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# Dip coating fabrication process for micro-tubular SOFCs

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ABSTRACT

This study investigates the dip coating fabrication process in detail. First the slurry formulation development is presented. This includes optimizing the concentration of slurry components. Controlling the coating thickness is then addressed. It is shown that a broad range of uniform thicknesses from a few microns up to a few hundreds of microns is achievable by adjusting the solid loading and/or the dip coating rate. This study reports that dip coating is a commercially reliable technique to fabricate micro-tubular solid oxide fuel cells not only because it is low cost but, more importantly, because it is fairly reproducible and highly controllable.


## 1. Introduction

Due to the progressively growing demand for energy sources, tremendous attempts have been made to develop and commercialize both planar and tubular SOFCs in the past few years. Although it has been reported that tubular SOFCs have a lower theoretical performance in comparison with planar ones [1], they have unique geometrical advantages resulting in considerable thermal shock resistance and sealless design [1,2]. These benefits, in fact, are more pronounced when micro-SOFCs are considered. Not only do micro-tubular SOFCs offer superior resistance against thermal stresses caused by thermal cycling, they also have high volumetric power density. These characteristics make micro-tubular SOFCs virtually unrivalled for a variety of applications such as transportation, portable electronic devices and auxiliary power supplies [3–5].

One of the significant challenges in commercialization of micro-SOFCs is their fabrication process mainly due to the fact that reaching a compromise between manufacturing costs and product quality is always a major concern. Since the fabrication process could strongly affect cell performance [6], a highly controllable and reproducible technique is necessary. Extrusion in combination with dip coating (or slurry coating) is recognized as a satisfactory fabrication process [7–9] which not only meets the above mentioned requirement, but is also low cost. Although it has been dealt with by many researchers, the effect of various fabrication parameters on the coating characteristics has seldom been reported in detail, to the best of our knowledge. Therefore, the purpose of this study is to investigate the effect of dip coating technique parameters on the attributes of the coating.

## 2. Experimental procedure

For the sake of simplicity, electrolyte-supported cells were utilized in this study. These tubes were characterized as extruded YSZ (8 mol% $Y_2O_3$) with a length of 200 mm, an outside diameter of 4.5 mm and an inside diameter of 4.2 mm, made by Adelan Ltd. Fig. 1 shows a flowchart of the dip coating process. The slurry components include inorganic powder (NiO <45 μm, Alfa Aesar, Ward Hill, MA and YSZ <5 μm, Fuel Cell Materials Co., Lewis Center, OH), dispersant (Menhaden fish oil, Tape Casting Warehouse, Yardley, PA), binder (polyvinyl butyral (PVB), Tape Casting Warehouse, Yardley, PA) and solvents (azeotropic mixture of toluene and ethanol). It is noteworthy that 50/50 vol.% was chosen as the mixing ratio of the NiO–YSZ composite and the weight percent of both the dispersant and the binder were calculated based on the solid loading. To make a highly homogeneous slurry and to minimize breakdown of the high molecular weight PVB molecules, a two-step milling process was used. The inorganic powder, dispersant and solvent blend were first mixed for 1 h using a planetary ball mill (Retsch, PM 100). The binder was then added and the milling continued for one more hour. After milling, the slurry was put under vacuum in order for the dissolved gas to be eliminated. A vibro viscometer (Malvern, SV-10) was used to measure the viscosity of the slurries. Also, to compare the adhesion characteristic of the slurries, a standard method for measuring adhesion by a tape test (ASTM D3359-08) was utilized.

Fig. 2 illustrates the dip coating setup and a coated micro-tube. The coating was applied using a vacuum system created by a syringe pump. In this setup, when the vacuum is applied (withdrawal step),

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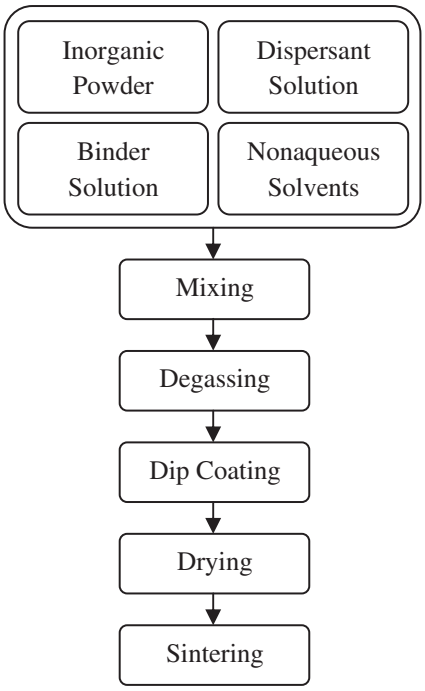


**Fig. 1.** Dip coating fabrication process.

the slurry is pulled up into the YSZ tube to coat the inside, or into a quartz support tube to coat the outside. The vacuum is then removed (infusion step) and the slurry leaves the surface of the YSZ tube. The coating was then dried at room temperature for 24 h and sintered at 1350 °C for 2 h. A scanning electron microscope (Hitachi S-2700) was finally used to characterize the fractured cross section of the tubes.

## 3. Results and discussion

### 3.1. Slurry formulation development

Basic stages in developing the slurry formulation are to optimize the amount of dispersant and binder. Menhaden fish oil has been extensively used as a dispersant in nonpolar solvent systems, since it

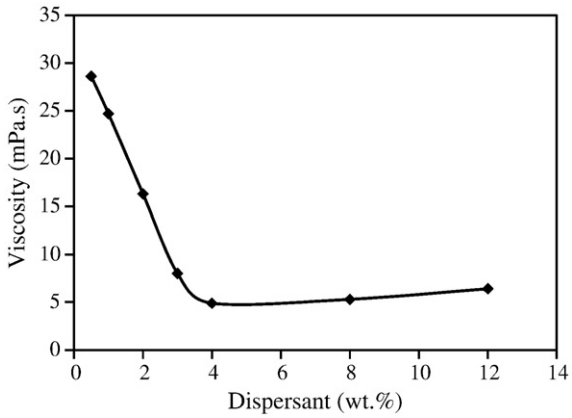


**Fig. 3.** Effect of dispersant concentration on the viscosity of NiO–YSZ slurries (solid loading = 0.42 cm/min).

is highly adsorbed onto particle surfaces [10]. Fig. 3 shows the effect of fish oil concentration on the viscosity of NiO–YSZ slurries (with a constant solid loading of 60 wt.%). Up to 4 wt.%, increasing the dispersant concentration will effectively result in decreasing the viscosity of the slurry. No significant influence on the viscosity is observed, however, when the concentration of the dispersant is increased further. This clearly reveals that fish oil molecules are effectively adsorbed on the NiO–YSZ surface up to 4 wt.% when saturation occurs. Therefore, the optimum concentration of the dispersant was determined to be 4 wt.%.

Having high molecular weight and great flexibility, polyvinyl butyral is a satisfactory binder in nonaqueous slurries [10]. Table 1 reports the adhesion strength of coatings with different concentrations of the binder. The importance of drying the coating should be initially emphasized. Surprisingly, no matter how much binder was used, all the coatings poorly adhered to the substrate when the drying duration was 1 h at room temperature. After 24 h drying at room temperature, however, the adhesion strength was a function of the binder concentration. Since PVB contributes to the adhesion characteristics of the slurry, the more PVB in the slurry, the higher the adhesion of the coating to the substrate. As stated in Table 1, no coating removal was observed in slurries with more than 7.5 wt.% PVB. Therefore, the minimum amount of the required binder is 7.5 wt.%. Nevertheless, since

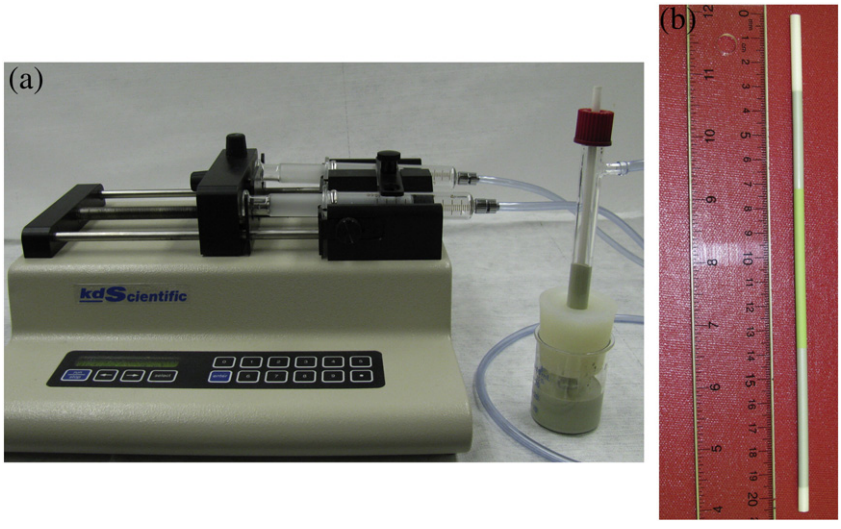


**Fig. 2.** (a) Dip coating setup and (b) a coated micro-tube (outside coating: NiO–YSZ anode, inside coating: LSM-YSZ cathode).