TECHNICAL REPORT ARCCB-TR-99002

THE USE OF THERMAL ANALYSIS IN THE CHARACTERIZATION OF A POLYMER SURFACE

MARK F. FLESZAR ALLISON WELTY 19990324 011

AD

MARCH 1999



APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

DTIC QUALITY INSPECTED 1

DISCLAIMER

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

The use of trade name(s) and/or manufacturer(s) does not constitute an official endorsement or approval.

DESTRUCTION NOTICE

For classified documents, follow the procedures in DoD 5200.22-M, Industrial Security Manual, Section II-19, or DoD 5200.1-R, Information Security Program Regulation, Chapter IX.

For unclassified, limited documents, destroy by any method that will prevent disclosure of contents or reconstruction of the document.

For unclassified, unlimited documents, destroy when the report is no longer needed. Do not return it to the originator.

REPORT DOCUMENTATION PAGE

Ş

Form Approved OMB No. 0704-0188

Automatic and a finite section of the section is a section. Index of the section is a section is a section in the section is a section is a section in the section is a section i				
T. AGENCY USE ONLY (Leave blank) 2. REPORT DATE S. REPORT TYPE AND DATES COVERED March 1999 That That S. FUNDING NUMBERS 4. THE AND SUBTILE THE USE OF THERMAL ANALYSIS IN THE AMCMS No. 6226.24.H180.0 CHARACTERIZATION OF A POLYMER SURPACE AMCMS No. 6226.24.H180.0 S. ATHORS) REPORT NUMBERS AMCMS No. 6226.24.H180.0 Mark F. Flesza: and Allison Weity REPORT NUMBERS AMCMS No. 6226.24.H180.0 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) R. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) REPORT NUMBER U.S. Army ARDEC REPORT NUMBER ARCCB-TR-99002 U.S. Army ARDEC Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Its SPONSORIG/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Its SPONSORIG	Public reporting burden for this collection of info gathering and maintaining the data needed, and collection of information, including suggestions for Davis Highway, Suite 1204, Arlington, VA 22202-4	rmation is estimated to average 1 hour per completing and reviewing the collection of or reducing this burden, to Washington Hea 302, and to the Office of Management and	response, including the time for revie information. Send comments regardin adquarters Services, Directorate for In Budget, Paperwork Reduction Project	wing instructions, searching existing data sources, Ig this burden estimate or any other aspect of this formation Operations and Reports, 1215 Jefferson (0704-0188), Washington, DC 20503.
A. TILE AND SUBTIVE PURIL THE USD OF THERMAL ANALYSIS IN THE CHARACTERIZATION OF A POLYMER SURFACE S. FUNDING NUMBERS CHARACTERIZATION OF A POLYMER SURFACE AMCMS No. 6226.24.1180.0 PRON No. PISK2DIDEX Mark F. Fleszar and Allison Weity REPORT NUMBERS 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) E. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Amy ARDEC REPORT NUMBER Benet Laboratories, AMSTA-AR-CCB-O Waterviller, NY 12189-4050 I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Amy ARDEC I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Amy ARDEC I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Amy ARDEC I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Amy ARDEC I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Amy ARDEC I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Amy ARDEC I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) I. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	1. AGENCY USE ONLY (Leave blank	2. REPORT DATE	3. REPORT TYPE AND	DATES COVERED
THE USE OF THERMAL ANALYSIS IN THE CHARACTERIZATION OF A POLYMER SURFACE AMCMS No. 6226.24.1180.0 PRON No. PJ8K2DIDEX 6. AUTHOR(5) Mark F. Fleszar and Allison Weity PERFORMING ORGANIZATION NAME(5) AND ADDRESS(E5) E. PERFORMING ORGANIZATION NAME(5) AND ADDRESS(E5) E. PERFORMING ORGANIZATION NAME(5) AND ADDRESS(E5) U.S. Army ARDEC Benet Laboratories, AMSTA-AR-CCB-O Waterview, INV 1188-0500 II. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) II. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) J. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) II. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) II. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) J. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) II. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) II. SPONSORING/MONITORING AGENCY NAME(5) AND ADDRESS(E5) J. SUPPLEMENTARY NOTES Thermal Analysis Society, Cleveland, OH. AGINCY REPORT NUMBER Table Distribution with the convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) hedi prover given monitor the prevent membrane, Intermal Analysis, and aga humatifying system membrane. Thermogravimetry (TGA) and Hifferential scanning ealoringer convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) hedi prower given annotic the prevence of contaminating ions in the agroups condecasta. Thermogravimetry (TGA) and Hifferential scanning ealoringer convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) hedi prower given consistof a polymer membrane, IDE (Jawa Set)	4. TITLE AND SUBTITLE	March 1999	Final 5	. FUNDING NUMBERS
	THE USE OF THERMAL ANALYSI CHARACTERIZATION OF A POLY	IS IN THE (MER SURFACE		AMCMS No. 6226.24.H180.0 PRON No. PJ8K2D1DEX
Mark F. Fleszar and Allison Weity PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) SPONSORING/MONITORING AGENCY NAME(S) ADDRESS(ES) SPONSORING/MONITORING AGENCY NAME(S) ADDRESS(ES) SPONSORING/MONITORING AGENCY NAME ADDRES	6. AUTHOR(S)	· · · · · · · · · · · · · · · · · · ·		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) E. PERFORMING ORGANIZATION REPORT NUMBER U.S. Army ARDEC Benet Laboratories, AMSTA-AR-CCB-O ARCCB-TR-99002 9. SPONDRING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) 10. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) 10. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army ARDEC Close Combat Armaments Center Presented at the 26 th Annual Conference of the North American Thermal Analysis Society, Cleveland, OH. Published in proceedings of the conference. Presented at the 26 th Annual Conference of the North American Thermal Analysis Society, Cleveland, OH. 12a. DISTRIBUTION / AVAILABILITY STATEMENT 12b. DISTRIBUTION CODE 13. ABSTRACT (Maximum 200 words) 12b. DISTRIBUTION (CODE are powerful tools that can be used to characterize the physical properties of a polymer membrane. Energy and the age numidifying system. Thermogravimetry (TOA) and Informating adormetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Thermal Analysis, and a gas humidifying system. Thermogravimetry (TOA) and Informating adormetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Energy. 14. SUBJECT TERMS 15. NUMSER OF PAGES 17. SECURITY CLASSIFICATION 16. SECURITY CLASSIFICATION 15. NUMSER OF PAGES 17. SECURITY CLASSIFICATION 16. SECURITY CLASSIFICATION 15. NUMSER OF PAGES 17. SECU	Mark F. Fleszar and Allison Welty			
U.S. Army ARDEC ARCCB-TR-99002 Bent Laboratories, AMSTA-AR-CCB-O ARCCB-TR-99002 3. SPONSORING /MONITORING AGENCY NAME(S) AND ADDRESS(ES) 10. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) 10. SPONSORING /MONITORING AGENCY NAME(S) AND ADDRESS(ES) 10. SPONSORING / MONITORING AGENCY NAME/SI 20. S. Army ARDEC Close Combat Armaments Center Ploating Arsenal, NJ 07306-5000 11. SUPPLEMENTARY NOTES 7. SupPLEMENTARY NOTES Presented at the 26 ⁴ Annual Conference of the North American Thermal Analysis Society, Cleveland, OH. Pablished in proceedings of the conference. 12b. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12b. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12b. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12b. DISTRIBUTION / AVAILABILITY STATEMENT Approved monorized the presence of contaminating ons in the aqueous condenset. TGA can easily measure the distribution of mala atalysts on a polymer membrane. Including system. Thermogravimetry (TGA) and atalysts on a polymer surface due to the much lower decomposition temperature of the polymer. DSC can be used to measure the concentration of Telfon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUMERE OF PAGES 16. SECURITY CLASSIFICATION INCLASSIFIED	7. PERFORMING ORGANIZATION NA	ME(S) AND ADDRESS(ES)	8	PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) 10. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) 11. SUPPLEMENTARY NOTES 12. SPONSORING / MONITORING Conference of the North American Thermal Analysis Society, Cleveland, OH. Published in proceedings of the conference of the North American Thermal Analysis Society, Cleveland, OH. 12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 12. DISTRIBUTION / AVAILABILITY STATEMENT Tacl cells are electrochemical systems that convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) field alloyse are polymer surface due to the much hower decomposition temperature of the polymer. DSC can be used to measure the distribution of meal atalysts on a polymer surface due to the much hower decomposition temperature of the polymer. DSC can be used to measure the concentration of Teflon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUMEER CP PAGES	U.S. Army ARDEC Benet Laboratories, AMSTA-AR-CCI Watervliet, NY 12189-4050	B-O		ARCCB-TR-99002
U.S. Army ARDEC Close Combat Armaments Center Preadmay Arsenal, NU 07806-5000 11. SUPPLEMENTARY NOTES Presented at the 26 th Annual Conference of the North American Thermal Analysis Society, Cleveland, OH. Published in proceedings of the conference. 12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 13. ABSTRACT (Maximum 200 words) Puel cells are electrochemical systems that convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) fuel all power system consists of a polymer rembrane, finely disbursed catalyst, and a gas humidifying system. Thermogravimetry (TGA) and tifferential scanning calorimetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Ion thromatography can monitor the presence of contaminating ions in the aqueous condensate. TGA can easily measure the distribution of metal atalysts on a polymer surface due to the mach lower decomposition temperature of the polymer. DSC can be used to measure the concentration of Teflon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUMABER CP PAGES Type Cell, Nafion, Thermal Analysis, analysis, Ion Chomatography 15. NUMABER CP PAGES 17. SECURITY CLASSIFICATION 18. SECURITY CLASSIFICATION 19. SECURITY CLASSIFICATION 10. Exercised Security CLASSIFICATION 18. SUBJECT TERMS 116. SECURITY CLASSIFIED 117. SECURITY CLASSIFIED </td <td>9. SPONSORING / MONITORING AGEN</td> <td>NCY NAME(S) AND ADDRESS(ES</td> <td>)</td> <td>0. SPONSORING / MONITORING AGENCY REPORT NUMBER</td>	9. SPONSORING / MONITORING AGEN	NCY NAME(S) AND ADDRESS(ES)	0. SPONSORING / MONITORING AGENCY REPORT NUMBER
11. SUPPLEMENTARY NOTES Presented at the 26 th Annual Conference of the North American Thermal Analysis Society, Cleveland, OH. Published in proceedings of the conference. 12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited. 13. ABSTRACT (Maximum 200 words) Tuel cells are electrochemical systems that convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) fuel 116 Ip over system consists of a polymer membrane, finely disbursed catalyst, and a gas humidifying system. Thermogravimetry (TGA) and lifferential scanning calorimetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Ion shromatography can monitor the presence of contaminating ions in the aqueous condensate. TGA can easily measure the distribution of metal atalysts on a polymer surface due to the much lower decomposition temperature of the polymer. DSC can be used to measure the concentration of Teffon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUMBER CF PAGES Viet Cell, Nafion, Thermal Analysis,	U.S. Army ARDEC Close Combat Armaments Center Picatinny Arsenal, NJ 07806-5000			
12a. DISTRIBUTION / AVAILABILITY STATEMENT 12b. DISTRIBUTION / CODE Approved for public release; distribution unlimited. 12b. DISTRIBUTION / CODE 13. ABSTRACT (Maximum 200 words) 13. ABSTRACT (Maximum 200 words) Fuel cells are electrochemical systems that convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) fuel lower system consists of a polymer membrane, finely disbursed catalyst, and a gas humidifying system. Thermogravimetry (TGA) and lifferential scanning calorimetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Ion rhromatography can monitor the presence of contaminating ions in the aqueous condensate. TGA can easily measure the distribution of metal atalysts on a polymer sufface due to the much lower decomposition temperature of the polymer. DSC can be used to measure the concentration of Teflon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUMBER OF PAGES 'uel Cell, Nafion, Thermal Analysis, 200 15. NUMBER OF PAGES 17. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 17. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION ULL 18. TEADOL 280,05500 19. SECURITY CLASSIFICATION	11. SUPPLEMENTARY NOTES Presented at the 26 th Annual Conferen Published in proceedings of the confer	ce of the North American Therma rence.	l Analysis Society, Cleveland	I, OH.
Approved for public release; distribution unlimited. 13. ABSTRACT (Maximum 200 words) Fuel cells are electrochemical systems that convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) fuel ellower system consists of a polymer membrane, finely disbursed catalyst, and a gas humidifying system. Thermogravimetry (TGA) and hifferential scanning calorimetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Ion intromatography can monitor the presence of contaminating ions in the aqueous condensate. TGA can easily measure the distribution of metal atalysts on a polymer surface due to the much lower decomposition temperature of the polymer. DSC can be used to measure the concentration of Teflon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUMBER OF PAGES ¹ / ₁ vel Cell, Nafion, Thermal Analysis, 200 18. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 17. SECURITY CLASSIFIED 18. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 10. TSEQUELT TERMS 10. SECURITY CLASSIFIED 10. LIMITATION OF ABSTRACT UNCLASSIFIED	12a. DISTRIBUTION / AVAILABILITY S	TATEMENT	1	2b. DISTRIBUTION CODE
13. ABSTRACT (Maximum 200 words) Fuel cells are electrochemical systems that convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) fuel cell power system consists of a polymer membrane, finely disbursed catalyst, and a gas humidifying system. Thermogravimetry (TGA) and lifferential scanning calorimetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Ion thromatography can monitor the presence of contaminating ions in the aqueous condensate. TGA can easily measure the distribution of metal atalysts on a polymer surface due to the much lower decomposition temperature of the polymer. DSC can be used to measure the concentration of Teflon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUM.B3R CF PAGES "uel Cell, Nafion, Thermal Analysis, Latalyst, Ion Chromatography 18. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFICATION OF THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION UNCLASSIFIED 17. SECURITY CLASSIFICATION JOR THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION UNCLASSIFIED 19. SECURITY CLASSIFICATION ULL SSIFICATION UNCLASSIFIED 18. TEAL 19. TEAL SECURITY CLASSIFICATION ULL SSIFICATION UNCLASSIFIED 19. SECURITY CLASSIFICATION ULL SSIFICATION ULL SSIFICATION ULL SSIFIED	Approved for public release; distributi	on unlimited.		
Hel cells are electrochemical systems that convert hydrogen into electricity without combustion. A proton exchange membrane (PEM) fuel bell power system consists of a polymer membrane, finely disbursed catalyst, and a gas humidifying system. Thermogravimetry (TGA) and lifferential scanning calorimetry (DSC) are powerful tools that can be used to characterize the physical properties of a polymer membrane. Ion chromatography can monitor the presence of contaminating ions in the aqueous condensate. TGA can easily measure the distribution of metal atalysts on a polymer surface due to the much lower decomposition temperature of the polymer. DSC can be used to measure the concentration of Teflon in a polymer blend by measuring the melting energy. 14. SUBJECT TERMS 15. NUMABER OF PAGES 'uel Cell, Nafion, Thermal Analysis, Latalyst, Ion Chromatography 16. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFICATION OF ABSTRACT UNCLASSIFIED 17. SECURITY CLASSIFICATION INCLASSIFIED 18. SECURITY CLASSIFIED 19. SECURITY CLASSIFICATION ULL 20. LIMITATION OF ABSTRACT UNCLASSIFIED 0. FABSTRACT ULL 20. LIMITATION OF ABSTRACT ULL	13. ABSTRACT (Maximum 200 words))		
14. SUBJECT TERMS 15. NUMBER OF PAGES 3uel Cell, Nafion, Thermal Analysis, 10 Catalyst, Ion Chromatography 16. PRICE CODE 17. SECURITY CLASSIFICATION OF REPORT INCLASSIFIED 18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 20. LIMITATION OF ABSTRACT UL	Fuel cells are electrochemical systems cell power system consists of a polyme differential scanning calorimetry (DSC) chromatography can monitor the preser catalysts on a polymer surface due to the of Teflon in a polymer blend by measu	that convert hydrogen into electri er membrane, finely disbursed cat) are powerful tools that can be use nee of contaminating ions in the ac e much lower decomposition tempo using the melting energy.	city without combustion. A p alyst, and a gas humidifying s ed to characterize the physical ueous condensate. TGA can erature of the polymer. DSC c	roton exchange membrane (PEM) fuel system. Thermogravimetry (TGA) and properties of a polymer membrane. Ion easily measure the distribution of metal an be used to measure the concentration
14. SUBJECT TERMS 15. NUMBER OF PAGES Fuel Cell, Nafion, Thermal Analysis, 10 Catalyst, Ion Chromatography 16. PRICE CODE 17. SECURITY CLASSIFICATION OF REPORT INCLASSIFIED 18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 20. LIMITATION OF ABSTRACT UL				
10 10 Catalyst, Ion Chromatography 16. PRICE CODE 17. SECURITY CLASSIFICATION OF REPORT JNCLASSIFIED 18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 20. LIMITATION OF ABSTRACT UL SN 7540-01-280-5500 Standard Form 298 (Bay, 2-89)	14. SUBJECT TERMS			15. NUMBER OF PAGES
17. SECURITY CLASSIFICATION OF REPORT JNCLASSIFIED 18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED 19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED 20. LIMITATION OF ABSTRACT UL 5N 7540-01-280-5500 Standard Form 298 (Bey 2-89)	Catalyst, Ion Chromatography			10 16. PRICE CODE
SN 7540-01-280-5500 Standard Form 298 (Bev. 2-89)	17. SECURITY CLASSIFICATION 18 OF REPORT UNCLASSIFIED U	B. SECURITY CLASSIFICATION OF THIS PAGE JNCLASSIFIED	19. SECURITY CLASSIFICA OF ABSTRACT UNCLASSIFIED	TION 20. LIMITATION OF ABSTRACT UL
	ISN 7540-01-280-5500			Standard Form 298 (Rev. 2-89)

TABLE OF CONTENTS

<u>Page</u>

INTRODUCTION	1
EXPERIMENTAL ANALYSIS	
RESULTS	2
CONCLUSION	5
REFERENCES	6

TABLES

1.	Anion and Metal Concentrations in Fuel Cell Condensate Samples	3
2.	Teflon Analysis by DSC	4
3.	New and Used Membrane Analysis by TGA	5
	LIST OF ILLUSTRATIONS	
1.	DSC scan of membrane samples and standard	7
2.	Oxidation of membrane and catalyst	

i

INTRODUCTION

Fuel cells are electrochemical devices similar in structure to batteries with two porous electrodes separated by an electrolyte. Electricity is generated through the chemical reaction of a hydrogen-based fuel and an oxidant-containing oxygen inside the fuel cell. Hydrogen flows over the anode, which is a porous electrocatalyst that splits the hydrogen into positively charged hydrogen ions and electrons. Electrons flow through the external circuit, while only hydrogen ions can pass through the anode and into the electrolyte toward the cathode. The returning electrons from the circuit reduce the oxygen at the porous cathode. The hydrogen and oxygen ions then react to form water and thermal energy.

The most critical element of the fuel cell is the membrane assembly. The membrane, typically Nafion, has polytetrafluoroethylene-like segments with sulfonic acid regions in the structure, and the surfaces are coated with a fine dispersion of various metal catalysts. Fuel gases require humidification to moisten the membrane in order to transport the protons from the anode to the cathode. The stability of the membrane and dispersion of the catalyst on the surface are critical elements to the success of a solid polymer fuel cell. Ion chromatography, differential scanning calorimetry (DSC), and thermogravimetric analysis (TGA) are powerful tools to monitor the exhaust condensate and to characterize the membrane.

EXPERIMENTAL ANALYSIS

Water samples of condensate taken periodically from both the anode and cathode side of a fuel cell during testing were analyzed. After the cell was disassembled, samples of the anode and cathode membrane were also analyzed.

Water samples were taken at periodic intervals during the fuel cell test and analyzed for anions by ion chromatography. We used a Dionex model DX-120 with an AG4 guard column and an AG4 separator column. Then emission spectroscopy for metals was conducted using a Perkin-Elmer model 6500 ICP-ES. The water samples for chromatography were analyzed without dilution or pretreatment. Accordingly, the standards and samples were sealed in 5-ml vials and placed in a model AS40 auto-sampler for analysis. The samples were injected into the system using a 25-microliter sample loop with a system flow rate of 2.8 ml/minute at a pressure of 600 psi. An analysis was run for 10 minutes using an eluent of sodium carbonate/sodium bicarbonate. The data were analyzed using a three-point calibration method for each ion. Samples for emission spectroscopy were also analyzed without pretreatment. These samples and standards were aspirated into an argon plasma, and a calibration curve of emission count versus concentration for the standards was established.

1

Samples of a new and used membrane were analyzed by DSC to determine the concentration of Teflon-like polymer backbone in the Nafion membrane. Also TGA was used to measure the concentration of catalyst on the membrane surface. Samples for both techniques were prepared by cutting a specimen 2.159-mm in diameter with a brass cork borer.

Samples for DSC analysis were weighed on a microbalance and encapsulated in hermetically-sealed aluminum pans. The samples and reference were placed in a Perkin-Elmer DSC 7 and scanned at 10°C/minute to 375°C. Then they were cooled at the same rate to room temperature to remove any possible thermal history. The samples were reheated at 10°C/minute to 350°C with the heat flow measured versus temperature. The area of the melting peak was measured and the enthalpy of melting determined. Using the measured enthalpy of a pure Teflon material, the percentage of Teflon in the membrane was determined.

Samples for TGA analysis were sectioned and placed in a Perkin-Elmer TGA 7. These samples were weighed, heated at 20°C/minute to 950°C, then held at temperature for 60 minutes. One set of samples was heated at 20°C/minute to 950°C, held at temperature for 30 minutes, then cooled at 20°C/minute to 50°C to measure any oxidation of the catalyst. The samples were analyzed in an air environment to oxidize the polymer membrane and the weight loss versus time and temperature was recorded.

RESULTS

Water samples of the deionized water supply and of the condensate from the fuel cell were taken at periodic intervals during the cell test and were analyzed for fluoride, chloride, and sulfate using ion chromatography. The samples were examined for metals by ICP-ES. From the results in Table 1, there was no detection of any anions or metals in the initial deionized water sample. In fact, there was no detection of metals in any of the samples. The cathode sample taken on 27 December 1997 showed a trace of both chloride and sulfate, but no fluoride in the condensate. Subsequent anode and cathode samples taken from 6 January on, showed detectable levels of all three anions. The cathode results for fluoride exhibited an increase in concentration with time, reaching a maximum on 11 January 1998, then decreasing through the remainder of the test. The anode samples also increased in concentration levels, but due to the limited number of samples, it could not be determined if this trend continued. The chloride results for both the anode and cathode had trace levels, except for the anode sample dated 6 January 1998. There was no explanation for this sudden increase in concentration. The cathode results for sulfate showed levels that were approximately three times higher than the observed levels for chloride. Sulfate concentrations in the anode samples were significantly higher, 1.8 mg/l on 11 January 1998 and 1.1 mg/l on 22 January 1998. Again, there were insufficient data to determine any trend in the anode for sulfate concentration during the cell test.

Samples	F	CI	SO4	Cr	Pt	AI	Mn	Cu	Fe
Cumpico	(mg/l)								
Initial Water Sample	ND								
Cathode #3 12/27/97	ND	0.2	0.4	ND	ND	ND	ND	ND	ND
Cathode #3 1/6-1/7	1.1	0.2	0.5	ND	ND	ND	ND	ND	ND
Anode #3 1/6-1/7	0.3	1.9	0.1	ND	ND	ND	ND	ND	ND
Anode #3 1/11/98	1.2	0.2	1.8	ND	ND	ND	ND	ND	ND .
Cathode #3 1/11/98	1.6	0.1	0.2	ND	ND	ND	ND	ND	ND
Anode #3 1/22/98	1.7	0.1	1.1	ND	ND	ND	ND	ND	ND
Cathode #3 1/22/98	1.4	0.1	0.2	ND	ND	ND	ND	ND	ND
Cathode #3 1/26/98	0.9	0.1	0.6	ND	ND	ND	ND	ND	ND
Cathode #3 1/31/98	0.4	0.1	0.4	ND	ND	ND	ND	ND	ND

Table 1. Anion and Met	I Concentrations in Fi	uel Cell Condensate Samples
------------------------	------------------------	-----------------------------

(Note: ND = No Detection)

It was obvious from looking at the ion chromatography data that fluoride, chloride, and sulfate were somehow being introduced into the water during the fuel cell operation. Based on the structure of the Nafion membrane, which contains Teflon and sulfonic acid groups, there must have been some interaction with the membrane. The presence of chloride was interesting because there were no identifiable sources of contamination in the system. To determine if the membrane was the source of the fluoride contamination, three samples of the membrane—new, used anode, and used cathode—were analyzed by DSC for Teflon. Figure 1 shows typical DSC scans for all three samples and the Teflon standard. The samples and reference materials all contain well-defined melting peaks with an onset of melting occurring between 310 and 320°C.

Table 2 shows that the average percentage of Teflon in the used cathode sample was reduced by 31 percent and in the used anode sample, it was decreased by 59 percent. Comparing the used cathode sample, with a 31 percent decrease in Teflon concentration, to the used anode sample, with a 59 percent decrease in the Teflon concentration, showed that the used anode sample had a significantly larger Teflon reduction occurring during the cell test. It should be noted that the membranes used in the fuel cell test came from the same lot of material as the new membrane and that all the samples were sectioned using the same sampling procedure. There was significant scatter observed in the data within each sample set. It is not clear whether this is variation in the samples themselves or in the technique. However, the scatter in the melting enthalpy measurement for the standard was much lower than that observed in the samples, indicating a heterogeneous distribution of the Teflon on the membrane. Based on a sample size of 2.159 mm and a surface area (one side only) of 3.66 mm², the new membrane had a distribution of 5.60 mg/mm², the used anode had a distribution of 1.35 mg/mm², and the used cathode had a distribution of 2.29 mg/mm².

Sample	Peak	Onset Temp	Enthalpy	Sample Weight	Teflon	Average Teflon
	(°C)	(°C)	(Joules/g)	(mg)	(%)	(%)
New Membrane	317.0	310.8	2.83	5.00	13.43	
New Membrane	316.9	309.5	2.68	5.44	12.72	
New Membrane	317.2	310.2	2.83	5.47	13.43	•
New Membrane	325.9	319.2	1.86	5.77	8.82	12.1 ±2.21
Used Anode	317.0	310.1	1.05	3.49	4.98	
Used Anode	318.4	312.7	1.08	2.90	5.13	
Used Anode	318.0	311.7	1.45	3.58	6.88	
Used Anode	326.0	322.0	0.58	3.20	2.75	4.93 ±1.69
Used Cathode	318.5	310.9	2.44	3.82	11.58	
Used Cathode	318.5	311.5	1.96	3.28	9.30	
Used Cathode	318.4	313.1	1.26	4.14	5.98	
Used Cathode	326.2	319.5	1.39	3.82	6.60	8.37 ±2.58
Teflon Standard	321.9	315.3	22.17	7.08		
Teflon Standard	321.2	315.0	22.85	6.14		
Teflon Standard	315.2	307.6	20.00	8.08		PTFE STD
Teflon Standard	317.2	308.5	19.26	11.84		21.07 ±1.71

Table 2. Teflon Analysis by DSC

It is interesting to note from Table 2 that the weight for each sample (all samples were taken using the same diameter cork borer) fell into three distinct groups: new 5.42 ± 0.32 mg, used anode 3.29 ± 0.31 , and used cathode 3.77 ± 0.36 . This follows the same trend as the average percentage for Teflon.

Another point concerning the membrane was the initial distribution of catalyst on the surface and whether there was any change in this distribution that occurred during the fuel cell operation. Using TGA, samples were oxidized in an oxygen environment, heated at a constant rate, then held at constant temperature to try and reduce the metal oxide that formed during heating. The literature cites the oxidation of finely dispersed particles of metals, such as platinum, as they are heated in an oxygen environment with a subsequent loss of oxide as the temperature is increased, until eventually all the oxide disappears. Figure 2 shows that after oxidation of the membrane, the sample was slow cooled at 10°C/minute back to room temperature. At about 75 minutes, the catalyst began to oxidize as noted by an increase in the sample weight.

Figure 3 shows an overlay of typical plots of weight loss versus temperature for the new membrane, the used membrane, and the 30 percent catalyst sample. As seen in Table 3, the results for 30 percent catalyst and 60 percent catalyst powder showed reasonable agreement, at 27.27 percent for the former and 57.60 percent for the latter. Surprisingly, the sample of used membrane contained 4.65 percent catalyst, while the new membrane contained 2.99 percent. This is most likely because of method used to apply the catalyst. Comparing the sample weights, the used samples had a higher average sample weight at 3.93 mg than the new membrane, which had an average weight of 2.31 mg. The catalyst loading was 1.27 mg/mm² for the used membrane and 0.82 mg/mm² for the new membrane.

	30% Pt	Powder	60% Pow	Pt-Ru vder	New Membrane		Used Me	Used Membrane	
	Sample wt.	wt. %	Sample wt.	wt. %	Sample wt.	wt. %	Sample wt.	wt. %	
Sample 1	2.160	25.68	3.401	54.97	1.821	3.200	4.026	4.16	
Sample 2	4.694	28.29	3.140	59.67	2.330	1.028	4.634	4.06	
Sample 3	2.035	27.76	1.987	58.16	3.203	4.810	3.137	5.73	
Sample 4					2.321	2.710		•	
Sample 5					1.849	2.640			
Sample 6					2.330	3.580			
Average		27.27 ±1.38		57.60 +2.40		2.99 ±1.25		4.65 ±0.94	

^C Table 3. New and Used Membrane Analysis by TGA

CONCLUSION

Analysis of condensate samples taken from the fuel cell test showed positive results for fluoride, chloride, and sulfate, while the water supply showed no detectable levels of contamination. Since there was no indication of anions in the water prior to the test, it must be concluded that the anions were the result of some interaction between the water, gases, and membranes. The dissolved fluoride most likely is the result of some chemical interaction with the Teflon. This is supported by the DSC scans that showed a decrease in Teflon in the used samples. It is also curious that the used anode sample had less Teflon than the used cathode sample, possibly indicating some preferential reaction at the anode. The presence of sulfate probably is the result of some oxidation of the sulfonic acid group in the Nafion. At this time, there is no explanation for the presence of chloride.

The TGA results for catalyst concentration show good agreement for the 30 percent catalyst and 60 percent catalyst powder. The new membrane had a significantly lower catalyst concentration than the used membrane, which may indicate some need to refine the method for applying the catalyst. Based on experimental data, TGA and DSC provide useful methods for the characterization of a fuel cell membrane without the need for extensive sample preparation.

5

REFERENCES

- 1. Baird, Stuart, *Alternate Energy Review*, Ontario Hydro, 1991.
- 2. Wendlandt, W.W., *Thermal Analysis*, 3rd ed., John Wiley and Sons, New York, 1986, pp. 179-181.
- 3. Turi, A.A., *Thermal Characterization of Polymeric Materials*, Academic Press, Inc., Orlando, 1981.

6



Figure 1. DSC scan of membrane samples and standard.



Figure 2. Oxidation of membrane and catalyst.





TECHNICAL REPORT INTERNAL DISTRIBUTION LIST

NO. OF COPIES

1

1

1

2

1

1

1

5

3

1

1

1

CHIEF, DEVELOPMENT ENGINEERING DIVISION

ATTN: AMSTA-AR-CCB-DA

-DB 1 -DC 1 -DD 1 -DE 1

CHIEF, ENGINEERING DIVISION ATTN: AMSTA-AR-CCB-E

-EA -EB

-EC

CHIEF, TECHNOLOGY DIVISION ATTN: AMSTA-AR-CCB-T -TA -TB -TC

TECHNICAL LIBRARY ATTN: AMSTA-AR-CCB-O

TECHNICAL PUBLICATIONS & EDITING SECTION ATTN: AMSTA-AR-CCB-O

OPERATIONS DIRECTORATE ATTN: SIOWV-ODP-P

DIRECTOR, PROCUREMENT & CONTRACTING DIRECTORATE ATTN: SIOWV-PP

DIRECTOR, PRODUCT ASSURANCE & TEST DIRECTORATE ATTN: SIOWV-QA

NOTE: PLEASE NOTIFY DIRECTOR, BENÉT LABORATORIES, ATTN: AMSTA-AR-CCB-O OF ADDRESS CHANGES.

TECHNICAL REPORT EXTERNAL DISTRIBUTION LIST

	NO. OF <u>COPIES</u>	NO. COP	OF <u>IES</u>
DEFENSE TECHNICAL INFO CENTER ATTN: DTIC-OCA (ACQUISITIONS)	2	COMMANDER ROCK ISLAND ARSENAL	
8725 JOHN J. KINGMAN ROAD		ATTN: SIORI-SEM-L	1
STE 0944		ROCK ISLAND, IL 61299-5001	
F1. BELVOIR, VA 22060-6218		COMMANDER	
COMMANDER		U.S. ARMY TANK-AUTMV R&D COMMAND	
U.S. ARMY ARDEC		ATTN: AMSTA-DDL (TECH LIBRARY)	1
ATTN: AMSTA-AR-WEE, BLDG. 3022	1	WARREN, MI 48397-5000	
AMSTA-AR-AET-O, BLDG. 183	1		
AMSTA-AR-FSA, BLDG. 61	1	COMMANDER	
AMSTA-AR-FSX	1	U.S. MILITARY ACADEMY	
AMSTA-AR-FSA-M, BLDG. 61 SC) 1	ATTN: DEPT OF CIVIL & MECH ENGR	1
AMSTA-AR-WEL-TL, BLDG. 59	2	WEST POINT, NY 10966-1792	
PICATINNY ARSENAL, NJ 07806-5000		ILS ADMY AVIATION AND MISSILE COM	
DIRECTOR		DEDSTONE SCIENTIEIC INEO CENTER	2
		ATTN: AMSAM_PD_OB_R (DOCUMENTS)	4
ATTN: AMSPL DD-T BLDG 305	1	REDSTONE ARSENAL AL 35898-5000	
ABERDEEN PROVING GROUND, MD	. *		
21005-5066		COMMANDER	
	•	U.S. ARMY FOREIGN SCI & TECH CENTER	
DIRECTOR		ATTN: DRXST-SD	1
U.S. ARMY RESEARCH LABORATORY		220 7TH STREET, N.E.	
ATTN: AMSRL-WM-MB (DR. B. BURNS)	1	CHARLOTTESVILLE, VA 22901	
ABERDEEN PROVING GROUND, MD			
21005-5066			
COMMANDER			
U.S. ARMY RESEARCH OFFICE		·	
ATTN: TECHNICAL LIBRARIAN	. 1		

NOTE: PLEASE NOTIFY COMMANDER, ARMAMENT RESEARCH, DEVELOPMENT, AND ENGINEERING CENTER, BENÉT LABORATORIES, CCAC, U.S. ARMY TANK-AUTOMOTIVE AND ARMAMENTS COMMAND, AMSTA-AR-CCB-O, WATERVLIET, NY 12189-4050 OF ADDRESS CHANGES.

P.O. BOX 12211

4300 S. MIAMI BOULEVARD

RESEARCH TRIANGLE PARK, NC 27709-2211