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Optimizing inkjet printing process to fabricate thick ceramic coatings

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ABSTRACT

7This article describes the use of Taguchi optimization and ANOVA techniques on inkjet printing process to determine optimal parameters for fabrication of thick ceramic coatings over glass substrates. Stable nanoparticle suspensions are synthesized through high energy milling of precursor powders with adequate quantities of binder and suspending solvent. Most often, inkjet printing process is being used for developing fine and thin layers (< 10 µm). However, an attempt is made to fabricate thick ceramic films by varying only IJP process parameters and without multiple layer deposition, thereby reducing efforts in ink synthesis and processing time of coated substrates. Three parameters of IJP were varied for developing a model that was used for precisely predicting the printed layer thickness under varying process parameters. ANOVA technique showed that open time interval in combination with nano particle concentration in the ink could potentially lead to thick coatings. The higher volume % of solvent in the diluted suspension ink under the influence of substrate heating contributed significantly to coffee stain effect with irregular surface coatings. However, increasing the concentration of nanoparticles in the diluted ink resulted in substantial improvement in thickness of the layer with simultaneous control of coating defects.

1. Introduction

Inkjet printing is an economical and cost effective micro fabrication technique used for deposition of thin layers on a target surface. The technique which was developed for the graphic printing community was eventually adopted as an economical process to deposit functional materials layer by layer over various types of substrates. Presently, inkjet printing has tremendous applications in solid oxide fuel cell (SOFCs) which requires very thin and dense ceramic coatings. Recently, inkjet printing (IJP) process as additive layer deposition technique has been utilized for fabricating thin as well as thick material layers by using nano particle suspensions. This development was successful as an effort to bridge the gap between thin as well as thick layer coating systems. However, the tremendous growth in many industries catered to the development of several types of printing systems to facilitate requirements in engineering and medical fields. Over the last two decades, several researchers have reportedly used inkjet printing process for manufacture of porous and dense oxide/ceramic layers. Recently, Rahul [1] developed completely stable, micro porous *t*-ZrO_2 membranes over steel substrates by low temperature sintering of inkjet printed coatings and compared micro structural characteristics of thin layer with bulk material through micro hardness, SEM and XRD. In another work, Rahul and Balasubramanian [2] inkjet printed YSZ nano particle suspensions on α-Al_2O_3 substrates to develop stable porous ceramic layers and studied the microstructure and crystal phase transformation. Wang [3] deposited sol-gel based precursor solution of gadolinium doped ceria over NiO-YSZ cermet anode using inkjet printing technique and estimated the sintering temperature of printed precursor film by using differential scanning calorimeter. Tomov [4] used porous metal supports for fabrication of SOFC coatings through direct ceramic inkjet printing (DCIJP) of anode (NiO/GDC) and electrolyte (GDC) material. Fasaki [5] inkjet printed titania films from water based suspensions using a macrojet solenoid print head and studied particle size distribution, stability, compatibility and photo catalytic performance. Mosiadz [6] deposited cerium gadolinium oxide ($Ce_{0.8}Gd_{0.2}O_2$) precursor solution on highly textured Ni-5%W strips using a 16-nozzle piezoelectric cartridge and a single electromagnetic nozzle. Fuller [7] reported fabrication of three dimensional (3-D) micro-electromechanical systems (MEMS) by ink-jet printing metal nano particle colloids. Yoo [8] conducted experimental studies on formation, spreading and drying of inkjet printed colloidal suspensions by using four different types of alcohol formulation and

Abbreviations: YSZ, Yttria stabilized Zirconia; IJP, Inkjet Printing; DOD, Drop on Demand

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assessed the monolayer forming mechanism by drop drying method.

As different types of thin material coatings were being developed through inkjet printing systems, we tried to develop thick coatings of ceramics/oxides in an attempt to introduce this technique for fabrication of oxidation resistance coatings. Most often, thick Yttria stabilized zirconia layers (> 50 μm) in thermal barrier coatings and wear/oxidation resistant coatings are fabricated through other dominant methods like plasma spray deposition, DC sputtering and electrophoretic deposition. It is a known fact that stabilized/doped zirconia exhibit improved properties compared to the pure counterpart. Pure zirconia material is unstable due to the phase transformation from monoclinic to cubic phases and vice versa. Hence, they are not very much useful for practical applications. However, stabilized or doped zirconia finds applications in the form of thin coated porous and non porous membranes in solid oxide fuel cells, oxygen sensors and biomedical implants.

In view of new applications, it was imperative to perform statistical optimization of the various parameters of inkjet printing process, specifically to study and understand the dynamic nature of parameters under the influence of precursor inks. Statistical optimization of IJP process parameters has not been comprehensively reported in scientific literature. However, very few researchers have demonstrated optimization of inkjet printing process parameters by image acquisition techniques [9].

Optimization of process parameters is carried out primarily to improve efficiency and produce high quality outputs for specific applications, while simultaneously attempting to reduce the cost and time for fabricating thick ceramic coatings. It is known that parameter design and optimization was introduced by Taguchi during 1980's for improving quality of manufactured products. Though many professionals have criticized this method for its narrow set of assumptions, it has been utilized by statisticians and engineers globally for effective quality control of products and processes. In comparison with optimization, conventional factorial experiments are most often conducted by varying one factor at a time and require larger number of experiments which potentially makes it an expensive and time consuming process. Moreover, factorial experiments do not take into account possible interactions between the parameters which entails to an inaccurate justification of the response function. Therefore, we used Taguchi orthogonal array to determine the optimal parameters for a known response function. In order to understand and check whether the optimal parameters are statistically significant, analysis of variance (ANOVA) was performed using steps described by Fischer [10]. From the several inkjet printing process parameters, we identified three parameters which could potentially contribute to substantial growth of layers. The parameters are open time (t_0, μsec), substrate temperature (T, °C) and ink content (W_i,%).

This research paper describes the optimization of inkjet printing process parameters using Taguchi DOE and ANOVA for determining optimal parameters that contribute to development of thick layers of Yttria stabilized zirconia on flat glass substrates through single layer depositions. Thus, reduction in number of layers could potentially decrease the processing time and make the process more effective and efficient. Additionally, an attempt is made to understand and determine the print parameters required to produce a well dried droplet with an approximate diameter within 0.5–1 mm and uniformly sized thin patterns exhibiting reduced coffee stain effect for various applications. Glass with very fine surface finish (Ra≅4 nm) was chosen as a candidate substrate so as to mitigate the surface variation effects that become a constraint in evaluation and assessment of layer thickness across multiple samples.

2. Materials and methods

2.1. Ink synthesis

5 mol% Yttria stabilized zirconia (YSZ, Screen-O-Graphic, India) agglomerated powder is taken as the precursor powder for preparation of ink. In order to inkjet print any material, it is essential to disperse the material homogeneously in a stable suspension with average particle size diameter in the submicron range. The precursor powder was dispersed in Terpineol (Vapor pressure≈10.5 mm of Hg at 20 °C) at 70:30 (solvent: powder) weight ratio. In order to achieve good ionic stability, 10 wt% Ethyl cellulose (Molychem, India) powder is introduced as a binder. This binder introduces ionic and electrostatic charge on the particle surface thereby imparting adhesive capabilities to the ink while it is deposited over non porous flat glass substrates. Additionally, dispersant media also should be capable of suspending the particles homogeneously without any unwanted charge neutralization. Moreover, it is known that the dispersant typically acts as a steric stabilizer in a colloid/suspension [11] and is effective in preventing crack formation during drying of the deposited film [12]. Thus, it was essential for any ink mixture to have a uniform particle size distribution that aids in formation of defect free layers, while printing/coating over various substrates.

The powders were agitated manually to form a mixture which was subjected to high energy milling in a planetary ball mill (Retsch, Germany) for 4 h 30 min at 160 RPM. After completion of milling process, concentrated YSZ ink was collected in 30 ml glass vials and stored at 20 °C temperature. An average particle size of 572 nm (SZ100 Nano Partica, Horiba, Japan) is measured using dynamic light scattering method with standard instrument specifications. The ink preparation methodology and stability assessment has been elaborately explained in our earlier articles [1,2].

2.2. Theory

Inkjet printing process involves shooting tiny droplets of suspended material on predefined patterns over the substrates. Subsequent drying of the wet drop pattern develops into a material layer. Most often, dispersant and carrier solvent contributes to creation of a porous geometry which is a typical characteristic of inkjet printing process. However, the porosity could be engineered to fully dense structures by multi layer depositions [13] or by varying the particle size distribution in the ink [2]. When compared with other deposition techniques, inkjet printing technique produces continuous thin layers and films with better control of thickness, porosity and composition for fabricating 2-dimensional as well as 3-dimensional features.

For coating ceramic inks through inkjet printing process, inks should be highly stable without any visible signs of sedimentation and possessing a uniform particle size distribution. The rheological properties of the ink should be adequate enough to generate uniform sized drops consistently through all the nozzles. A dimensionless number called ohnesorge number is used to describe the tendency of drop formation and splitting by relating viscous forces with inertial and surface tension forces. Derby [14] formulated a criterion Z for inkjet printing by relating Reynolds number (Re) and Weber number (We). Z is defined as inverse of ohnesorge number.

$$Re=\frac{v^2\rho a}{\sigma},\ We=\frac{v\rho a}{\eta}$$

$$(OH)^{-1}=Z=\frac{\sqrt{\sigma\rho a}}{\eta}$$

where, η is dynamic viscosity of ink, σ is surface tension, ρ is the density, a is characteristic length which is often taken as nozzle diameter(100 μm) and v is the drop velocity. For a functional material layer, uniformity in thickness and fine surface finish is beneficial. Non