

Fabrication of Highly Ordered Porous Alumina

There is intense interest in the use of ordered porous materials as templates for fabrication of electronic, optoelectronic, photonic, microanalytical, and microelectromechanical devices. Researchers at Tokyo Metropolitan University and the NTT Basic Research Laboratory in Japan have reported the fabrication of highly ordered porous alumina by a two-step process. First, an array of shallow (~ 20 nm) concave features is formed in an aluminum sheet by indentation with a single-crystal SiC mold containing a hexagonally ordered array of convex features. The mold is produced by conventional electron beam lithography methods, and indentation is accomplished by pressing the mold against the aluminum at a pressure of ~ 2800 kg/cm². Next, the aluminum is anodized in oxalic acid at a constant voltage to produce an array of pores that initiate at each of the convex features. The authors describe specific anodization conditions that produce arrays of highly parallel pores as small as 60 nm in diameter and up to 30 μm deep. Extension of this approach to mass production of nanochannel-array-based devices is considered feasible because of the excellent quality of the template material, the relative simplicity of the process, and the ruggedness and reusability of the SiC mold.

From: J. Electrochem. Soc., 148, B152 (2001)

Chemical Modification of Proton Exchange Membrane Fuel Cell Catalysts

Continuing efforts are underway to improve the performance of proton exchange membrane fuel cells by increasing catalyst utilization efficiency at the cathode, which is often limited by the poor proton conductivity of the catalyst layer. A common method is to add a proton-conducting polymer, such as Nafion, to the catalyst layer. However, due to the morphology, low gas permeability, and poor electronic conductivity of Nafion, there is a limit to the level of Nafion that can be added before cell performance is adversely affected. Researchers at Memorial University of Newfoundland in Canada and H. Power Corporation in the U.S. have reported a new method to provide proton conductivity in the catalyst layer. The surface of the carbon black used as a Pt catalyst support was chemically modified by attachment of proton-conducting sulfonic acid groups via a silane linkage. It was found that such treatment before Pt deposition yielded better cell performance than if performed after Pt deposition. Optimum performance was reached for both types of treatment at 10% Nafion loading, and this performance was significantly better than that for untreated catalyst at the same Nafion loading. Comparable performance for the untreated catalyst was obtained only when the Nafion loading reached 30%.

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Local Detection of Dissolved Sulfur Species from Inclusions in Stainless Steel Using Ag Microelectrode

Pitting of stainless steel is often initiated at MnS inclusions in the surface. To gain insight into the mechanism of MnS dissolution, researchers at the University of Illinois developed a technique to determine hydrosulfide (HS⁻) from individual MnS inclusions. Webb, Paik, and Alkire used a microelectrochemical cell with a 25-μm diameter Ag electrode to characterize MnS dissolution from inclusions 20-30 μm in diameter. The Ag reacts electrochemically with hydrosulfide to yield Ag₂S and one electron. A second one-electron transfer yields Ag₂S and water. The Ag electrode is not electrochemically active with other sulfide species that could result from MnS dissolution, such as sulfite, thiosulfate, and sulfate. Using this microelectrochemical cell, the researchers found that at the rest potential in acidic conditions the MnS inclusions dissolved

to produce hydrosulfide. At higher anodic potentials in neutral solutions, no hydrosulfide was detected by the Ag microelectrode. The authors note "the results suggest two different reaction paths for inclusion dissolution, a chemical dissolution path and an electrochemical path." In the electrochemical path, the MnS inclusions dissolve to produce thiosulfate.

From: Electrochem. and Solid-State Lett., 4, B15 (2001)

Sensitive End-Point Detection for Dielectric Etch

In the production of integrated circuits, it is often crucial to accurately detect the end-point time during process steps where masked dielectric films are etched. The most common end-point method is optical emission spectroscopy (OES), which detects changes in the concentrations of etch reactants and/or products in the plasma via changes in the intensity of spectral lines corresponding to these species. However, OES has disadvantages with respect to sensitivity and resolution that limit its usefulness. Hudson and Dassapa at Lam Research Corporation have reported an end-point detection method based on measurement of a voltage signal from the bias-compensating high-voltage power supply of the electrostatic ceramic chuck which holds the wafer during the etch process. This voltage is extremely sensitive to the electrical resistance between the chuck and the plasma, of which the resistance of the silicon oxide film being etched is a key component. The intensity of the end-point signal is essentially independent of exposed oxide area and the method can be used for wafers with exposed oxide areas which comprise as little as 0.008% of the wafer area. The technique is currently being used successfully in a commercial wafer production facility for control of standard via etch processes, and applications in advanced fabrication (e.g., dual damascene via etch) are anticipated.

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Corrosion-Resistant Zeolite Coatings for Aluminum

Chromate conversion coatings are widely used to combat corrosion of aluminum alloys and other metals. The coating fabrication process uses Cr(VI), a known human carcinogen, so the search for environmentally- and health-conscious alternatives with equal or superior performance features continues to be a focus of research. Researchers at the University of California-Riverside have reported an *in situ* crystallization process that produces a coating with outstanding corrosion-resistance, wear-resistance, thermal stability, adhesion, and paint compatibility features. The authors deposited a crystalline aluminosilicate coating onto Al-2024-T3 parts from an aqueous solution of tetraethylorthosilicate, tetrapropylammonium hydroxide, sodium hydroxide, and aluminum hydroxide. The part was suspended in the solution and subjected to a 175°C heat treatment for about 16 hours, during which time a uniform, 35 μm thick, nonporous zeolite coating formed on all exposed surfaces of the part by a crystallization mechanism that occurs directly at the solid-liquid interface. Intracrystal porosity is avoided in these films because the structure-directing agent (tetrapropylammonium ion) remains in the zeolite structure and completely fills the pores.

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