Understanding Artifacts in Impedance Spectroscopy

Impedance spectroscopy (also commonly referred to as electrochemical impedance spectroscopy) is widely used to investigate corrosion processes, ion-conducting solids, fuel cells, electrode/electrolyte interfaces, and electrochemical reaction mechanisms and kinetics. While these measurements can provide valuable quantitative information, they must be performed with expertise and care to ensure that measurement artifacts in the experimental data do not lead to incorrect conclusions. J. A. Eastman and collaborators at Argonne National Laboratory recently published an extensive article that examined some of the principal sources of errors that typically plague practitioners of impedance spectroscopy. The authors demonstrated the effects of capacitance-to-ground (at the coaxial cables and the sample terminals) on three- and four-electrode measurements, and demonstrated that these high frequency artifacts (which manifest themselves as large magnitude inductances) can be quantitatively accounted for when the leakage capacitance is accurately known and included in the circuit analysis. While shortening the coaxial cables as much as possible is a good experimental practice, the authors clearly determined that it is still necessary to measure the cable and instrument capacitance and include them in the circuit analysis model if quantitative fits of model to experiment are desired.

From: J. Electrochem Soc., ***162**, H47 (2015).*

Effects of Pb Doping on Hole Transport Properties and Thin-Film Transistor Characteristics of SnO Thin Films

Oxide semiconductors have attracted considerable attention as next-generation channel materials for thin-film transistors (TFTs). While these materials offer high field effect mobility, they are mostly n-type. High-performance p-type oxides are needed for TFT for transparent low-power-consumption complementary circuits. Fortunately, SnO-based materials may provide p-type TFT oxides with acceptable carrier mobility when doped. Doped oxides must maintain a high off-state current and a high field-effect mobility. Researchers at the Tokyo Institute of Technology have investigated the effect of Pb doping on SnO and also demonstrated p-type TFTs using $Sn_{1-x}Pb_xO$ films. The researchers posit that for SnO-based p-type oxide TFTs, the hole concentration, resistivity, trap density, and crystal structure are critical in the doped material. With a higher level of Pb doping, the field-effect mobility decreased more rapidly than the Hall mobility. The authors also related the overall parametric response of the doped oxide TFT to the influence of trap states, dopant concentration, resistivity, and Fermi level pinning, which caused the high off-state current. Importantly, they identified that the contribution of dopant orbitals to the valence band maximum (VBM) will be important for useful p-type oxide TFT channel materials.

From: ECS J. Solid State Sci. Technol., ***4**, Q26 (2015).*

An Electrochemical Method for Measuring Localized Corrosion under Cathodic Protection

Since the early 20th century, cathodic protection (CP) has been a common means of mitigating corrosion of vulnerable structures, such as steel pipelines. This history notwithstanding, direct means of measuring the corrosion rate of a cathodically protected surface are rare and uncommon in practice. Traditional electrochemical methods based on the Stern-Geary relationship are generally restricted to charge-transfer controlled reaction kinetics. In many practical scenarios, the reaction rate is dependent on the mass transfer rate of cathodic reactants, such as oxygen, to the surface. Researchers at Deakin University in Australia have developed a new electrochemical method using coupled micro-electrode array sensors to address these limitations. With the micro-electrode array under CP, the nominal cathodic current density measured across the entire array or at singular electrodes is analyzed to estimate the anodic current density (*i.e.*, corrosion rate) at each electrode in the array. To demonstrate the ability to measure localized corrosion and its distribution under CP, the authors correlated calculated corrosion rates of carbon steel electrodes in aqueous sodium chloride solution with volume loss realized on the electrodes after exposure. As stated by the authors, the method may serve as a starting point for development of new tools for direct corrosion rate measurements for CP applications.

From: ECS Electrochem. Lett., ***4**, C1 (2015).*

Synthesis and Electron Microscopy of Superalloy Nanowires

Nickel-rich Ni-Fe-Cr superalloys possess excellent corrosion resistance properties. Much literature exists for their chemical and mechanical properties and behavior, except when they are formed in isolated nano-dimensional solids. Researchers from the Indian Institute of Science in Bangalore used a template-assisted, electrochemistry-based methodology for synthesizing Ni-Fe-Cr nanowires to begin exploring their properties. An anodic alumina membrane containing 200-nm diameter pores was used to form nanowires while electrodepositing at constant current from an electrolyte solution containing the chloride salts of Ni, Fe, and Cr. Scanning electron microscopy (SEM), combined with energy dispersive spectroscopy (EDS), revealed long (~5 µm) uniform diameter nanowires having on average composition of 77:18:5 weight percent Ni:Cr:Fe. Selected area diffraction (SAD) results indicated that the nanowire was a mixture of amorphous and ultrafine crystalline microstructure. A scanning transmission electron microscopy (STEM)-high angle annular dark field (HAADF) analysis showed similar results: uniform composition of 80:15:5 weight percent Ni:Cr:Fe. The authors investigated the possibility of microstructure engineering by inducing crystallization via electron beam heating. Crystalline regions within an amorphous matrix were identified and created. The as-synthesized nanowires were also characterized via magnetization curves. Regardless of wire orientation or randomness, low magnetic coercivity (~150 Oe) was found for this superparamagnetic material.

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Enhanced Electro-Optical Properties of Electrically Controlled Birefringence Cells

Liquid-crystal display (LCD) technology depends on control of the interactions between the LC molecules and the substrate surface. Surface alignment of the LC molecules is induced by treatments, such as rubbing and ion-beam (IB) bombardment, both of which have advantages in treating large areas. However, rubbing polyimide (PI) with a cloth produces electrostatic charge and inhomogeneous alignment. Researchers from Korea studied the use of a La_2O_3 layer (spin-coated, then annealed at various temperatures) subjected to IB irradiation and its impact on electro-optical (EO) properties. IB irradiation was shown via contact angle measurements to increase the surface energy of the La_2O_3 film, resulting in more uniformly aligned LC molecules through a stronger interaction between them and the surface compared to LC intermolecular interactions. Electrically controlled birefringence (ECB) cells were fabricated to test the transmittance and the response times. A cell made of the La_2O_3 film annealed at 500 °C exhibited a lower threshold voltage (30% lower at 90% transmittance) than that for a rubbed PI surface. Additionally, the La_2O_3-containing cell had rise and fall times two times quicker than the PI-containing cell. The authors attribute the improved EO properties to a higher dielectric constant and thus a larger electric field.

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