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Review paper

Review of mechanical characterization methods for ceramics used in energy technologies

J. Wei, G. Pećanac, J. Malzbender*

Forschungszentrum Jülich GmbH, Institute for Energy and Climate Research, IEK-2, 52425 Jülich, Germany

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Abstract

The reliability and robustness of advanced electrochemical devices depends critically on the thermo-mechanical properties of the ceramic components. In addition to their elastic behavior, fracture characteristics are important due to the inherent brittleness. Furthermore, the elevated temperature operation raises questions with respect to viscous and creep deformation as well as the potential failure due to creep rupture. It is outlined how considered aspects can be assessed for sealant and ceramic cell materials that are typically used for solid oxide fuel/electrolysis cells and oxygen transport membrane materials. Particular emphasis is directed towards a discussion of strengths and weaknesses of available methods. Considered are micro-mechanical methods that yield information on the local properties and macro-mechanical methods being representative for the global behavior of the materials. An outlook on the choice of mechanical analysis methods is given.

Keywords: C. Mechanical properties; C. Fracture; Oxygen transport membranes; Ceramics; Solid oxide fuel cells; Sealants

Contents

*Corresponding author. Tel.: +49 2461 616964; fax: +49 2461 613699.
E-mail address: J.malzbender@fz-juelich.de (J. Malzbender).

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1. Introduction

The interest in electrochemical devices has increased in recent years [1], in particular solid oxide fuel cells (SOFCs) are at the edge of commercialization [2]. In order to increase performance, thin layer concepts became increasingly the focus of studies, where the electrochemical active layer is mechanically supported by a porous substrate [2]. Since these devices operate at elevated temperatures where, in addition to long term chemical stability [3,5], creep deformation and thermo-mechanical stability are crucial parameters for sustainable operation [6,8], ceramic materials appear to be most suitable for this purpose [9].

However, basic problems such as reliability and robustness [10] are always an issue for brittle ceramic materials. Due to their favorable transport properties, materials which are used as components in solid oxide electrolysis cells and as mixed ionic electronic conducting cathodes in SOFCs also found meaningful interest for the use as oxygen transport membranes [11], which emphasizes the basic need to understand and characterize necessary mechanical parameters.

Whereas the active layers can fail due to overloading by thermal or chemical strains, critical materials with respect to the long term mechanical reliability are porous substrates and sealant materials [12,13]. Specimen preparation and testing are rather straight forward for metallic materials, however, the brittleness of ceramic materials imposes limitations onto useable methods for these kind of materials [5]. Several promising substrate materials have been well studied so far [14,6,7,15,16,5,8], however, new compositions, designs and operating conditions require further investigations. Hence, whereas micro-mechanical properties can be extracted via indentation techniques [17], the macro-mechanical characteristics are usually assessed using bending methods [18,19], yielding the elastic and fracture characteristics. Since design can be planar and tubular, testing methods for characterization of the latter type of geometry have also to be discussed.

Note that the rather low tensile strength of the ceramic materials along with the associated fabrication and clamping difficulties renders very often bending tests to be the only alternative to assess the macro-mechanical fracture behavior. The thereby assessed characteristic fracture strength, along with the Weibull modulus, can be used to determine failure probabilities under a given stress situation [20] and hence can be used as guidelines for the component design which is often supported by finite element simulations.

The sealing of SOFCs is typically reallized using glass–ceramic materials [21], which have a superior chemical stability compared to metallic sealants. Although being advantageous with respect to their stress relaxation potential, metallic sealants have shown to be especially problematic under hydrogen containing SOFC dual atmosphere exposure that lead to pore formations and eventually rupture [22]. For the use in OTMs they appear to be less problematic [23].

The use and limitations of mechanical testing methods for ceramics typically used in the outlined applications is discussed in the following and different testing methods are compared. Necessary geometrical restrains onto available testing methods are discussed, since typical design rely on either planar or tubular components, i.e., tubular designs can be disadvantageous for SOFC [24], but advantageous for oxygen transport membranes [25].

2. Indentation testing

2.1. Elastic modulus and hardness

In the case of instrumented indentation testing, elastic modulus and hardness are usually determined from the load–depth curve [26] following the procedure derived by Oliver and Pharr [27]. The applied loads range typically from below the mN range up to a few N leading to rather small μm range displacements. Although Vickers tip indentation is the most common test, other indenter tips like Berkovich, Rockwell, Knoop or Shore can also be used leading to slight differences in the analysis procedure [28].

The elastic modulus is calculated from the unloading curve, which represents elastic response of the material. The hardness is derived from the maximum load and the corresponding contact depth. The basic importance of the elastic modulus is that it defines the elastic behavior of the material, which makes it a key input parameter for analytical and numerical calculations that link loads (strains), stresses and deformation.

Typically the hardness corresponds to a plastically deformed volume (in case of ceramics it can also be a volume of micro-cracked material). The elastically tested volume is approximately ten times larger than the plastic zone. Hence for low loads the properties correspond more to that of a single grain, whereas at higher loads also the effects of grain boundaries and pores are considered [29]. Especially at low loads the test is strongly affected by the specimens' roughness leading to rather large uncertainties in the obtained data [30].

The general advantage of the indentation test is that it is a fast serial test and only a small specimen volume is required, so macroscopically it can be considered as a non-destructive test. The disadvantage is that the properties can be representative only for the location where the test is carried out.

Elastic properties and hardness values for materials typically used in energy applications are given for example in [31]. Hardness impressions can lead to additional effects like formation of slip planes as exemplified in Fig. 1 [16], that also affect the measured properties due to the associated displacement [32].

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