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**POSITRON STUDIES IN CATALYSIS RESEARCH
PROGRESS REPORT (SEPTEMBER 1993-APRIL 1994)**

AND FINAL REPORT FOR DE-FG02-90ER12103 from 1/15/90 - 1/14/94

Introduction

During the past eight months, we have made progress in several areas relevant to the eventual use of positron techniques in catalysis research. We have come closer to the completion of our positron microscope, and at the same time have performed several studies in our non-microscopic positron spectrometer which should ultimately be applicable to catalysis. These studies involve using depth-profiled positron spectrometries to observe the growth dynamics of metal silicides on silicon substrates and to observe defects in glassy polymers, and the use of bulk positron lifetime measurements to observe pore-size variations in zeolites. The current status of our efforts in each of these areas is summarized in a separate section following.

Construction of the Positron Microscope

For more than four years, we have been working to construct a second-generation positron microscope. This project is nearing completion, and we expect to be collecting our first microscope data by the end of the coming summer. The microscope has been designed to operate in a number of modes, and not all of these will be available at first, but we should be able to begin immediately on a number of very interesting extensions of studies we have begun on our existing positron spectrometer. While these studies are proceeding, we will continue to upgrade and improve the microscope, adding new functionality and additional modes of operation. We summarize below areas of microscope construction that have seen progress during the last eight months. For reference, we include as Figure 1 a schematic illustration of the microscope as a whole, with all of the ultimate operating modes that have been designed indicated.

Since September, our primary efforts in positron microscope construction have been in three areas. In all cases, work had already begun before this time period, and will continue thereafter. However, these were the areas that received the majority of our attention during the last eight months.

1. Optical Element Construction

The entire optical design for the microscope and much of the overall mechanical design was complete before September, but many of the detailed mechanical drawings of beam-optical components and mounting hardware needed to be generated. Most of these drawings are now complete, and we are in the process of transferring them to our machine shop for fabrication. Roughly half of the microscope is already built and has been assembled and tested. Both positron gun and electron gun sections have been tested, and the critical first remoderator is operating. These sections have been used to evaluate the overall design concepts used throughout the microscope. A number of modifications to the

Figure 1. Design of the Michigan Positron Microscope.

The main elements of the primary positron beam, the brightness-enhanced (remoderated) positron beam and the electron beam are shown. The different modes of operation indicated are: positronium formation fraction F_{ps} , Doppler broadened gamma ray spectroscopy (DBS), reemitted positron spectroscopy (RPS), scanning Auger electron microscopy (SAM), positron reemission microscopy (PRM), low energy electron microscopy (LEEM), positron annihilation lifetime spectroscopy (PALS) and scanning electron microscopy (SEM).

initial ideas were made as a result of our experience with these first sections, and these have been incorporated into the remaining drawings.

As of this writing, a number of additional modifications still remain to be finished, and these are our primary task during the next several months. In particular, we are redesigning a number of accessory components used to align the microscope optical elements, as well as making several changes in existing alignment components. In addition, we are re-designing several elements involved in making in-vacuum electrical connections to optical elements.

2. Data Collection Software

Significant progress was made during the past eight months in generating the custom computer software that will be required to operate the microscope. All designs for this software are complete and roughly one third of the actual code has been written. Unfortunately, we have had to postpone completion of this project for the time being to conserve manpower, and hence completion of the software will be delayed until after the bulk of the microscope hardware is finished. However, some initial measurements will still be possible using manual procedures and the actual software will be available roughly six months after we are able to return to this project

3. Electronic Sub-Assemblies

We have completed several of the many electronic power supplies and other sub-assemblies that the microscope will require during this period. Although this project has been given fairly low priority during this time, good progress was made nonetheless. By the beginning of the coming summer, we should have completed enough of the required electronic assemblies to permit the microscope to operate. Additional work on the remaining items can proceed in parallel with initial data collection.

Doppler Broadening Spectroscopy of Metal Silicides (See pages 4-5)

Positron Lifetime Spectroscopy of Glassy Polymers (See pages 6-7)

Positron Lifetime Measurements of Pore-Sizes in Zeolites (See page 8)

Depth-Profiled Positron Study of CoSi₂/Si. B.D.

WISSMAN, D.W. GIDLEY, W.E. FRIEZE, Dept. of Physics,
Univ. of Michigan.— Depth-profiled Doppler-broadening spectroscopy (DBS) of positron annihilation radiation is employed in the investigation of epitaxial cobalt silicide films grown thermally on Si substrates *in situ*. DBS is well-suited for the study of such systems due to its non-destructive nature, and the high sensitivity of positrons to defects. In an earlier positron re-emission experiment ¹ the positron diffusion length in the films was found to be short (of order 150 Å). This is indicative of a large rate of positron trapping either in the film or at the silicide-Si interface. In this experiment DBS is used to further investigate the nature of the positron trapping, and to study the diffusion and drift of positrons near the Schottky well and the associated depletion region. Preliminary results support the conclusion that the trapping rate at the interface is relatively small; thus the majority of positrons are trapping in defects in the film.

¹ B.D. Wissman, D.W. Gidley, and W.E. Frieze, Phys. Rev. B **46**, 16058 (1992).

- ☐ Prefer Poster Session
☒ Prefer Standard Session
☐ No preference

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Silicide Growth

$\text{CoSi}_2/\text{Si}(111)$

Suggested title of session
in which paper should be placed
Session in Glasses

Abstract Submitted
for March General Meeting of the
American Physical Society
21-25, March, 1994

Depth Profiled Positron Lifetime Spectroscopy of Glassy Polycarbonate. L.XIE, D.W.GIDLEY, Dept. of Physics, H.A.HRISTO, A.F.YEE, Dept. of Mat. Sci. & Eng., Univ. of Michigan*, Ann Arbor, Michigan 48109. --- A fast timing technique has been implemented in a variable energy positron beam. The mean positron implantation depth ranges from 50nm to 700nm. The positron lifetime spectra are collected by using secondary electrons produced by positron implantation as start signals and positron annihilation radiation (511 keV γ -rays) as stop signals. The time resolution is measured to be 0.5 nsec, and the 2 nsec lifetime component (τ_2) in polycarbonate is clearly resolved. The results show that τ_2 , its relative intensity I_2 and hence the hole volume fraction (F_h) are respectively 10%, 30% and 50% lower than the corresponding bulk values for positrons implanted close to the polymer surface, and approach the bulk values as the implantation depth exceeds 400nm. Molding process, moisture, gas residues and positronium diffusion cannot fully account for such a change. Our present interpretation is that the molecular chains near the surface are free to relax much faster toward the equilibrium state than those in the bulk, this produces a closer packed (in the direction perpendicular to the surface) molecular structure near the surface.

* Work supported by a grant from DOE, NO. DE-FG02-88ER45366.

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PS: τ_{0-Ps} , I_{0-Ps} vs. depth

PC: τ_{0-Ps} , I_{0-Ps} vs. depth

Positron Lifetimes in Zeolites

Zeolite Name	o-Ps lifetime, intensity		PALS Diameter (Å)	Specified Diameter (Å)
	Before baking	After baking		
hy	2.67ns, 3.70%	5.77ns, 2.43%	13.5	13-16
3A	1.47ns, 5.74%	4.02ns, 7.28%	11.8	11
nh4	1.94ns, 5.15%	4.77ns, 6.32%	12.6	12
hzsm	2.62ns, 1.98%			6.5 (channel)
4A	1.51ns, 8.26%	4.17ns, 10.21%	12.0	11
unknown	2.61ns, 11.69%	4.71ns, 13.95%	12.5	unknown

Notes:

1. These data were acquired last summer (1993) using one of our bulk positron lifetime spectrometers. It is used as a calibration of our PALS analysis model to determine hole size diameter.

2. Baking was performed in air @400°C.

3. Error bars are about 0.02nsec for o-Ps lifetimes, 0.05% for the intensities.

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