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Short communication

Long-term thermal cycling of Phlogopite mica-based compressive seals for solid oxide fuel cells

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Abstract

Planar solid oxide fuel cells (SOFCs) require sealants to function properly in harsh environments at elevated temperatures. The SOFC stacks are expected to experience multiple thermal cycles (perhaps thousands of cycles for some applications) during their lifetime service in stationary or transportation applications. As a result, thermal cycle stability is considered a top priority for SOFC sealant development. In previous work, we have developed a hybrid mica-based compressive seal with very low leak rates of $2–4 \times 10^{-2}$ to 10^{-3} sccm cm^{-1} at 800 °C, and showed stable leak rates over limited thermal cycles. In this paper we present results of long-term thermal cycle testing (>1000 thermal cycles) of Phlogopite mica-based compressive seals. Open-circuit voltage (OCV) was measured on a 2 in. × 2 in. 8-YSZ plate with the hybrid Phlogopite mica seals during thermal cycling in a dual environment (2.75% H_2/Ar versus air). During two long-term cycling tests, the measured OCVs were found to be consistent with the calculated Nernst voltages. The hybrid mica seal showed excellent thermal cycle stability over 1000 thermal cycles and can be considered a strong candidate for SOFC applications.
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Keywords: Open-circuit voltage; Phlogopite mica; Solid oxide fuel cell; Leak rates; Thermal cycle

1. Introduction

Planar solid oxide fuel cells require a special sealant or sealants in order to function properly at elevated temperatures in the SOFC environment, which involves exposure to both oxidizing (air) and reducing (fuel) atmospheres. The sealant needs to provide zero or low leak rates to avoid direct mixing of the fuel and oxidant gases or leakage of fuel gas from the stack. It has to demonstrate long-term thermal and chemical stability in the SOFC environments (5000 h or more). Finally, it has to survive multiple thermal cycles (possibly thousands of cycles for some applications) during lifetime service in stationary or transportation applications. To do so, it has to be able to withstand transient stresses developed during startup or shutdown, and residual stresses due to mismatch in thermal expansion of different SOFC stack components. Currently, there are three primary approaches for SOFC seal development: rigid glass (or glass-ceramics and glass fiber composite) seals [1–5], metallic brazes [6,7], and compressive seals [8–12]. Among these studies, none has investigated the long-term thermal cycle stability (i.e., hundreds of cycles or more). As the thermal cycle stability appears to be a top priority for SOFC seal development, this paper reports the results of long-term thermal cycle testing of compressive mica seals. This study is a continuation of previous work on the "hybrid" mica compressive seal which has exhibited very low leak rates compared to conventional mica gasket seals [6], and also demonstrated thermal cycle stability over a limited number of thermal cycles. In this paper, we present results for two long-term thermal cycle stabilities of two "hybrid" mica seals. Open-circuit voltage (OCV) was used to characterize the thermal cycle stability of these mica seals using a standard 8-YSZ electrolyte plate. In addition, leak rates were measured and compared to the estimates calculated from OCVs.

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2. Experimental

2.1. Materials and processing

The mica used in this study is a commercially available Phlogopite mica paper (McMaster-Carr, Atlanta, GA). The mica paper is composed of discrete mica flakes overlapping with each other [12]. The thickness is about 0.004 in. and the paper contains 3–5% of organic binders. In this study, two "hybrid" mica seals (i.e., mica paper sandwiched between two glass layers [8]) were tested for long-term thermal cycle stability. The glass layers were made by tape casting of a Ba–Al silicate glass developed at PNNL for SOFC sealing applications. The cast glass tapes had a thickness of about 0.015–0.020 in. after drying. The compressive stresses applied to the seals were 100 psi (sample #1) and 50 psi (sample #2). The compressive load was applied using a pneumatic cylinder and compressed air.

2.2. Open-circuit voltage test

In order to assess the sealing capability of the seals, OCV tests were conducted using 2 in. × 2 in. dense 8-YSZ plates prepared by slip casting 8-YSZ powders (TOSOH, Zirconia, TZ-8Y, Japan), followed by sintering at 1450 °C for 2 h. The sintered plates were machined to the desired size (2 in. × 2 in.) and thickness (∼1–2 mm), and then screen-printed with silver paste on both sides. After electrode sintering, Pt wire leads were connected for the OCV tests. A dense 8-YSZ plate was pressed between an Inconel600 top cap (2 in. ×2 in. with a wall thickness of 0.2 in.) and an alumina bottom support. The hybrid mica seals were placed between the 8-YSZ plate and the Inconel600 fixture. A schematic drawing of the OCV test fixture and the mica seal arrangement is shown in Fig. 1. The OCV measurements were conducted at 800 °C after dwelling at temperature for about 1.5–2 h. A low-hydrogen content gas (2.55–2.71% H_2/balance Ar with ∼3% H_2O) was used as the fuel with variable flow rates. Air was used as the oxidant on the cathode side with a flow rate of 100–200 sccm (standard cubic centimeter per minute). The samples were first fired slowly to 600 °C for binder burnout, followed by heating to 850 °C for 1 h and then cooling to the test temperature (800 °C) for 2 h. After the first dwell at 800 °C for 2 h, the samples were furnace cooled to 100 °C to initiate thermal cycling. The temperature profiles for the thermal cycling are shown in Fig. 2 for sample #2 (pressed at 50 psi). Sample #1 (pressed at 100 psi) was cycled with a different profile, i.e., rapid heating from 100 to 800 °C in

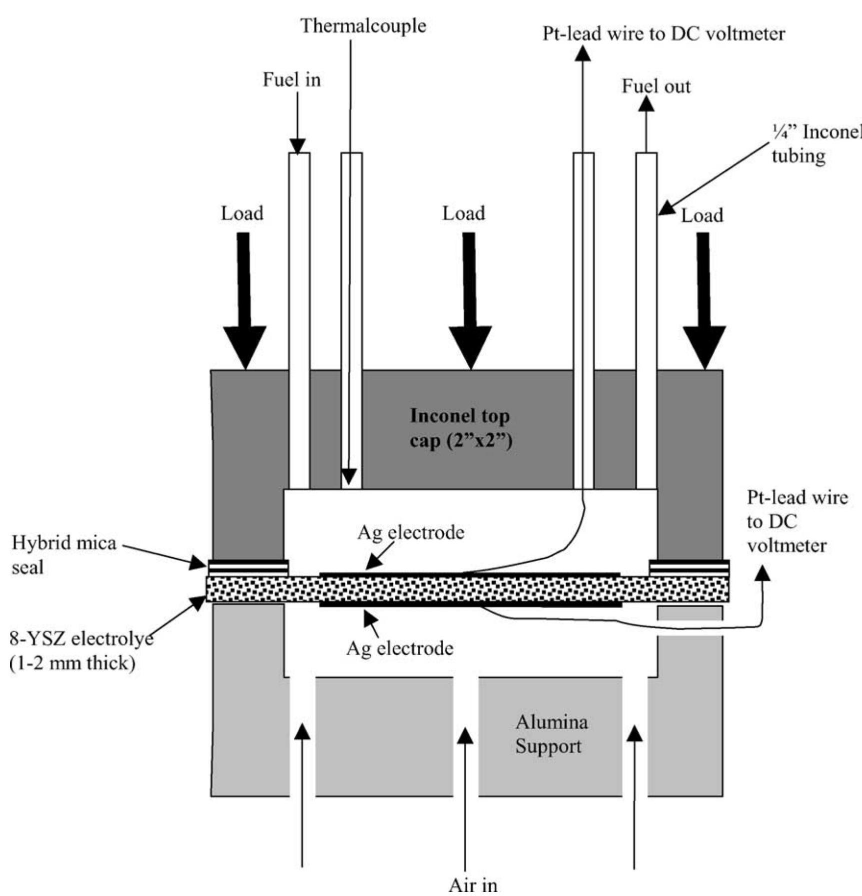

Fig. 1. Schematic showing the open-circuit voltage test fixture (2 in. × 2 in.) of dense 8-YSZ plate with compressive mica seals on the fuel side. The pressing cap was made of Inconel600 with four 1/4 in. Inconel600 tubes. The 8-YSZ plate was supported on an alumina block with three bottom holes for airflow.

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