AD-A182 211 A PULSED FIELD GRADIENT NUCLEAR MAGNETIC RESONANCE SPECTROMETER FOR DIREC (U) NORTHWESTERN UNIV EVANSTON IL D H WHITMORE 24 APR 87 AFOSR-TR-87-0847 1/1

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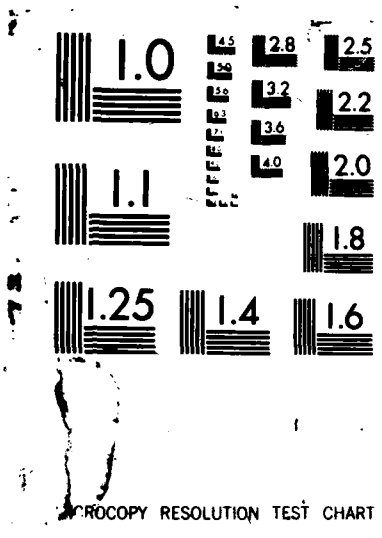
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ROCOPY RESOLUTION TEST CHART

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Whitmore, Donald Herbert

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A pulsed field gradient nuclear magnetic resonance spectrometer was successfully constructed which is based on a superconducting magnet and has magic-angle spinning capabilities. A dewar facility is an integral part of this system so as to permit investigation of ionic diffusion down to 77°K, along with a sample heating arrangement for diffusion measurements up to 300°C. This spectrometer has been employed in the measurements of protonic diffusion coefficients in β"-alumina solid electrolytes and cation, anion and self-diffusion coefficients in amorphous polymeric electrolytes.

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Final Technical Report

on

Grant No. AFOSR-85-0098

A Pulsed Field Gradient Nuclear Magnetic Resonance Spectrometer for Direct Measurement of Ionic Diffusion in Solid Electrolytes

Principal Investigator

Donald H. Whitmore

Northwestern University

Evanston, Illinois 60201

April 1987

Final Technical Report on Grant No. AFOSR-85-0098

We have successfully designed and constructed a working NMR pulsed field gradient (PFG) spin-echo spectrometer system for the direct observation of ion diffusivities in solid and amorphous electrolytes. A dedicated part of this system is a large-bore superconducting magnet installed in a low-loss helium dewar which was available for this purpose in Northwestern's Materials Research Center NMR Facility Laboratory. The moderate field of 40 KOe provided by this magnet represents a compromise between the needed working volume (a 4-inch diameter room-temperature bore) to accommodate pulse field gradient NMR apparatus and the requisite field strength to improve sensitivity. An inert gas flow cryostat furnace, designed for high-resolution NMR magnets by Oxford Instruments, is an integral part of our system; this unit includes controllers and transfer lines and permits measurements to be made over the temperature range from 77 to 600K.

The pulsed spectrometer electronics used in this new system were patterned after an earlier circuit design originated at Northwestern University [Gibson et al., Rev. Sci. Instr. 52, 1509 (1981)], but was complemented by a high-power, wide-band transmitter and a dedicated microcomputer for signal averaging. The pulsed field gradient electronics and the pulsing unit, constructed in our electronics shop following the original circuit design of Karlicek and Lowe [J. Mag. Resonance 37, 75 (1980)], allows gradient fields of 10 Oe/cm which is suitable for our new system

An important advance in techniques for the direct observation of diffusion coefficients in solids and liquids results from a combination of coherent averaging NMR methods and the pulsed field gradient approach In principle, the most powerful of these averaging methods is that of magic-angle spinning (MAS), a capability which we have incorporated into our PFG

spectrometer system. Our MAS probe is a custom-made unit with variable temperature capability which allows multifrequency solid-state NMR and permits relaxation measurements to be accomplished on less sensitive nuclei such as nitrogen in the fast proton conducting electrolyte ammonium hydronium 8"-alumina.

Using our new PEG spectrometer, we have to-date made diffusion coefficient measurements on: (a) some amorphous polymer electrolytes (D_{Li} and D_F in polyethylene oxide/$LiCF_3SO_3$ complexes); (b) protonic ammonium/hydronium 8"alumina and hydrogen uranyl glasses ($H_{5n-2}UO_2(IO_6)_n \cdot mH_2O$).

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