Materials Science & Engineering A 585 (2013) 32–38

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

# Metallic sandwiches with open porosity facings and closed porosity cores for SOFC interconnects

Justin A. Scott [1], David C. Dunand *

*Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208, USA*

ARTICLE INFO

*Article history:*
Received 6 January 2013
Received in revised form
21 June 2013
Accepted 24 June 2013
Available online 20 July 2013

*Keywords:*
Ferritic steels
Nickel alloys
Fuel cells
Porous materials
Mechanical characterization
Powder metallurgy

ABSTRACT

Iron- and nickel-based sandwich structures with open porosity facings and closed-porosity cores were created by melt infiltration and powder metallurgy, respectively, for application as interconnects in high-temperature fuel cells. For E-Brite (Fe–Cr–Mo) sandwiches, open porosity faces were created by evaporation of NaCl particles mixed with the metallic powders, while closed porosity in the core resulted from partial sintering of the pure metallic powders. Sandwiches from J5 (Ni–Mo–Cr–Ti–Mn–Al–Y) were produced by infiltrating the liquid alloy into a sandwich scaffold of permanent $Al_2O_3$ hollow spheres in the core and leachable $NaAlO_2$ particles in the facings. Mechanical properties of both sandwich types were measured in three-point bending and indicated similar modes of failure by face yielding. Stiffness measurements closely match model values for E-Brite sandwiches but are below expected values for J5 sandwiches. In the case of sandwich yield load, calculated values for E-Brite slightly underestimated experimental values, while J5 experimental performance was significantly overestimated.



## 1. Introduction

Metallic foams display many properties that make them attractive for use in lightweight structural applications [1–3]. Their combination of low density, high energy absorption, and high specific strength and stiffness make them well-suited as elements for sandwich panels [4–6]. Metallic foams are typically employed as the core layer sandwiched between two thin, dense alloy sheets [7]. Mechanically, this configuration can offer significant benefits for weight-optimized bending stiffness [8–12] and, in some cases, even yield-limited design as demonstrated with functionally graded Al-based foams [13,14]. To date, however, fully porous sandwiches, with open-porosity facings sandwiching a closed-porosity core, have not been studied, neither in terms of fabrication methods nor mechanical properties.

Planar solid-oxide fuel cells (SOFC) are typically connected in series into stacks, with interconnects providing electrical connectivity across cells and physical separation between the fuel at the anode-side of one cell from the air at the cathode-side of the adjacent cell. One of the most common interconnect designs consists of a conductive metallic plate with channels on each side of the bipolar plate to allow for gas flow [15–18]. Some recent designs for SOFC interconnects utilize a sandwich structure with thick, open-porosity facings and a thin, pore-free core [19–21]. In this lightweight interconnect architecture, which is especially advantageous for mobile SOFCs applications, the open pores in the facings can serve as fluid channels for fuel and oxidant across the electrodes while the dense, pore-free core acts as a physical barrier between them [18]. It is also possible for the core of the interconnect sandwich to be a closed-porosity or syntactic foam layer instead of a pore-free material, which enables meeting the minimum strength and stiffness requirement of the interconnect with less weight/material. Since SOFCs operate at high temperature (typically at 700–800 °C), Fe-based chromia-forming alloys (i.e., ferritic alloys) are commonly considered the best candidate materials for SOFC interconnects due to their high oxide conductivity compared to alternative alumina- and silica-forming iron-based alloys [16].

Though less prominent in the literature, Ni-based alloys are also considered suitable for SOFC interconnects. While their coefficient of thermal expansion is not as well matched with ceramic SOFC components compared to ferritic steels, they offer better mechanical strength, which is advantageous in auxiliary power units for mobile applications where stacks may be subject to impact and vibrations. As only a few Ni-based alloys have been studied for SOFC applications, mostly in bulk form, limited information is available on the properties of porous Ni-based foams for SOFCs. Here, a replicated J5 foam studied previously

* Corresponding author. Tel.: +1 847 491 5370; fax: +1 847 467 6573.
*E-mail addresses:* jscott@u.northwestern.edu (J.A. Scott), dunand@northwestern.edu (D.C. Dunand).
[1] Present Address: The Minerals, Metals & Materials Society, 184 Thorn Hill Road, Warrendale, PA, 15086-7514, USA.

0921-5093/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.msea.2013.06.086

[22,23] is used for the facings while a thermoreversible gelcasting (TRG) technique is employed to create a syntactic foam core with J5 and hollow alumina spheres. TRG involves the formation of a reversible, physically cross-linked polymer gel that, after cooling to room temperature, can be reheated and re-casted. The process is well suited for complex shapes and the high aspect ratios of components in SOFC systems [24].

Here, we describe two new methods, based on casting (for a Ni-based alloy) and powder metallurgy (for a ferritic steel), to create light-weight sandwiches with a novel, fully porous structure: open porosity facings and closed porosity cores. Despite the critical importance of preventing deformation of interconnects in the performance of SOFC, there is a paucity of data on the subject of flexural properties. We thus examine the flexural properties of these sandwich beams with thicknesses relevant to SOFC interconnects. A better understanding of the bending behavior is expected to influence the design of the stack and help determine the limitations of mechanical loading in mobile applications.

## 2. Experimental procedures

### 2.1. Processing

Sandwich structures were created using two alloys designed for SOFC interconnects: E-Brite (Fe–26Cr–1Mo, wt%) and J5 (Ni–22.5Mo–12.5Cr–1Ti–0.5Mn–0.1Al–0.1Y, wt%). For both alloys, sandwiches used for mechanical testing consisted of facings with open porosity of ~50% surrounding a closed-porosity core.

#### 2.1.1. E-Brite sandwiches

E-Brite sandwiches were prepared using a powder metallurgy approach. Two batches of powder blends were prepared, similar to the methods described in Ref. [25]. Type E1 consisted of elemental Fe (APS 6–10 μm, 99.5% purity), Cr (APS < 10 μm, 99.8% purity), and Mo (APS 3–7 μm, 99.95% purity) acquired from Alfa Aesar (Ward Hill, MA). Powders were mixed in proportions corresponding to the E-Brite composition and blended with 50 vol% NaCl place-holder powders (crushed and sieved to 53–106 μm) to be used for producing the facings. For type E2, the elemental powders were mixed but no NaCl place-holder was added to create a denser core. A thin layer of E1 powders was poured into a 27.9 mm diameter steel die, spread flat, and hand-pressed with a steel punch to a height of ~0.6 mm. A second layer of E2 powders was added, spread flat, and hand-pressed with the punch as before, to a height of ~0.6 mm. A third layer of E1 powders (with the same mass as the first one) was poured, spread flat, and the entire compact was cold pressed at 350 MPa. The resulting compact was vacuum sintered at 1250 °C for 4 h, resulting in evaporation of the NaCl place-holder along with densification and interdiffusion of the metallic powders, as previously reported in Ref. [25].

Porosity of the facing was determined by Archimedes density measurements performed with water as the medium on a sample prepared solely with E1 powders, pressed and sintered under the same conditions as the sandwiches. Similarly, closed porosity of the E-Brite core was calculated by taking Archimedes density measurements in water on a separate sample prepared with only E2 powders. The same E-Brite bulk density of 7.7 g/cm$^3$ for E-Brite [26] was used in both cases.

#### 2.1.2. J5 sandwiches

Sandwich structures of J5 alloy were produced by infiltrating with liquid J5 a scaffold containing three layers with two types of place-holders.

The core, (J1), was first created via thermoreversible gelcasting (TRG) to obtain a flat beam that served as a permanent core scaffold. For this step, as-received hollow alumina spheres (ALODUR KKW, < 500 μm, 98 wt% $Al_2O_3$; Treibacher Schleifmittel; Andersonville, GA) were sieved, retaining those in the range 355–500 μm. These hollow alumina spheres were further sorted by suspension in chloroform. Since chloroform has a higher density (1.48 g/cm$^{-3}$) than the closed, hollow alumina spheres of the desired size ( < 1.40 g/cm$^{-3}$), these raised to the surface, while open spheres and fragments settled to the bottom of the becker. The closed alumina spheres retrieved from the surface were rinsed with acetone and dried before further processing. A TRG system was subsequently produced with 6.7 wt% triblock copolymer, poly(methyl methacrylate)–poly(n-butyl acrylate)–poly(methyl methacrylate) (PMMA–PnBA–PMMA; 9k–53k–9k g mol$^{-1}$; Kuraray, Japan) dissolved in 2-propanol, which preferentially solvated the PnBA midblock [24]. The mixture was sealed in a glass vial and sonicated in water heated to ~60–70 °C until the solution appeared a homogenous cloudy white indicating the transition to a low-viscosity solution via dissociation of the polymer end-blocks [27,28]. Solids, consisting of presorted, hollow alumina spheres (as described above) and alumina powders (0.36 μm median particle size; Baikowski Malakoff, Inc.; Malakoff, TX) in a ratio of 20:1 by volume, was then dispersed in the solution in two batches about 5 min apart. Finally, a small amount ( < 1 cc) of Aerosol AY-65 (Cytec; West Patterson, NJ) dispersant was added to stabilize the slurry. The warm slurry was then cast onto glass slides into circular steel washers (35 mm diameter opening, 1 mm thick) that were lubricated with vacuum grease. A second glass slide was placed on top of the washer and pressed down lightly to prevent cracking any of the hollow alumina spheres while flattening the slurry. Gelation rapidly occurred during cooling and the castings were further allowed to dry for 24 h in a fume hood. The cylindrical samples were then demolded and cut into ~10 × 30 mm$^2$ rectangles, which were heated (7 °C/min) in a vacuum furnace (~1 × 10$^{-6}$ Torr) for 1 h at 600 °C (to achieve copolymer burnout) and 1 h at 1525 °C (for sintering) before being furnace cooled.

The second type of placeholder used for faces (J2), consisted of sodium aluminate ($NaAlO_2$) powders (~45 mesh; Alfa Aesar; Ward Hill, MA), which were prepared similarly to a method outlined previously [22,23]. Since the as-received powders were too fine for this study, they were cold-pressed at 350 MPa and fired at 1500 °C for 1 h in air before crushing and sieving to the range of 355–500 μm.

With their longest dimension remaining vertical, three sintered J1 cores were placed upright in a 25 mm diameter alumina crucible, parallel to each other and each separated by a 5 mm gap. These beams formed the cores of three sandwich structures. Sodium aluminate powders were then poured in the remainder of the crucible to the height of the vertical beams filling the space between the cores and creating a preform filling the crucible. An ingot of J5 alloy, provided by the National Energy Technology Laboratory (Albany, OR) and sectioned into cubes of ~1 mm$^3$, was placed on top of the preform but separated by a 3 mm thick alumina disc spacer to prevent possible reaction between J5 and place-holders during heating. The crucible was heated at 7 °C/min in vacuum (~1 × 10$^{-6}$ Torr) to 1450 °C and maintained for 1 h at this temperature to ensure complete melting of the J5 ingot. Argon was then introduced to the system to a pressure of 80 kPa and the pressure was maintained for 3 min, causing the melt to flow through the gap between the crucible wall and the spacer and infiltrate the pores of the preform. The ingot was subsequently furnace cooled while maintaining the pressurized argon for 1 h, ensuring that solidification occurred under pressure.

The infiltrated composite was cut into three sections with a high-speed diamond saw, each containing a J1 core surrounded by J2 facing materials (infiltrated sodium aluminate). Further cutting