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Experimental set up for the mechanical characterization of plane ITM membrane at high temperature

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ABSTRACT

Mixed ionic and electronic conductors (MIEC) are promising materials for ion transport membrane (ITM) based technologies. A reliable design of these membranes requires a better knowledge of their macroscopic mechanical properties. These properties are linked to the composition of membrane materials, but also in a large part to the manufacturing process. Then, the mechanical characterization set-up must allow using specimen very similar in term of manufacturing process as the final parts in. The use of the diametric compression test on thin plates is investigated here, combined with full-field measurements. Guideline for sample size, solution for classical artifact and a dedicated I-DIC algorithm are proposed. Then, Young modulus and tensile strength of seven MIEC compositions of the lanthanum-ferrite perovskite series ($La_{(1-x)}Sr_xFe_{(1-y)}Ga_yO_{3-\delta}$, $La_{0.5}Ba_{0.5}Fe_{0.7}Co_{0.3}O_{3-\delta}$ and $La_{0.5}Ca_{0.5}Fe_{0.7}Co_{0.3}O_{3-\delta}$) were determined at room temperature and at 900 °C.

1. Introduction

Many breakthrough technologies such as ion transport membrane (ITM) for gas separation and SOFC for electricity production, require ceramic membranes with high mechanical reliability under working conditions. It is often one of key points for the scale up to the industrial application. The sealing systems of membranes and other mechanical constrains, such as thermal or chemical induced expansion of the material can lead to failure and finally membrane breakage [1–6]. So, to membrane material selection a better knowledge of the mechanical properties (i.e., elasticity, strength, creep,. . .) is necessary. The mechanical properties required for a reliable prediction are not the "intrinsic properties" of the material but those of the final parts [7]. Then, the scale-up of this technology requires the development of an experimental set up adapted to the final shape of the membrane in order to obtain meaningful data in sense of numerical prediction. Today, for ITM application, the two main geometries are tubular and planar membranes. For tubular membranes, C-ring test permit to access quite easily to Young modulus, tensile strength and fracture toughness [8]. Two other types of test, namely the O-ring tests [7] and the four points bending test on half-tubes [9], can also be used to characterize the mechanical properties of tubular membranes. For planar membranes, usual tests are not fully satisfactory.

For thin plates, the review of Wei et al. [7] reported the main mechanical tests and some of their limitations. The suitable tests are the biaxial bending tests. The sample specimens usually have circular shape, with a ratio thickness to diameter lower than 0.5. Four different biaxial bending tests could be reported in the literature: "Ring and Ring" [10–12], "Ball and Ring or Miniaturized Disk Bend Test" [13–15], "Ball and Three Balls" [16–18] and "Disk under pressure and Ring" [14,19]. This last one, which is seldom used, has not been treated by Wei et al. [7]. The "Ring and Ring", "Ball and Ring" and "Ball and Three balls" present a large scattering of values, because of the too little volume submitted to the load [14]. Moreover, the contact between the specimens and the punch has a huge influence on the estimated strength. Indeed, the stresses on the contact are defined by the Hertz theory [16,17] and so, the usual estimations of stresses from bending theory are no more suitable if the failure is initiated by shear stress under the contact. To bypass this difficulty, the punch could be substituted by a fluid and an elastomer film that applies a pressure on one face of the specimen. This test corresponds to the "Disk under pressure and Ring". For all these tests, the stresses are determined thanks to analytical expression linked to geometrical parameters and load in accordance to plates and shells theory. In order to estimate the elastic properties, the deflection in the center of the specimens is recorded by a linear transducer (LVDT). This measurement is

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local and strongly depend on the position of the transducer. Then the elastic properties and ultimate strength estimated from these data are strongly influenced by local default and the position of transducer. It is worth to note that none of these tests is used to characterize creep. Classically creep is studied by compression test on pipe [7]. State of art shows that there is no usual creep test for planar geometry. Finally, one of the key challenges is the development of new mechanical tests dedicated to planar membrane characterization that allows identifying elastic parameters, yield strength with a lower scattering, and that can be suitable for creep test. In this paper, a particular attention is given to the description of set up proposed with post-treatment method, but without considering the effect of atmosphere on mixed ionics electronics conductors (MIEC) which are the support of these developments.

The technics of full-field measurement allow overcoming the problem of local measures and local effects. The main advantage of the full-field measurement by optical set up is that there is no contact with the specimen. The Digital Images Correlation (DIC) and integrated DIC (I-DIC) have been developed for more than 10 years now in the field of material mechanics [19]. These methods give access to non-local properties and offer a new promising way for creep characterization and other non-linear mechanical behavior at high temperature [21]. The main problem is the accuracy of strain measurement which is usually around 10^{-4} [22] regard to 10^{-6} by classical strain gauge. For ceramic materials, like MIEC membranes dedicated to ITM application, strains are in the range of 10^{-5} for the elastic part. Then, a dedicated method and algorithm are proposed hereafter to catch low displacements.

To be able to use such method on membrane specimens, one face has to be observed by a camera. One solution is to perform a diametric compression of membrane disc. This configuration is far from standardized Brazilian test for concrete with cylindrical specimen presenting a ratio thickness to diameter superior to 2. However, lower ratio can be used thanks to adapted analytical and numerical post-treatment [23].

In what follows, first, the experimental set-up proposed for the diametric compression of thin disc from room temperature (R.T) to 900 °C is detailed. The image acquisition and treatment are explained and the dedicated algorithm too. Then, the validation of the whole method is done thanks to separate validation of the algorithm and the mechanical set up. The accuracy for Young modulus and tensile strength identifications are then estimated. Finally the test is used to characterize seven MIEC compositions of the lanthanum-ferrite perovskite series ($La_{(1-x)}Sr_xFe_{(1-y)}Ga_yO_{3-\delta}$, $La_{0.5}Ba_{0.5}Fe_{0.7}Co_{0.3}O_{3-\delta}$ and $La_{0.5}Ca_{0.5}Fe_{0.7}Co_{0.3}O_{3-\delta}$) at R.T. and at 900 °C under air environment.

2. Experimental setup

The diametric compression of disc leads to compression stresses along the vertical axis and tensile stresses in the perpendicular direction (Fig. 1). Considering brittle materials with lower tensile strength than compressive strength, the failure occurs along the loading axis where extension takes place. Direct estimate of the tensile strength is not possible from the maximum load before failure and the geometry. However, tensile and compressive behaviors are obtained from the recording of the complete displacement field. However, the post treatment of the test presents many pitfalls.

The first plight concerns the tensile strength. Indeed, the failure can be initiated by shear stresses at the contact area [24]. To avoid the failure at the contact area, the slipway between loading plates and sample must be carefully chosen according to their stiffness at the temperature of the test. Whatever the slipway, the fracture surfaces have to be conscientiously observed to be sure of the origin of breakage. The second plight is linked to the setting position which allows solid rigid body motion of the sample. This point will be corrected thanks to numerical post-treatment during the Digital Image Correlation [23]. The last mechanical difficulty concerns the risk of buckling of the sample, this risk can be estimated following hereafter reasonning.

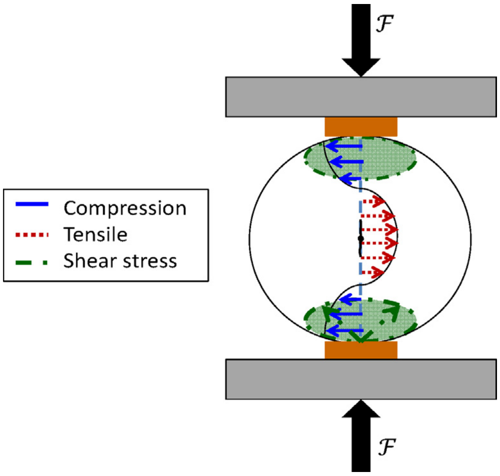

Fig. 1. theoretical stress field along vertical axis for diametric compression.

In case of rectangular thin plate with a fixed width, the lowest critical buckling load is obtained for square plate [25]. Assuming that circles are stiffer than squares, a low bound of the critical load for diametral compression can be estimated from the circumscribed square [25]:

$$F_b \approx \frac{\pi^2 E e^4}{3(1-\nu^2)D^2}$$

where E is the Young modulus, ν the Poisson ratio, e the thickness and D the diameter of the disk. If there is no buckling, considering plane stress assumption, the tensile stress σ_r that initiate the diametral breakage in agreement with Rankine criterion can be linked to the load of rupture F_r using the analytical solution of the diametral compression [26]:

$$\sigma_r = \frac{2F_r}{\pi e D}$$

Then, to ensure a diametral breakage, the sample design must ensure that the critical buckling load is higher than the diametral breakage load. This condition leads to:

$$\frac{e}{D} > \sqrt{\frac{3(1-\nu^2)}{2}\frac{\sigma_r}{E}}$$

The thickness to diameter ratio is equal to 0.04, with a disc of 25 mm in diameter and 1 mm in thickness. From mechanical properties of usual ceramics with Young modulus from 50 GPa to 200 GPa, and tensile strength from 10 to 100 MPa, the right part of the inequality is in the range 0.005 to 0.029. So, there is no risk of buckling for thin disk of 25 mm in diameter and 1 mm in thickness if samples are flat. The risk of buckling increases with shape's default. Empirically, an out of flatness close to 400 μm is critical.

The others plights are linked to the optical acquisition for Digital Image Correlation. In addition to usual precaution for such measurement [20,27], the high temperatures add some specific optical artifact: convective air movement, heat haze and radiation fluctuation induce by thermal resistance of furnace. Two of these artifacts are herein be by-pass thanks to set-up design and the last one is corrected by embedded routines in the Digital Images Correlation.