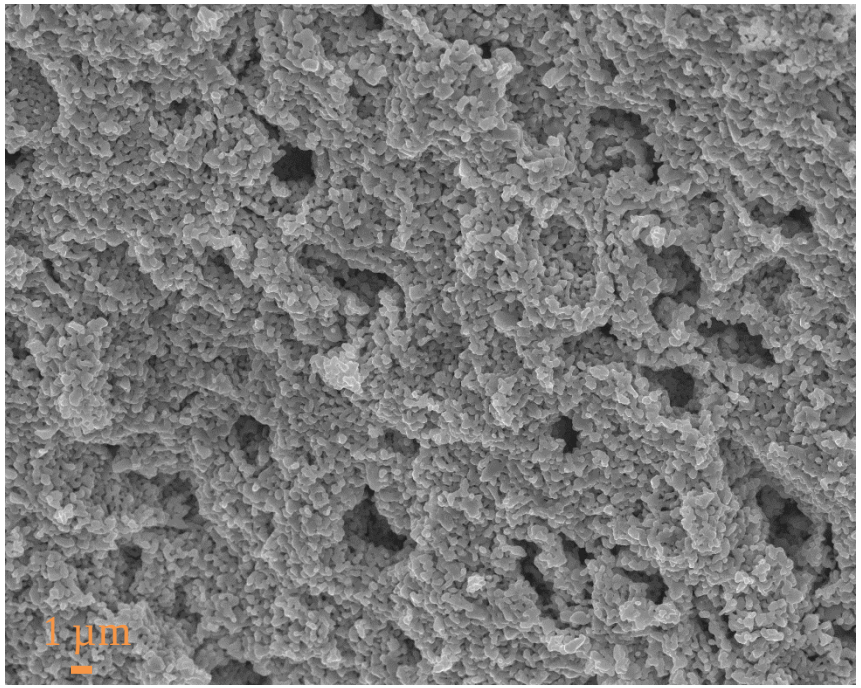


LNF, PVP, 50 vol% starch
Cracked cross section

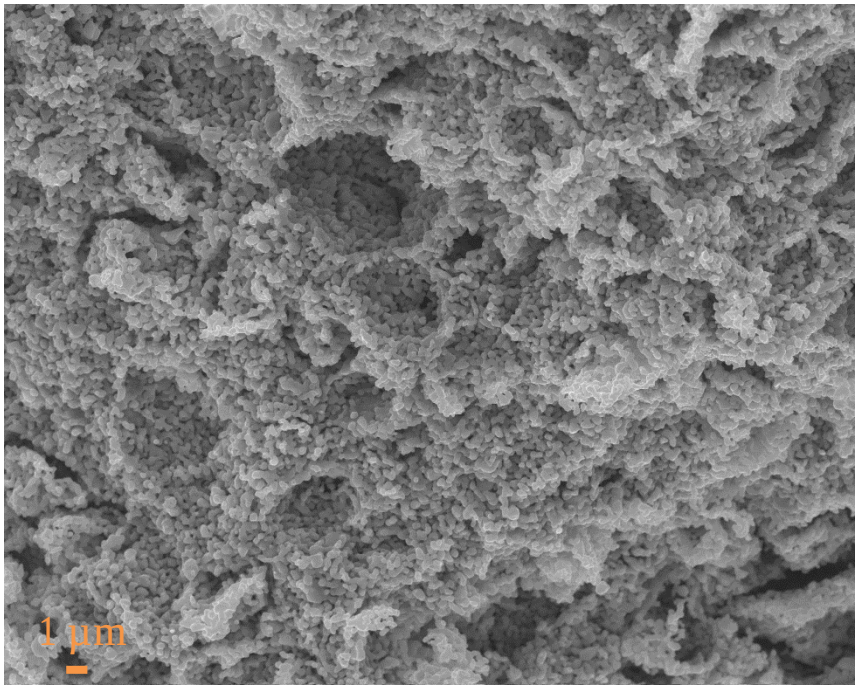


Microstructural characterization of sintered porous LNF layers

LNF w PVP & 25 vol% rice starch
Cracked cross section

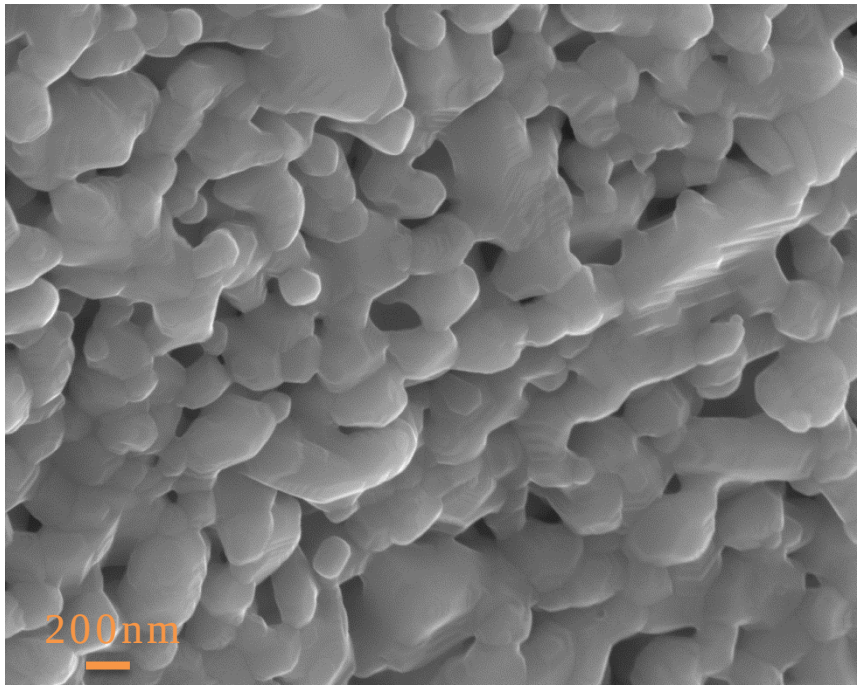


LNF w PVP & 50 vol% rice starch
Cracked cross section

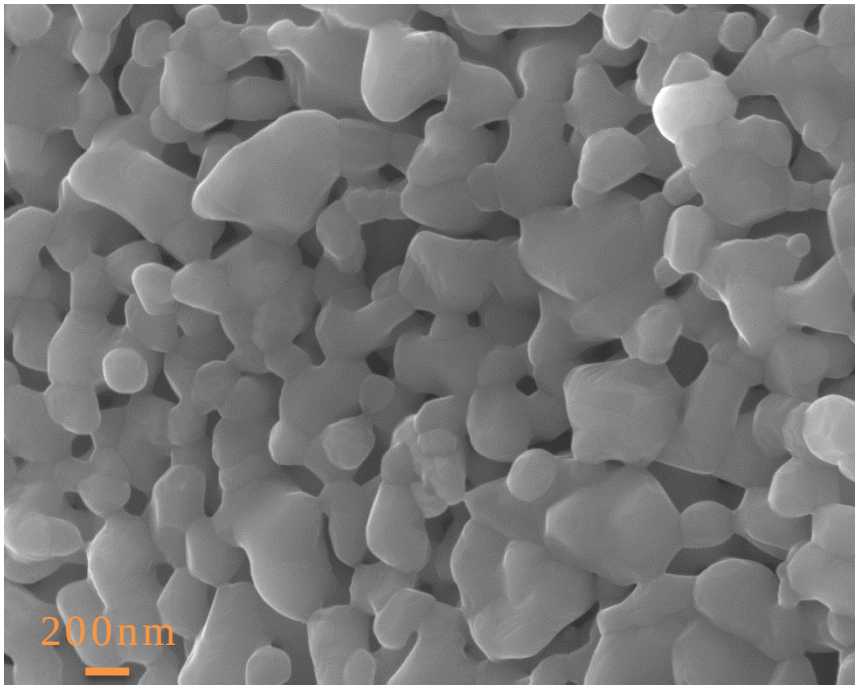


Microstructural characterization of sintered porous LNF layers

LNF, PVP, 25 vol% starch
Top surface

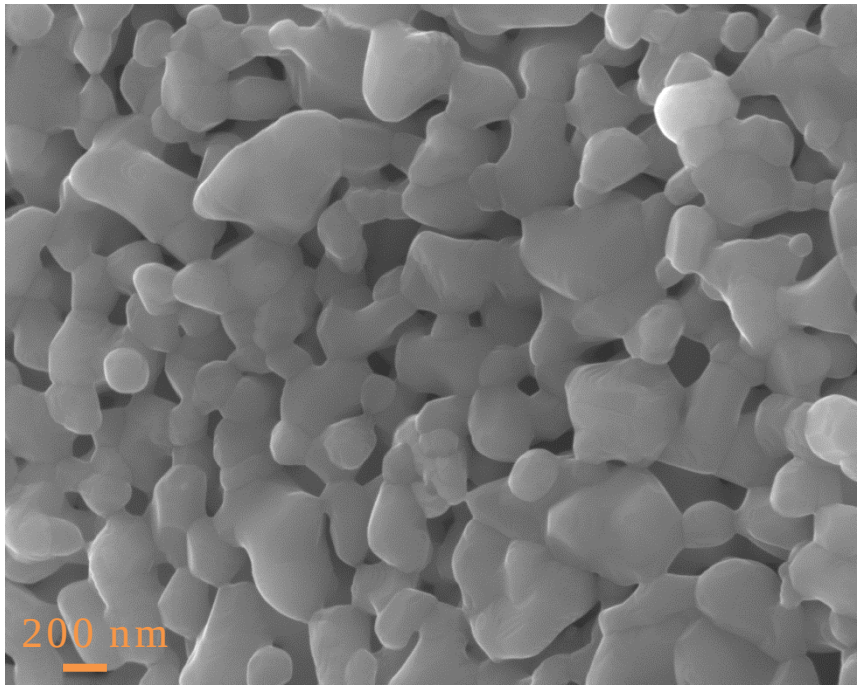


LNF, PVP, 50 vol% starch
Top surface

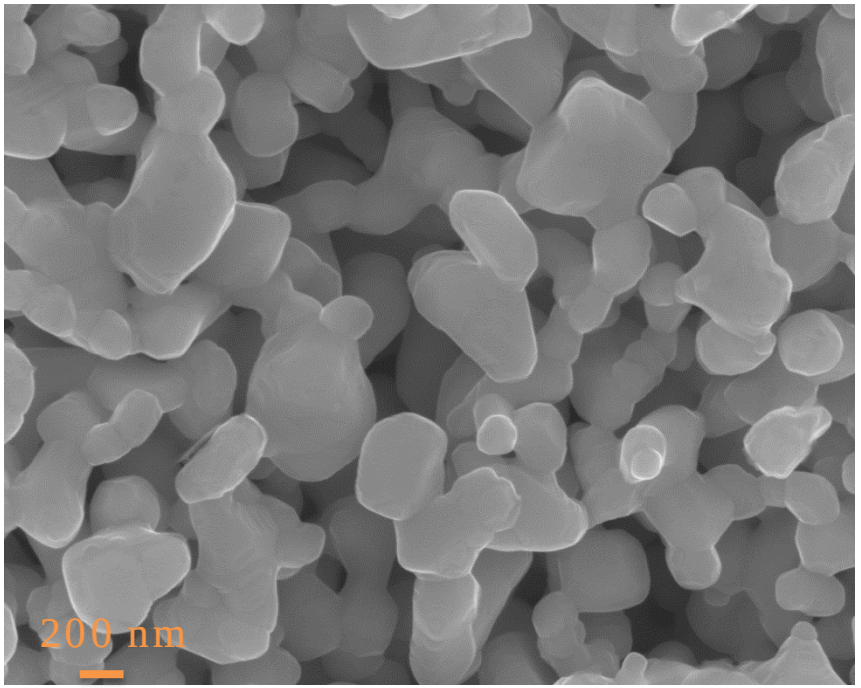


Microstructural characterization of sintered porous LNF layers

LNF, PVP, 50 vol% starch
Top surface



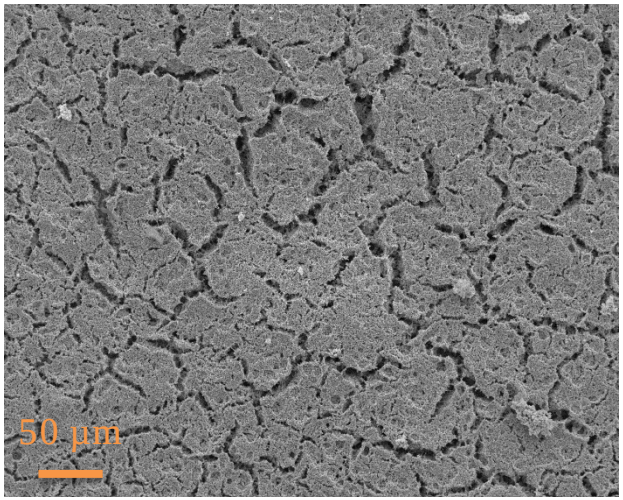
LNF, PMAA, 50 vol% starch
Top surface



Conclusion & outlook

- LNF structures with a dual porosity distribution have been achieved using PVP and PMAA as dispersant and rice starch as macro-pore former.

- Successfully implementing the gel consolidation casting step is expected to mitigate crack formation and delamination.



- A quantitative analysis of the pore sizes and electrochemical performance of the electrode layers are future plans

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