

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

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1. AGENCY USE ONLY (Leave blank)

2. REPORT DATE

June 15, 1994

3. REPORT TYPE AND DATES COVERED

Final Report 1 Sep 93-31 Aug 94

4. TITLE AND SUBTITLE

Scanning Tunneling Microscopy/Atomic Force
Microscopy for Study of Nanoscale Metal Oxide Particles
(Destructive Adsorbents)

5. FUNDING NUMBERS

DAAH04-93-G-0457

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REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

U.S. Army Research Office
P.O. Box 12211
Research Triangle Park, NC 27709-221110. SPONSORING/MONITORING
AGENCY REPORT NUMBER

ARO 32398.1-CH-DRS

11. SUPPLEMENTARY NOTES

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12a. DISTRIBUTION / AVAILABILITY STATEMENT

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12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

A modern scanning tunneling microscopy/atomic force microscopy instrument was purchased. This instrument is allowing physical characterization of nanoscale particles of metal oxides. These particles are known to be effective reagents for the destruction of military toxins as well as chlorocarbons by a process we call destructive adsorption.

DTIC QUALITY INSPECTED 6

94 7 12 06 0

14. SUBJECT TERMS

Nanoscale particles, destructive adsorption, atomic force
microscopy, scanning, tunneling, scanning probe

15. NUMBER OF PAGES

9

16. PRICE CODE

17. SECURITY CLASSIFICATION
OF REPORT

UNCLASSIFIED

18. SECURITY CLASSIFICATION
OF THIS PAGE

UNCLASSIFIED

19. SECURITY CLASSIFICATION
OF ABSTRACT

UNCLASSIFIED

20. LIMITATION OF ABSTRACT

UL

AD-A281 417

94-21306

Scanning Tunneling Microscopy/Atomic
Force Microscopy for Study of Nanoscale
Metal Oxide Particles (Destructive Adsorbents)

Final Report

June 14, 1994

U.S. Army Research Office

DAAHO4-93-0457

Kansas State University
Department of Chemistry
Manhattan, Kansas 66506

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DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
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Distribution/	
Availability Codes	
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I. Introduction

The development of solid reagents that adsorb and simultaneously destroy toxic substances is important for air purification and as an alternative to incineration. Toxic chemicals such as organophosphorus, -halogen, -sulfur, and nitrogen compounds are not very amenable to treatment by high performance catalytic processes since these heteroatoms are notorious for catalyst poisoning.

Solid reagents that might serve as effective destructive adsorbents must have high capacity, and convert the toxic materials to non-toxic substances. Ideally, the heteroatoms (P, Cl, Br, N, S) would be extracted from the organic parts of the molecules and "mineralized" (converted to an innocuous metal salt).

In attempts to synthesize and develop such destructive adsorbents, we have concentrated our efforts on nanoscale metal oxides. The development of the most efficient solid reagents depends on our ability to prepare ultrafine powder with very high surface areas and intrinsic surface reactivities.

This research direction leads us into the exciting field of nanostructured materials, which possess novel and hybrid properties between molecular and bulk solid limits. Metal and semiconductor nanoscale materials have already been demonstrated to possess unique magnetic, optical, and physical properties. We are contributing to this field by studying the unique surface chemistry of nanoscale insulator particles.

From these points of view, we believe that continued work on the chemistry of such particles has importance both to basic and applied science. Further understanding of their synthesis, properties, and chemistry and their development into useful materials is the driving

force behind the prior and proposed work described herein.

As an important aspect of characterizing these nanoparticles, a scanning tunneling microscope/atomic force microscope. (STM/AFM) was purchased. This instrument allows observation of the particles before and after use as destructive adsorbents. Morphological properties such as surface defects are believed to be responsible for the chemical reactivity with toxic chemicals (chemical agents, chlorocarbons, etc.). In order to understand and correlate defect types and concentrations with reactivity, STM/AFM experiments are now in progress.

II. Instrument Purchased

The STM/AFM instrument, model SPM 30, was purchased from WYCO corporation. The instrument consists of four major components: the microscope unit, the scanning probe interface, the stage/viewfinder controlled, and the computer system. To support the use of the microscope unit, a few necessary accessories were purchased, too. Those are two piezoelectric scanner modules of different scan-ranges, STM probe , AFM probe, probe holders, and cantilever carriers. An ink-jet printer and a color video copy processor were purchased from Hewlett Packard and Mitsubishi, respectively, to support the outcome of the resulting images.

III. Preliminary Results

Three MgO powder samples with different surface area and surface morphology were studied by AFM. The surface area was 37, 241 and 325 m²/g, for samples A, B, and C, respectively. Those samples showed very different reactivity on the decomposition of chlorinated carbon components, which is speculated to be related to a different surface morphology. The fact that the samples were very fine powders gave a few technical difficulties in carrying out the AFM measurements, which were overcome by pressing the powder into a pellet at a pressure of 12000 psi. Figure 1 shows the surface morphology of the samples obtained by AFM. As apparent from the micrograms, samples have dramatically different surface morphology. Sample A consists of plates which are ~200 nm wide and ~50 nm thick stacked together along one direction. The alignment of the plates is speculated to be caused by pressure applied during the preparation of the pellet. The sample C consists of spheres of a diameter of ~100 nm. The surface of the sample C is rougher than sample A. Sample B is rather similar to A, though the sample also had a feature of sample C: in some section of the pellet, spheres were observed mixed with the plates. These AFM observations are consistent with TEM studies carried out previously on similar samples, which revealed two different kinds of particles: sphere and plates. The difference in the morphology suggests that the identity of the most exposed crystalline facet and possibly of defects are not identical for the MgO samples. An effort to try to identify the facets of the particles is underway by carrying out the AFM measurements at atomic resolution.

IV. Students, Postdoctorals, and Research Associates Involved

Olga Koper, current Ph.D. student

Dong Park, postdoctoral research associate

Shawn Decker, current Ph.D. student

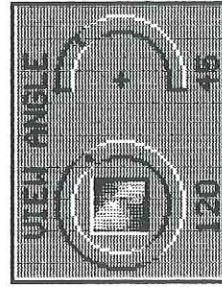
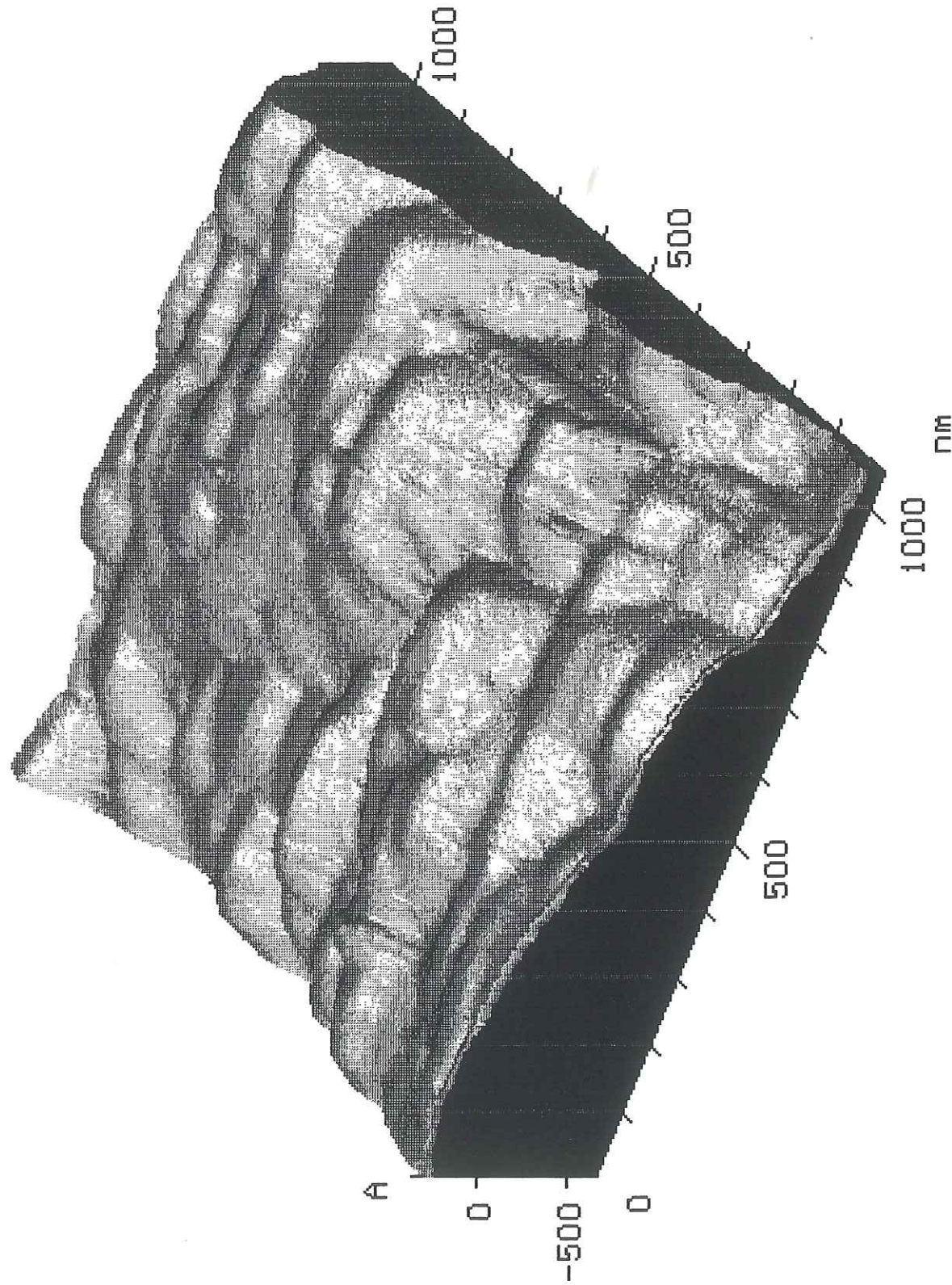
Yan Jiang, current M.S. student

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Surface Plot

May 23, 1994

17:31:54



>>Ak40.AFM

Time: 13:04

Jun 06, 1994

Zoom: 1.00

HYKO SPM

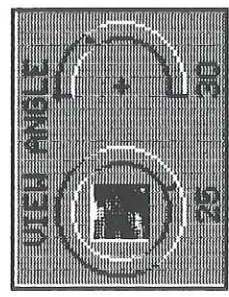
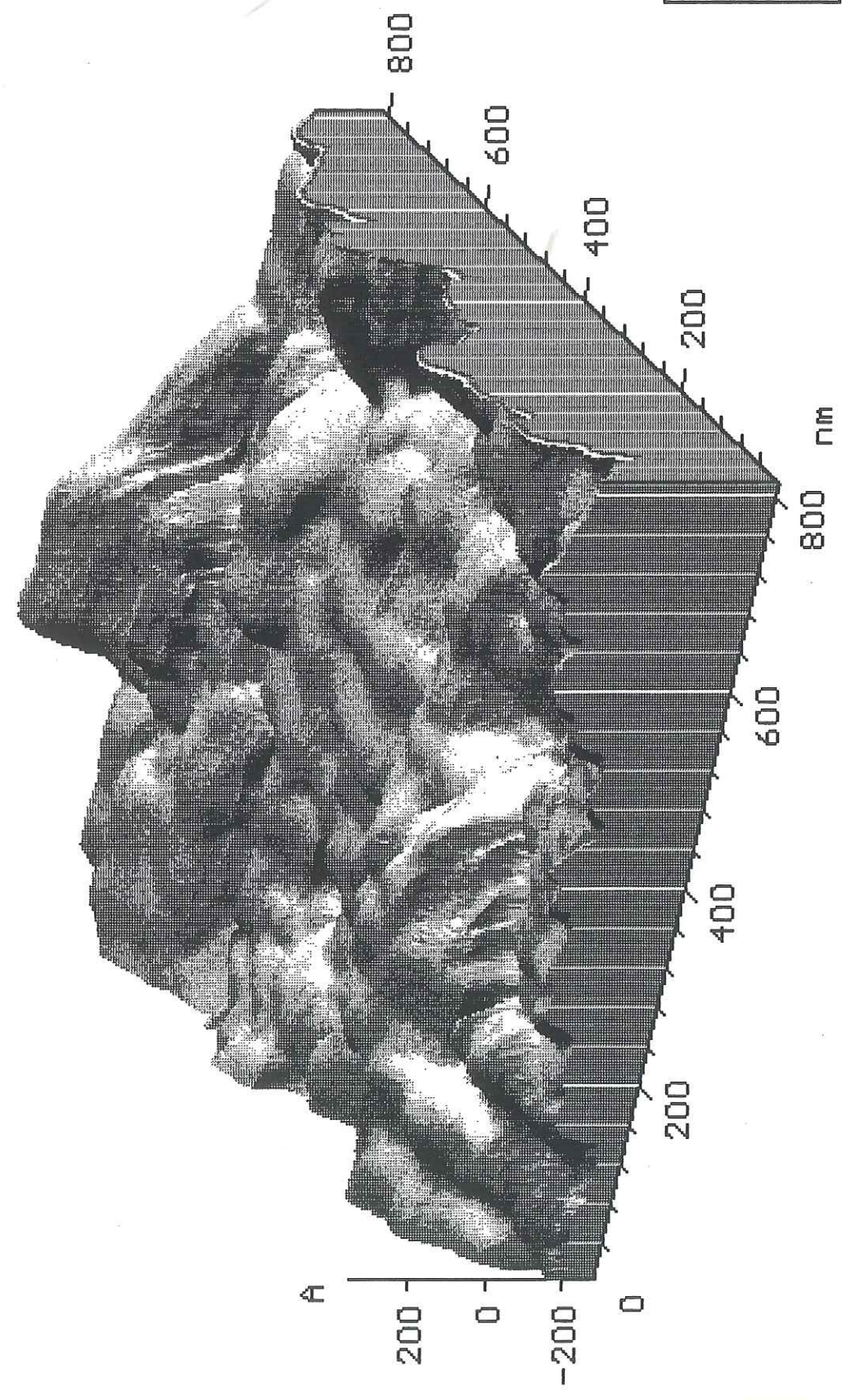
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Surface Plot

May 06, 1994

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May 06, 1994

Zoom: 1.00

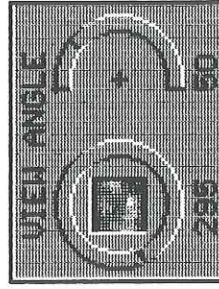
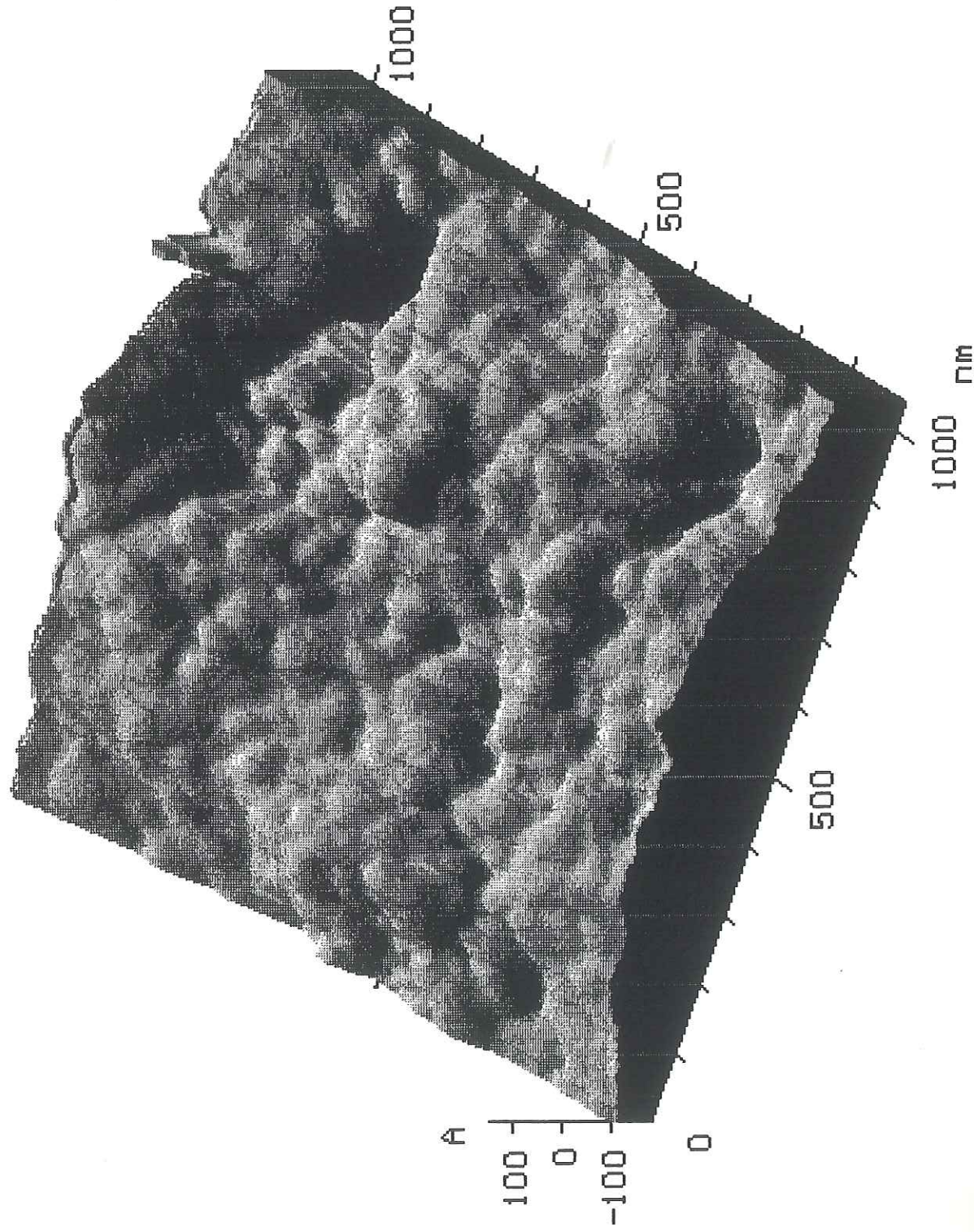
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Surface Plot

May 09, 1994

14:03:16



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Time: 13:27

Jun 06, 1994

Zoom: 1.00

HYKO SPM