Reactive Spray Deposition

Justin Roller, Technical Officer, High Temperature Fuel Cells Group / Agent Technique University of British Columbia

Syringe Pump Application Note
AN9

Introduction

Reactive spray deposition technology (RSDT) is a method of depositing films or producing nanopowders through combustion of metal-organic or metal-inorganic compounds dissolved in a solvent. This technology, also called flame-assisted deposition, will produce powders of controllable size and quality by changing conditions to control the stoichiometry of the final product. This results in a low-cost, continuous production method suitable for producing solid oxide fuel cell (SOFC) components and material in an open environment without the need for heat treating. RSDT can also deposit functionally graded film continuously.

Delivery of precursor solvents was accomplished using the gradient capability of Teledyne Isco Syringe Pumps.

Methods

Reactive spray deposition technology (RSDT) is being developed in Canada at the NRC-IFCI for processing of novel films and formation of nanopowders. The process involves pumping a dissolved metal-organic or metal-inorganic precursor through an atomizing nozzle and combusting the atomized spray. The atomization of the spray can be accomplished by nozzles that atomize by ultrasonics, air shear, liquid pressure and heat, or a combination of energy inputs. Precursor solutions containing the metal reactants required in the deposited film are pumped under pressure to the nozzle by use of a Teledyne Isco syringe pump. In addition, some techniques feed the precursors to the nozzle as an aerosol and the nozzle in not used in the atomization process.

In yet another version of the technique, a dissolved liquefied gas is added to the precursor solution to aid in atomization through a technique known as supercritical fluid atomization. Regardless of the nozzle type, the atomized spray is then combusted by an ignition source such as a single pilot flame from a point source or a ring of pilots surrounding the exit of the nozzle. An optimal ignition point must be chosen since igniting too close to the exit of the nozzle results in a fuel rich mixture that does not burn easily while igniting too far away results in an oxidant rich mixture. Pilot gases consist of methane and oxygen or an oxy-acetylene type gas. Pilot gases are supplied to the system by mass flow controllers or by passive rotameters.

Depositions onto substrates usually occur by impinging the flame on the desired substrate and allowing the reaction to occur long enough to achieve the desired thickness of film. If a nano-structured or dense film is desired, the flame should penetrate the boundary layer of the substrate. Longer flames (i.e. distance from nozzle to substrate) and higher concentrations of precursor material favor nucleation of particles and agglomeration prior to growth from the vapor phase directly on the substrate. This results in a powdery agglomeration of particles with poor adhesion. Care must be taken to prevent thermal shock to the substrate by controlling the heat-up and cool-down to deposition temperatures. This is generally done by heating the substrate from the back by resistive heaters or by another flame. Additionally, the heat-up and cool-down must be performed without the reactive precursors present so that a constant deposition temperature is maintained during film growth.

Alternatively, the flame can be directed at a sheet of cooling air, water, or a cooled metal surface for collection of powder particles instead of a growing film.

Advantages of RSDT

Multi-layer and/or functionally graded materials can be deposited in one single process by simply changing the precursor material and deposition conditions. Changing of the precursor material in a controlled manner can be accomplished by using existing HPLC technology for gradient elution. Functionally graded SOFC cathode materials have been explored by Meilin Liu and researchers at Georgia Tech.

SOFC components can be fabricated via other routes, such as electrochemical vapor deposition (EVD), chemical vapor deposition (CVD), physical vapor deposition (PVD), sol-gel, RF-sputtering, spin coating, slurry spraying, plasma spray, and screen-printing. The driving force in SOFC material development is a low cost, rapid processing method that can be done in one continuous process without the need for long sintering times at elevated temperatures. Each technique has its advantages and disadvantages, which usually involve high costs such as vacuum, long sintering steps, or large energy requirements.

One area of intense research is electrolyte deposition. Electrolyte thickness directly results in a higher ohmic resistance, especially at lower temperatures. A shorter oxygen path would be beneficial in lowering the operating temperature at an equivalent ohmic resistance. Conventional processing methods such as tape casting, screen-printing, slurry coating, and colloidal methods result in large shrinkage associated with the removal of plasticizers and binders in further sintering steps. Thickness of the electrolyte layer is also practically limited by these techniques.

Last modified December 20, 2007

Teledyne Isco, Inc.
P.O. Box 82531, Lincoln, Nebraska, 68501 USA
Toll-free: (800) 228-4373 • Phone: (402) 464-0231 • Fax: (402) 465-3091
E-mail: IscoInfo@teledyne.com