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**PREPARATION AND CHARACTERIZATION OF SOLID ELECTROLYTES FOR
SOLID OXIDE FUEL CELLS**

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Quarterly Report

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During this quarter, we have analyzed EXAFS spectra of rare earth doped ceria and identified Gd and Sm doped ceria electrolytes for further investigations in this project. Although electrical properties of the material have been examined in detail, very little work has considered the microstructural/property relationships, particularly in relationship to atomic and geometric structures of these ceria based oxides. Earlier we have observed an ionic conductivity of 8.3×10^{-2} S/Cm in the $(\text{CeO}_2)_{0.8}(\text{GdO}_{1.5})_{0.2}$ at 800 C. which is approximately four times that of Y_2O_3 doped ZrO_2 at the same temperature. The fraction of Ce^{+4} ions reduced to Ce^{+3} as a function of temperature and oxygen partial pressure. The partial reduction of cerium oxide generates mobile electrons and causes electronic conductivity in the electrolyte at temperatures above 500 C. In this quarter, we have attempted to measure the microstructural properties using SEM, TEM and HVC STM techniques. The PI has generated funds to acquire HVC STM from National Science Foundation. He has involved in the purchasing procedures since this instrument is expensive and requires custom made design etc. Dr. R. Bobba has issued the design and requirements to the university purchasing department in order to solicit bids from commercial vendors.

In the previous quarter, one of the graduate student and the research associate have initiated the study of the performance of commercial Nation 117, 115, and 112 and compared with platinum and platinum ruthenium. The graduate students and other staff in the project spent time to familiarize with the operation of the fuel test station. This included understanding the effect of back pressure, gas flow rate, and humidifier temperature. The current output was found to be satisfactory and is same for the similar membranes. Inadvertent heating of the fuel cell to 146 C

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resulted in higher current output indicating that short term heating of the Nafion membrane may be beneficial even though the temperature for continuous operation should not exceed 90 C. A series of experiments were conducted by this team with various catalyst loading of 4 and 12 mg/cm² operating on unpressurized air and pressurized air at different temperatures. Our measurements suggest increased loading does not offer significant benefits at high current densities, improving anode utilization was considered necessary for the improvement of the anode performance. The techniques for application of the anode were thus modified especially with regard to improving the interfacial contact between the catalyst and the membrane.

Mr. Tommy Rockwood joined in this project to develop graduate thesis in fuel cells. He was asked to utilize the impedance spectroscopy for interfacial characterization of electrochemical systems and improve the existing methods and conditions. In the last quarter we have acquired EG&G PAR Model 273 potentiostat/galvanostat and HF impedance analyzer for ac impedance and cyclic voltammetry measurements. One of the techniques he is suggested was three-electrode impedance spectroscopy. This technique is particularly useful for the characterization of subcomponent and interfacial properties. He will be developing thesis entitled " Interfacial and Surface Characterization of Mixed Ionic and Electronic Conductors for SOFCs "The scope of his research effort ranges from theoretical consideration of ion transport mechanism to pragmatic realities of actual applications.

We have completed the measurements of XAS, in a series of membranes that are tested earlier using our fuel cell test station. The effect of alloying element on the Pt d band occupancy was determined by analyzing the Pt L₃ and L₂ Near Edge X-ray Absorption Spectroscopy (XANES). We have obtained the effect of the alloying element on the Pt-Pt bond distance by analyzing the Pt L₃ EXAFS. We have also measured the K edge XANES of the Pt and Ru. Phase and amplitude data for the Pt-Pt interactions were derived from Pt foil data using the PEEF program. We have also used this program to derive phase and amplitude parameters for Pt-Ru, Ru-Pt, Ru-Ru, and Ru-O interactions. Analysis of XAS showed that the respective edge jumps at the Pt L₃ edge were 0.11 and 0.09. This indicates respective Ru and Pt loadings were 1.63 mg/cm² and 0.90 mg/cm². This gives an atomic ratio of Ru to Pt of 3.34:1

5

ACCOMPLISHMENTS: Summary:[10/15/1996 to 9/30/1996] The original intent of the proposal was to develop " Materials Research Instrumentation Laboratory " conducive to Fuel Cell research. We have completed this task by acquiring, installing and testing successfully. We have encouraged our students to participate in every effort. We have studied the AC impedance of the entire family of rare earth dopants in Cerium Oxide. We have identified Sm and Gd impurities are the best ionic conductors for SOFC applications. We have measured the EXAFS of these rare earths first time using our synchrotron radiation source at Center for Advanced Microstructures and Devices (CAMD). Simultaneously we have initiated our research effort in characterizing Pt/Ru electrocatalysts on conventional Nafion membrane. The PI has presented three papers in national conferences including HBCU-PETC conference and 37th Power sources conferences. This project has supported 12 minority students to attend the conference.