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Fracture of Porous Ceramics: Application to the Mechanical Degradation of Solid Oxide Cell during Redox Cycling

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A model based on the Phase Field approach has been developed to simulate the local fracture in porous electrode microstructures. The model capacity to predict the crack nucleation and propagation has been studied with theoretical considerations. For the model validation, micro-compression tests have been performed on porous YSZ micro-pillars. As expected, the compressive fracture strength has been found to decrease with the material porosity. Moreover, a transition in fracture mode has been detected from a brittle behavior toward a local damage when increasing the porosity. It has been shown that the model is able to reproduce correctly the experimental results. The validated model has been used to simulate the fracture of the YSZ backbone induced by the Ni re-oxidation in a typical Ni-YSZ cermet. The generation and localization of the micro-cracks have been discussed according to the degree of Ni re-oxidation and the local morphology of the electrode microstructure.

Introduction

Solid Oxide Cell (SOC) is a high-temperature electrochemical device that can be operated in both fuel and electrolysis modes. It is constituted of a dense electrolyte made of Ytria Stabilized Zirconia (YSZ) sandwiched between two porous electrodes. The hydrogen electrode is classically made of a cermet composed of nickel (Ni) and YSZ (Ni-YSZ) (1,2). Because of system failures in operation (e.g. related to fuel shortage or to air re-introduction during the system shutdown), the nickel is liable to be re-oxidized inducing its volume expansion (3). The Ni swelling within the cermet generates high tensile stresses in the YSZ backbone leading to the apparition of micro-cracks (3). For classical anode supported cell, the mechanical damage of the ionic conducting phase within the cermet results in a substantial degradation of the overall cell performances (4-9). It is therefore required to further enhance the hydrogen electrode robustness for a higher redox tolerance. For this purpose, it is essential to predict accurately the formation and the distribution of micro-cracks in the YSZ network during Ni re-oxidation.

Nevertheless, the accurate prediction of cracks nucleation and propagation in the complex microstructures of porous ceramics is still a subject of investigation. Moreover, there is a lack of data on fracture properties for the porous YSZ. In this frame, mechanical characterization using uniaxial compression testing on micro-pillars has been carried out at different porosities considering two types of zirconia stabilized with 8% and 3% molar of Yttrium (i.e. 8YSZ and 3YSZ respectively). The experimental data have been used to validate a model based on the Phase Field Method (PFM) applied to fracture mechanics. The fracture of the YSZ backbone induced by the Ni re-oxidation has been then simulated for a typical Ni-YSZ cermet. The generation and localization of the micro-cracks have been discussed according to the degree of Ni re-oxidation and the local microstructural properties of the electrode.

Experiments

Sample Preparation

Samples with low and intermediate porosities were manufactured using a specific protocol. The preparation consisted in sintering pellets of compacted 8YSZ powders at different temperatures (i.e. 1270°C, 1230°C, 1200°C and 1150°C) during 1 hour under air. To produce samples with higher porosity, a typical Ni-8YSZ cermet was used by dissolving the nickel in HNO₃ acid at room temperature. The same protocol was applied on a Ni-3YSZ cermet to obtain a highly porous 3YSZ material. Finally, the study was complemented by the characterization of a fully dense 8YSZ membrane.

For each sample, a polished cross-section was observed by Scanning Electron Microscopy (SEM) to check the microstructure homogeneity and to estimate the sample porosity. For instance, the SEM images for the 8YSZ samples have revealed a homogenous microstructure with a higher porosity for the lower sintering temperature (Fig. 1). In addition, 3D Focused Ion Beam – Scanning Electron Microscopy (FIB-SEM) reconstructions have been realized. On the one hand, the porosity has been measured on the 3D volumes to confirm the estimation obtained with the 2D images. On the other hand, the reconstructions have been used as inputs for the multiscale modeling approach.

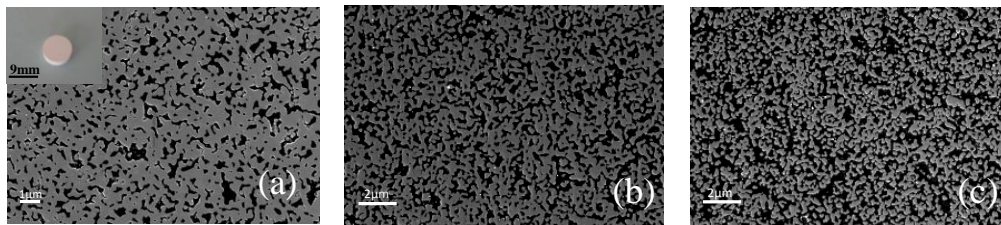


Fig 1. SEM observations of 8YSZ samples sintered at: a) 1270°C, b) 1230°C, c) 1150°C under air. The insert in Fig. 1a shows the pellet after sintering.

After manufacturing, the specimens for the mechanical characterizations were directly milled in the pellets using a Plasma Focused Ion Beam (PFIB) working with Xenon ions. The specimens were etched in the shape of micro-pillars for uniaxial micro-compression testing (Fig. 2a). In this frame, a special attention was paid to fulfil the geometrical recommendations regarding the specimen dimensions for accurate measurements (9,10). Indeed, as shown in Fig. 2b, the micro-pillars present a nearly

perfect axisymmetric geometry with a section of $60 \pm 0.5 \mu\text{m}$. Moreover, the rule on the aspect ratio between the height of the pillar and the diameter, which is recommended to range between 2 and 3, was satisfied. Due to the Gaussian shape of the ion beam, the pillars were slightly tapered but a special attention was paid to limit the angle down to 6° . Finally, it has been checked by 3D reconstructions that the microstructure is not affected by the sample preparation (Fig. 2c). Indeed, the microstructural properties, which have been computed on the two volumes, remain unchanged. For instance, the tortuosity factors of the YSZ phase are equal to 1.35 before and after milling.

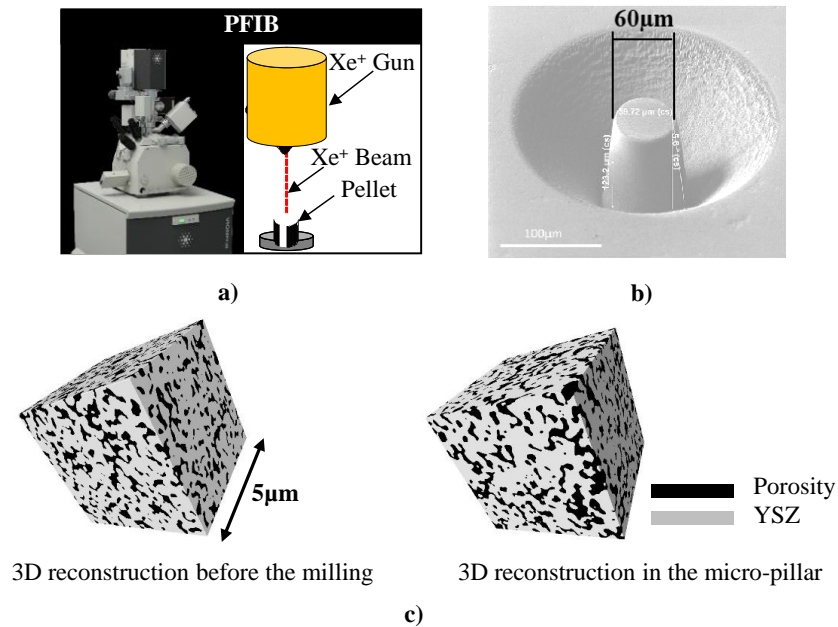


Fig 2. Procedure for the specimen preparation: a) Schematic representation of the Xenon PFIB used for the milling, b) micro-pillar obtained after milling, c) rendering volumes for the two reconstructed microstructures of the 8YSZ sample sintered at 1270°C (in the pellet and the micro-pillar).

Mechanical Testing

The micro-compression test has been chosen as it generates a quasi-uniform stress field in the tested pillars. Moreover, this approach is well adapted to the characterization of the thin cermet substrates as used in this work to prepare samples after Ni dissolution (cf. previous section). The micro-mechanical tests were performed using a nano-indenter (G200, Keysight technologies, USA) equipped with an optical microscope. Using a circular flat punch tip of $100 \mu\text{m}$ in diameter, the pillars were compressed with a constant strain rate of 0.05 s^{-1} . For each investigated porosity, several pillars were tested to measure the compressive fracture strength of the porous materials. Approximately five tests were conducted up to the sample failure to determine the fracture strength. Other complementary experiments were stopped just before the sample collapse. For these specimens, the distribution of cracks within the microstructure has been characterized on 2D FIB slices in order to assess the damage mode in the material. All the experimental results are provided in Table I.

Results for Samples with Low and Intermediate Porosities. As an illustration, a typical stress-strain curve is provided in Fig. 3 for the 8YSZ sample sintered at 1270°C . As

expected, a decrease of the fracture strength was found with decreasing the sintering temperature (i.e. with increasing porosity). The measured fracture strengths were 1455 ± 77 MPa, 950 ± 5 MPa, 470 ± 4 MPa and 124 ± 6 MPa for the samples sintered at 1270°C , 1230°C , 1200°C and 1150°C , respectively (Table I).

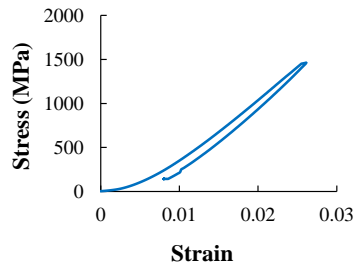


Fig 3. Stress-Strain curve at loading and unloading for 8YSZ samples sintered at 1270°C . The tests were stopped before the fracture.

For the tested samples stopped just before the total failure of the pillars, the visualization of cracks in the microstructure has shown the presence of large cracks parallel to the loading direction (Fig. 4). These observations point out a brittle behavior for the 8YSZ at low and intermediate porosities (11).

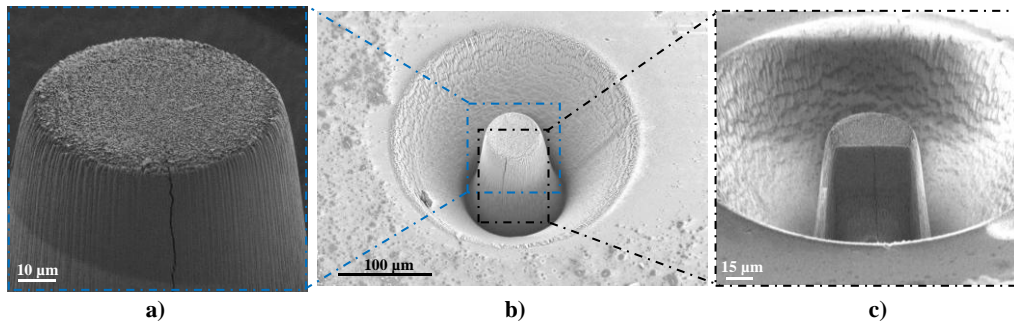


Fig 4. Ex-situ characterization after mechanical loading of the 8YSZ pillar sintered at 1270°C (low porosity): a) SEM image of a longitudinal crack on the edge of the pillar, b) SEM image of the tested pillar, c) FIB-SEM cross section showing the large crack in the bulk of the pillar.

Results for Samples with High Porosities. For the highly porous 8YSZ sample, the fracture strength, estimated to 25 ± 3 MPa, is consistent with the data measured on the 8YSZ pellets at low and intermediate porosities (Table I). Furthermore, as expected, the compression strength for the highly porous 3YSZ is significantly higher than for 8YSZ. Indeed, a value of 147 ± 22 MPa was measured in this case.

Post mortem characterizations were also realized for the pillars tested just below the threshold leading to the complete failure. As shown in Fig. 5, for 8YSZ and 3YSZ, a distribution of micro-cracks is detected in the bulk of the samples in the upper part of the pillar. This observation means that a diffuse damage is induced in the microstructure for these highly porous materials. Therefore, a transition has been highlighted from a brittle behavior at low and intermediate porosities towards a local damaging with micro-cracks at high porosity (11).

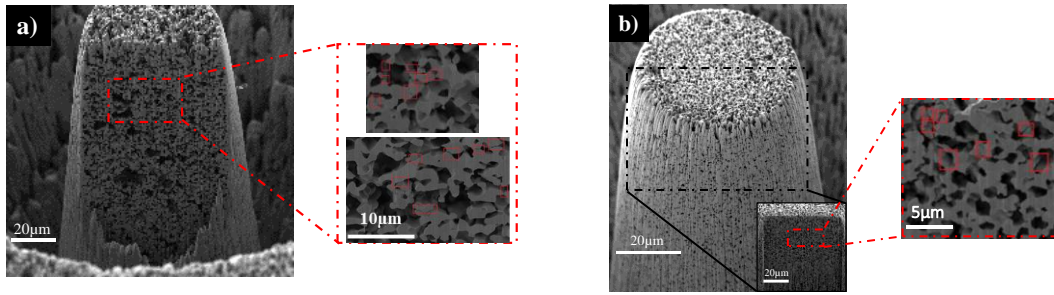


Fig 5. Ex-situ characterization of the highly porous pillars after the mechanical loading with a zoom showing the micro-cracks in the bulk of the pillar (highlighted by red rectangles): a) 8YSZ, b) 3YSZ. The tests were stopped before the fracture.

The compressive fracture strength is plotted in Fig. 6 as a function of the porosity for 8YSZ samples. An exponential decrease of the fracture strength is observed when increasing the porosity. It can be noticed that the transition towards an asymptotic behavior at high porosity is concomitant with the change in the fracture mode (Table I). Therefore, it can be proposed that the evolution of the fracture strength with the porosity is also dependent on the underlying mechanism controlling the rupture. All these experimental data have been used for the validation of the numerical model.

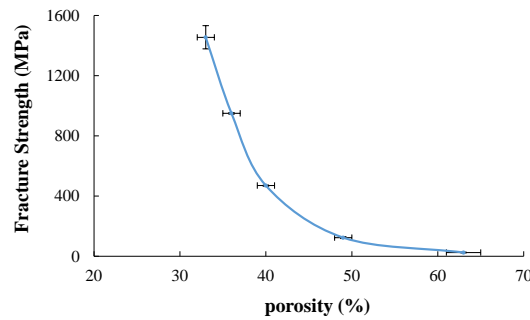


Fig 6. 8YSZ compressive fracture strength as a function of the porosity.

TABLE I. Results of the Mechanical Testing in uniaxial compression of micro-pillars. Highly porous samples refers to samples obtained after Ni dissolution.

Material	Porosity (%)	Fracture strength (MPa)	Fracture mode
Highly porous 3YSZ	63	147 ± 22	Local damaging
Highly porous 8YSZ	63	25 ± 3	Local damaging
8YSZ-T1150°C	49	124 ± 6	Brittle
8YSZ-T1200°C	40	470 ± 4	Brittle
8YSZ-T1230°C	36	950 ± 5	Brittle
8YSZ-T1270°C	33	1455 ± 77	Brittle
Dense 8YSZ electrolyte	0	2800 ± 400	Brittle

Modelling the fracture in porous electrodes

Model Description

The PFM approach has been selected to study the initiation and the growth of cracks in porous ceramics since it is mesh independent. The model is based on a diffuse

description of the crack through a smooth scalar damage variable \mathbf{d} . This variable is referred as the phase field and can evolve from zero to one. The pristine material corresponds to $\mathbf{d} = 0$ while the total rupture is reached when $\mathbf{d} = 1$. The region of transition between the two states is controlled by a length scale parameter ℓ . Marigo et al. (12-14) were the first to propose a general theoretical framework for the development of this method on the basis of the Continuous Damage Mechanics (CDM). The key idea of their work is the reformulation of the fracture problem as the minimization of an energy functional without any additional criterion. In the present study, the implemented PFM is the one developed by Miehe et al. (15) allowing a robust staggered scheme for the numerical resolution. In this approach, the minimization of the energy functional is performed at a frozen damage state for the phase-field (i.e. $\mathbf{d} = cte$) leading to solve a ‘classical’ elastic problem:

$$\bar{\mathbf{v}} \cdot \bar{\boldsymbol{\sigma}}(\bar{\boldsymbol{\varepsilon}}, d) = 0 \text{ for the domain } V \text{ with } \bar{\boldsymbol{\sigma}} \cdot \bar{\mathbf{n}} = \bar{\mathbf{t}} \text{ on } \partial V \quad [1]$$

Where $\bar{\boldsymbol{\sigma}}$ and $\bar{\boldsymbol{\varepsilon}}$ are the stress and the strain tensor respectively. The term $\bar{\mathbf{t}}$ is the applied force and $\bar{\mathbf{n}}$ is the outward normal to the domain surface ∂V .

Then, the resolution at a given loading (i.e. $\bar{\mathbf{u}} = cte$) yields the equation for the phase field computation when the crack propagates:

$$2(1 - d)\mathcal{H} = G_c \left(\frac{d}{\ell} - \ell \Delta d \right) \quad \text{with } \mathcal{H}(\bar{\mathbf{x}}, t) = \max_{s \in [0, t]} \Psi^+(\bar{\mathbf{u}}(\bar{\mathbf{x}}, t)) \quad [2]$$

where G_c is the critical energy release rate of the material. The term \mathcal{H} is referred as an ‘history’ loading field taking into account the damage process irreversibility (15). It is expressed as the maximum of the energy component denoted Ψ^+ . This term comes from the decomposition of the elastic strain energy into a component due to tension (i.e. Ψ^+) and a component due to compression (i.e. Ψ^-).

Evaluation of the Model Relevance to Study the Fracture in Porous Microstructure

It has been shown by many authors that the PFM model is able to simulate accurately the propagation, the branching and the multi-cracking mechanisms in complex geometries (16). Nevertheless, there is still a controversy on the PFM capacity to accurately predict the crack nucleation. Indeed, it has been proposed that the length scale parameter ℓ could be linked to the Irwin length and thus related to the physical properties controlling the crack initiation (i.e. fracture strength and toughness) (17-19). In this condition, the PFM approach should constitute a relevant method to model the crack nucleation. However, other authors have claimed that ℓ is just a numerical parameter without any physical meaning (20). In this context, a special attention has been paid to interpret the real nature of ℓ and to evaluate the model capability to accurately predict the crack nucleation. For this purpose, the PFM simulations have been compared to the results given by the validated mixed criterion proposed by Leguillon (21). This part of the study has been conducted for two different configurations: an ideal V-notched sample and a crack blunted by a cavity. Afterwards, the model has been applied to simulate the crack initiation and propagation in representative porous microstructures. It is worth noting that all the simulations have been carried out in 2D plane strain assumption considering a symmetrical three-point bending test.

V-notched Sample. The geometry of the implemented V-notched sample with different opening angles 2β is depicted in Fig. 7a (i.e. $\beta \in \{30^\circ, 45^\circ, 60^\circ\}$). In this case, the crack is initiated from the stress singularity induced by the V-notch tip. A typical force-displacement curve simulated with the PFM is shown in Fig. 7b. It can be noted that the force is progressively increased up to reach a critical loading P_c corresponding to the fracture initiation. The apparent fracture toughness K_{Ic}^{app} is then calculated from the maximum loading using the following equation (22):

$$K_{Ic}^{app} = \left(\frac{3P_c L}{2bh^2} \right) h^{(1-\lambda)} f\left(\frac{a}{h}\right) \quad [3]$$

Where $f\left(\frac{a}{h}\right)$ is a dimensionless factor taken from (22), b is the beam thickness fixed to the unity for the 2D simulations and λ is the singularity order calculated using the equation reported in (22). The evolution of the fracture toughness deduced from the PFM simulations has been evaluated for the 8YSZ material as a function of the opening angle. The results have been compared to the prediction given by the mixed criterion in (Fig. 7c). It can be seen that the two curves are nearly perfectly superimposed. This result has been obtained with an *ad hoc* value of the length scale parameter denoted ℓ_{8YSZ} . The PFM can thus reproduce the increase of the apparent fracture toughness with increasing the opening angle. This evolution involves a change in the criterion controlling the initiation (21). Indeed, for small opening angles (i.e. $\beta = 30^\circ$), the crack nucleation is mostly governed by an energy criterion whereas both energy and stress criteria are involved for larger angles. It can be noticed that the same study has been performed considering the 3YSZ material (paper under preparation). In this case, it has been found that the evolution of the apparent fracture toughness with the notch-opening angle can be also retrieved by the PFM method considering a length scale parameter for the 3YSZ (ℓ_{3YSZ}). Besides, a relation has been highlighted between these above mentioned parameters (i.e. ℓ_{8YSZ} and ℓ_{3YSZ}) of the phase field and the Irwin characteristic lengths for the two materials. From this analysis, it has been shown that the PFM is able to predict the crack onset from a stress singularity.

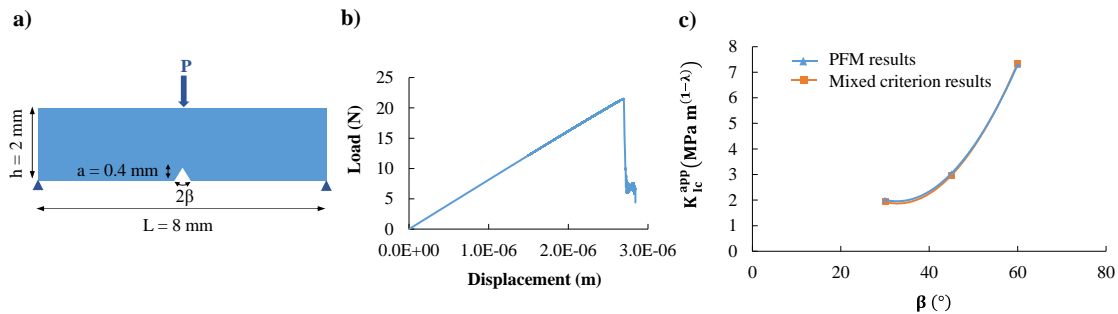


Fig 7. Three point bending test on a V-notched sample: a) implemented geometry b) Load/Displacement curve for 8YSZ material and $\beta = 60^\circ$, c) comparison between PFM and mixed criterion results for 8YSZ material.

Crack Blunted by a Cavity. The same analysis has been carried out by a crack blunted by a cavity of different radii (Fig. 8a). In this configuration, the crack is initiated from the stress concentration induced by the pore associated with the macro-crack. For this

geometry, the apparent fracture toughness K_{Ic}^{app} is related to the critical loading P_c calculated with the PFM method as follows (23):

$$K_{Ic}^{app} = \left(\frac{P_c}{b\sqrt{h}} \right) f \left(\frac{a}{h} \right) \quad [4]$$

Where $f \left(\frac{a}{h} \right)$ is a dimensionless factor taken from (23). The computed relative toughness (i.e. $R = K_{Ic}^{app}/K_{Ic}$) has been plotted as a function of the pore radius in Fig. 8b. It has been found that the PFM simulations are in good agreement with the predictions of the mixed criterion. Indeed, the PFM is able to retrieve the increase of the apparent toughness with the cavity size (24). This strengthening effect is due to the blunting of the crack leading to a relative toughness ratio R higher than unity. All these statements show that the implemented PFM model is also able to predict the crack nucleation from a stress concentration. It is worth noting that these results have been obtained for a value of ℓ different from the one used in the configuration of the V-notched sample. Therefore, this parameter is not only dependent on the material properties but also on the local geometry where the crack initiates (paper under preparation).

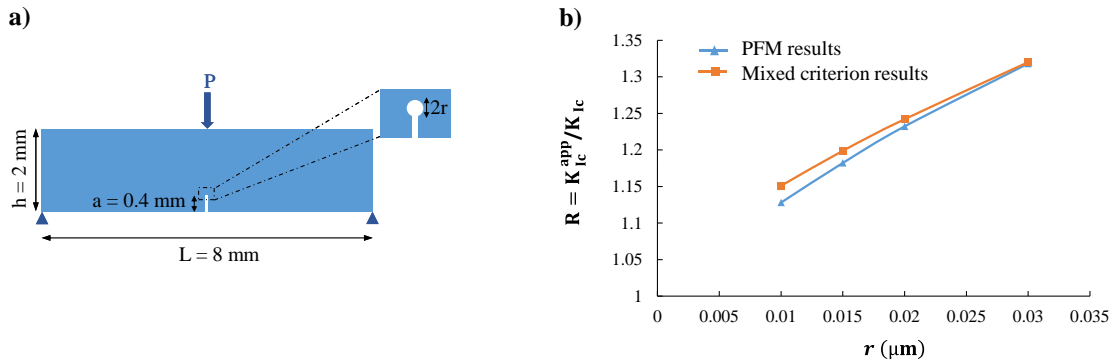


Fig 8. Three point bending test on a sample with a crack blunted by a cavity: a) implemented geometry, b) comparison between PFM and mixed criterion results for 8YSZ material.

Crack Initiation in Porous Microstructures. In complementarity with the previous ideal geometries, the capacity of the PFM to simulate the fracture in porous microstructures has been investigated. For this purpose, the simulated domain is described by a porous medium as depicted in Fig. 9a. The microstructures have been generated using the Gaussian random field model. Indeed, this method provides numerical 3D microstructures that mimic accurately the morphology of the real electrodes (25). In this study, 2D slices with similar microstructural characteristics (i.e. same phase volume fraction and characteristic length) have been extracted from the generated 3D volume. In order to address the impact of porosity on the fracture toughness, microstructures with different porosities $\varepsilon \in \{10\%, 20\%, 30\%\}$ have been generated. The study has been conducted for the 3YSZ material for which experimental data (i.e. fracture toughness with the porosity fraction) are available in the literature (26). It can be noticed that the relation [4] has been used to express the apparent toughness as a function of the critical loading calculated with the PFM. The evolution of K_{Ic}^{app} with the porosity is shown in Fig. 9b. It can be noted that the phase field predictions capture the decrease of the

apparent fracture toughness with the increase in porosity. Moreover, a slight variation of the predicted K_{Ic}^{app} has been found for the two simulated slices taken from the same 3D microstructure (Fig. 9b). This result is explained by the local variation of the computed microstructure at the crack tip. Moreover, a quite good agreement has been found with the experimental data. Indeed, even if the predicted toughness is higher than the experimental one, the evolutions of the two curves with the porosity are similar. The difference in the absolute value of the measured and simulated toughness could be explained by the 2D assumption and by the synthetic microstructures, which are not exactly the same than the tested ones. Nevertheless, from this analysis, it can be concluded that the PFM model is well adapted to study the fracture in the porous microstructures.

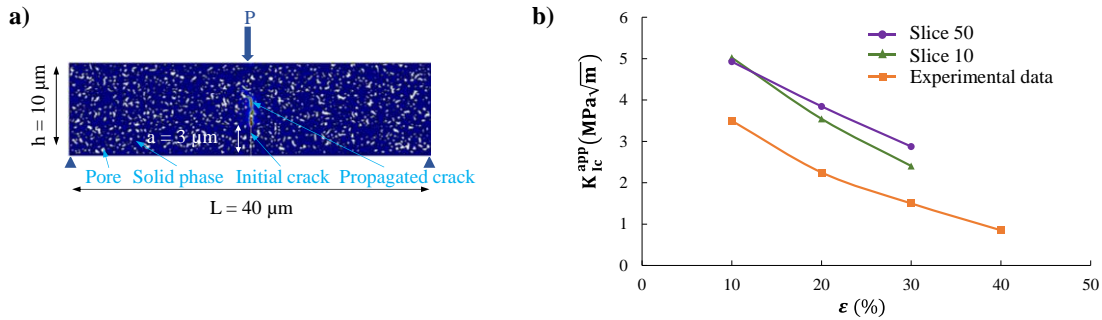


Fig 9. Three point bending test on a porous microstructure: a) implemented geometry, b) the evolution of the apparent fracture toughness with the porosity for two different slices of the generated 3D microstructures.

Multiscale Approach and Model Validation

For a complete validation of the phase field method, the experiments reported in the previous section have been modelled using a multiscale approach. For this purpose, the compression test has been simulated in 2D taking advantage of the axisymmetric geometry of the micro-pillars (Fig. 10a). An equivalent homogenous medium has been considered for the simulations for which the effective elastic properties have been computed by homogenization on the 3D reconstructions (27). The macroscale simulations for the whole micro-pillars provided the displacements, which have been applied as boundary conditions on the edge of a 3D microstructure (Fig. 10b). It can be noticed that the simulated volume is located in the upper part of the pillar where the stresses are the highest due to the slightly tapered shape of the pillar. The distribution of the principal stresses in the microstructure is given in Fig. 10c for an applied uniaxial compressive displacement on the micro-pillar of 2.8 μm . As expected, part of the YSZ network is submitted to non-negligible tensile stresses (Fig. 10b). Therefore, these regions constitute zones where the cracks are liable to nucleate. To explore this possibility, the PFM model has been applied. Preliminary results have shown that the method is able to reproduce the decrease of the compressive fracture strength with the porosity as experimentally observed in Fig. 7. Therefore, the phase field model appears as a relevant numerical tool to study the fracture in the electrode 3D microstructure.

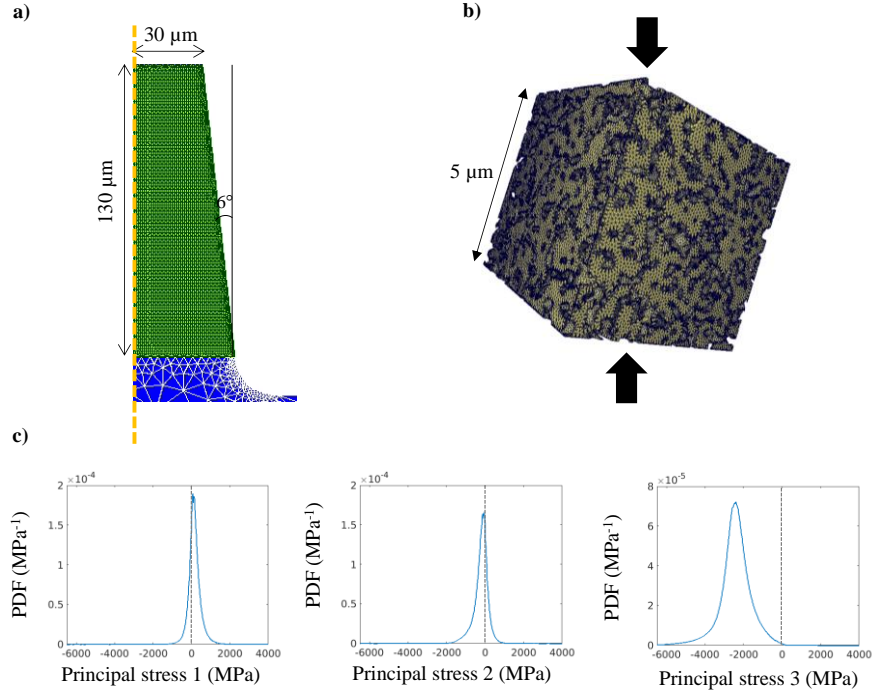


Fig10. Multiscale approach: a) axisymmetric geometry of the micro-pillar, b) porous YSZ microstructure under compression, c) probability density function (PDF) of the principal stresses.

YSZ Fracture during Ni Re-oxidation

After the PFM model validation, the local damage within the YSZ backbone induced by the Ni re-oxidation has been addressed. For this purpose, the initially implemented model has been extended to take into account the Ni expansion during re-oxidation. In this objective, the Ni swelling has been simulated considering an analogy with a thermal dilatation as follows:

$$\bar{\epsilon}_{th} = \alpha_{th}(T - T_{ref}) \quad [4]$$

where α_{th} is the thermal expansion coefficient for Ni. The temperature difference $(T - T_{ref})$ has been chosen in such a way that the thermal strain $\bar{\epsilon}_{th}$ can reproduce the Ni volume expansion. It is worth noting that the coefficient for YSZ has been set to zero to avoid the dilation of this phase during the simulation. Besides, it has been shown in (3) that the complete re-oxidation of the Ni is reached for a volume increase of 70%. Thus, a degree of Ni re-oxidation can be affected to each Ni volume expansion. In this study, PFM simulations have been conducted on a Ni-YSZ volume of $2 \mu\text{m}^3$ (Fig. 11). It has been found that the first crack is created in the YSZ network when the degree of Ni re-oxidation reaches a critical value of around 11% (Fig. 11). These results mean that the damage in the ceramic occurs almost at the early stage of the Ni re-oxidation. Therefore, even before the catastrophic rupture of the electrolyte in the cell (triggered for a degree of re-oxidation of around 60-65% at 800°C (28)), the cermet is subjected to an internal damage that will decrease the cell performances. Finally, it can be observed in Fig. 11 that the first crack has been generated into a thin YSZ ligament surrounded by Ni. This ligament was thus submitted to a high tensile stress during the re-oxidation. Therefore, the local fracture in the YSZ is highly sensitive to the local morphology of the cermet.

From this point of view, the developed PFM approach is a powerful method to analyze the role of the microstructure on the electrode robustness.

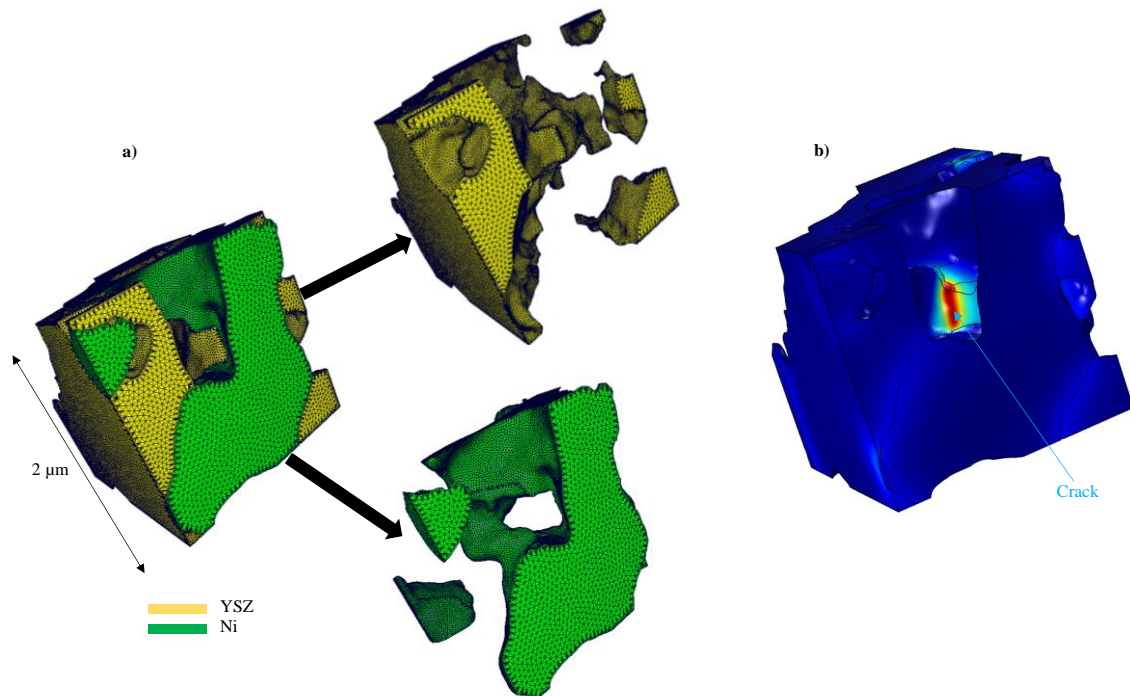


Fig 11. Simulation of the fracture induced by the Ni re-oxidation: a) 3D reconstruction of a Ni-YSZ cermet, b) visualization of the resulting crack.

Conclusion

Micro-compression tests have been performed on porous YSZ micro-pillars tested over a large range of porosities. It has been found that the compressive fracture strength decreases with increasing the pore volume fraction. The tested samples have been characterized to assess the type of damage in the microstructure. A transition has been observed from a brittle behavior at low porosity (below 50%) towards a local damage at high porosity. All these experimental data have been used to validate a numerical model based on the Phase Field Method (PFM) to simulate the local damage in the 3D microstructure. The relevance of this model to accurately predict the crack nucleation in porous material has been previously confirmed with theoretical considerations. It has been shown that the simulations with the PFM model are in good agreement with the theoretical prediction given by the mixed criterion. In addition, it has been verified that the PFM model is able to accurately simulate the expected decrease of the fracture toughness with the porosity increase. As a final validation, it has been shown that the evolution of the experimental compressive fracture strength with porosity is well captured by the numerical model. The PFM approach has been then applied to study the local rupture in the YSZ backbone induced by the Ni re-oxidation. It has been found that the first cracks can be generated even for a very low degree of Ni oxidation.

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