FINAL REPORT

NASA COOPERATIVE AGREEMENT NCC3-486

"SPACE ENVIRONMENT STABILITY AND PHYSICAL PROPERTIES OF NEW MATERIALS FOR SPACE POWER AND COMMERCIAL APPLICATIONS"

September 23, 1997

PRINCIPAL INVESTIGATOR:

Dr. Paul D. Hambourger

INSTITUTION:

Cleveland State University
E. 24th Street at Euclid Avenue
Cleveland, OH 44115
(216) 687-2439

GRANT PERIOD:

July 3, 1996 - July 2, 1997

I. GOALS AND OBJECTIVES

To test and evaluate suitability of materials for use in space power systems and related space and commercial applications, and to achieve sufficient understanding of the mechanisms by which the materials perform in their intended applications. Materials and proposed applications included but were not limited to: Improved anodes for lithium ion batteries, highly-transparent arc-proof solar array coatings, and improved surface materials for solar dynamic concentrators and receivers. Cooperation and interchange of data with industrial companies as appropriate.

II. ACCOMPLISHMENTS vs GOALS AND OBJECTIVES

Useful and informative results were obtained on virtually all materials investigated. For example, the stability of ITO-based arc-proof transparent coatings was greatly improved by substitution of silicon oxide for magnesium fluoride as a dopant. Research on "air-doped" ITO films has yielded new insight into their conduction mechanism which will help in further development of these coatings. Some air-doped films were found to be extremely pressure-sensitive. This work may lead to improved, low-cost gas sensors and vacuum gauges; it is continuing under the succeeding Cooperative Agreement NCC3-522. Work on another promising transparent arc-proof coating (titanium oxide) was initiated in collaboration with industry. Graphite oxide-like materials were synthesized and tested for possible use in high-energy-density batteries and other applications. We also started a high-priority project to find the cause of unexpected environmental damage to the exterior of the Hubble Space Telescope (HST) discovered on a recent Shuttle mission. Materials were characterized before and after

exposure to soft x-rays and other threats in ground-based simulators. This work is continuing under NCC3-522. It is essential for adequate protection of HST and other costly, long-duration orbiting facilities.

Two technical publications were authored during the grant period (copies of cover pages attached). Further publications will result as projects continue under NCC3-522.

III. INTERACTION WITH INDUSTRY

Several of our research projects attracted attention from commercial businesses. For example, the work on high-resistivity arc-proof coatings has attracted the attention of coatings users and manufacturers, one of whom is providing us with test samples.

IV. COSTS

All work was accomplished within budget. There were no cost overruns.

Paper O-9

High-Transparency Thin Films with Tailorable Sheet Resistivity

A. M. Pal, A. J. Adorjan, and P. D. Hambourger
Cleveland State University; Cleveland, OH* (216) 687-2439

J. A. Dever
NASA Lewis Research Center; Cleveland, OH (216) 433-6294

Henry Fu
Ohio Aerospace Institute; Cleveland, OH (216) 433-6294

*Work at Cleveland State University supported by NASA Lewis Research Center, Cooperative Agreements NCC3-383 and NCC3-486

40th Society of Vacuum Coaters Technical Conference
April 12-17, 1997
New Orleans, La

SYNTHESIS AND THERMAL STABILITY OF GRAPHITE OXIDE-LIKE MATERIALS

Ching-cheh Hung and Jean Corbin***
**NASA Lewis Research Center, Cleveland, Ohio 44145*
***Department of Chemistry, Cleveland State University, Cleveland, Ohio 44115*

Introduction

In a recent study of the graphite oxide reaction with $AlCl_3$, a product was found to have an Al:C atomic ratio as high as 1:4. Heating this material in air resulted in Al_2O_3 with a surface area of 80 m^2/gm [1]. This result suggests the possible applications in the areas of batteries, catalysts, and sensors.

Graphite has been used as the reactant for graphite oxide synthesis since its discovery in 1860 [2]. In this research, the possibility of using today's commercially available non-graphite carbon to synthesize graphite oxide was studied. The products were studied by examining their structure, chemical composition, thermal stability and reactivity to $AlCl_3$.

Experimental

The method developed by Hummers and Offeman [3] was used to synthesize graphite oxide. This method uses H_2SO_4, $NaNO_3$ and $KMnO_4$ as the reactants. In this research five different kinds of carbon materials were used to react with these chemicals. They were, in the order of decreasing x-ray diffraction (XRD) peak height, crystalline graphite powder (300 mesh, 99%), graphite powder (<1 micron, 99.9995%), graphite sheet (0.254 mm thick, 99.9%), graphitized carbon fiber (Amoco VCB-45, 10 micron diam., 99%), and activated carbon (1100 m^2/gm, containing small amounts of Al and Si).

The graphite oxide-like materials thus obtained were heated in nitrogen at 100, 150, and 200°C for 20-24 hours. The heating rates were such that the above three decomposition temperatures were reached in 16, 16, and 180 minutes, respectively. The sample mass before and after such heating was measured. The carbon mass loss during these reactions was estimated by finding the differences between the carbon masses in the reactants and the products, which were calculated from the products of the sample masses and their carbon mass %. These carbon mass losses as fractions of total mass loss, as well as total carbon mass in the reactant, were then calculated.

The bulk compositions were analyzed using a "Leco" process for carbon and sulfur contents, and inductively coupled plasma mass spectrometry (ICPMS) for metal contents, both described elsewhere [4]. The surface compositions were analyzed using x-ray photoelectron spectroscopy (XPS). Their structures were studied using x-ray diffraction (XRD) data. Their microscopic views were obtained from scanning electron microscopy (SEM). Their semi-quantitative chemical analyses were conducted using their energy dispersive spectra (EDS).

The products obtained from the precursors of crystalline graphite powder, sub-micron graphite powder, and activated carbon were exposed to $AlCl_3$ to test their ability to hold large quantities of aluminum. They are exposed to $AlCl_3$ at temperature histograms of from 120°C to 193°C for 42 hours, and finally 225°C for 0.2 hour to evaporate the unused $AlCl_3$.

Results and discussion

Chemical analysis of the products of the graphite oxide synthesis reactions is described in Table 1. All samples contained large quantities of C and O.

The XRD data indicated that none of these products had graphite peaks. The sample made from graphite sheet had sharp peaks at 5.23 and 2.62 Å. Together with their high sulfur content (Table 1), it is believed to be a H_2SO_4-graphite intercalated compound with an identity period of 5.23 or 10.46Å. This sample was not a graphite oxide-like compound and was not studied any further.

The sample made from crystalline graphite was the only one that had the graphite oxide peak at about 8 Å. All other samples has no XRD peaks, suggesting amorphous structures. Since they did not have graphite oxide peaks but are compounds of carbon and oxygen, they are called graphite oxide-like materials.

Table 2 describes the mass loss of the graphite oxide and graphite oxide-like materials during their thermal decomposition. For graphite oxide made from crystalline graphite, most of the mass loss occurred before they reached 150°C. For all other samples, most of the mass loss occurred before they reached 100°C.