

SAN097-1841C
SAND--97-1841C I-18

Microbeam RBS on Flat Panel Displays

CONF-961120--3
RECEIVED

JUL 30 1997

OSTI

H. Schöne, D. Walsh, R. T. McGrath, J.H. Burkhart

Sandia National Laboratories, Albuquerque NM 87185

Abstract:

We have demonstrated the utility of microbeam - Rutherford Back Scattering (μ -RBS) in spatially resolved studies of operational plasma effects on the interior surfaces of plasma flat panel displays manufactured by Photonics Imaging. The experiments were performed at the Sandia Nuclear microprobe using a 2.8 MeV He beam with an average beam spot size of less than $8\mu\text{m}$. The interior surface of the top panes of the flat panels is composed of approximately 800 nm of MgO on top of a 2000nm thick PbO layer. μ -RBS of sample panels operated under varying conditions measured changes in the surface MgO film thickness due to plasma erosion and redeposition as accurately as ± 1.5 nm. The high accuracy in the MgO thickness measurement was achieved by inferring the MgO thickness from the shift of the Pb front edge in the RBS spectrum. An estimate for the thickness accuracy as a function of the acquired statistics is presented. The surface of the flat panels' bottom panes is also comprised of MgO on top of PbO. However, troughs $\sim 100\mu\text{m}$ wide by $10\mu\text{m}$ deep were partially filled with phosphor and cover the entire width of the surface. This leaves only $100\mu\text{m}$ long sections of MgO within the trough exposed. Using μ -RBS, we were able to analyze the surface composition of these regions.

1 - Introduction:

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

ng

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, make any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Gas plasma displays are low cost due to their simple construction and the materials used. They exhibit high brightness, good efficiency, high resolution at large sizes. These attributes along with their slim form factor, truly flat display area and distortion free image have made gas plasma displays the preferred large area flat panel display type. The display life time is typically tens of kilohours, but can be severely shortened by plasma sputter erosion of the display's surface. The erosion is believed to occur in the high field gradient area of the pixel. The size of the eroded area is on the order of the pixel size ($60 \times 60 \mu\text{m}^2$ or smaller). The poor spatial resolution of RBS using a "normal" beam spot size of 1 mm or more, precludes studying the effects and extent of the erosion and redeposition of the surface material by producing, in effect, an averaged measurement from regions of both erosion and redeposition. While optical micrographs and SEM images will show changes in the surface topology, they can not determine MgO thickness. Moreover, RBS is more sensitive to small, but operationally significant, contamination of the surface MgO than SEM. The results presented here demonstrate the feasibility of nuclear ^{k-beam} ~~beam~~ ^{beam} RBS studies for obtaining the spatially resolved film thickness - measurements required to elucidate the effects of operating conditions on the pixel erosion speed and redeposition patterns of flat panel displays. V

The basic construction of the tested ac gas plasma flat panel display manufactured by Photonics Imaging consist of two glass panes, each having a linear array of typically 40 - $60 \mu\text{m}$ wide electrodes spanning the whole pane. The electrode side of the panes is coated with a dielectric (PbO) and topped with a $\sim 700 \text{ nm}$ thick MgO film, which is an efficient electron emitter. Both panes are stacked together with electrodes facing and rotated by 90 degrees, forming a grid of crossing electrodes. The panes are fused together

with a spacing of $\sim 100\mu\text{m}$, evacuated and back-filled with a high Z noble gas. A cross sectional sketch of a generic gas plasma flat panel is shown in figure 1. The bottom pane surface of color plasma displays is different in that it has $\sim 100\mu\text{m}$ wide by $\sim 10\mu\text{m}$ deep troughs parallel to the direction of the electrodes over the entire surface. Parts of the troughs are filled with phosphor, leaving exposed only the pixel area that is defined by two crossing electrodes.

2 - Experiment:

A 2.8 MeV He ion beam was electrostatically scanned across a $250 \times 250\mu\text{m}^2$ surface area. The beam position and the measured recoil energy were recorded in list mode. The beam spot was typically smaller than $8\mu\text{m}$ in diameter with an intensity of about 10 pA requiring exposure times of up to 6 hrs. for a single 2D RBS map. Recording the data in list mode allows us to analyze the spectra off-line for beam induced signal changes over time. However, no changes of the MgO film as a function of exposure time were observable. The targeting system of our RBS setup allows us to place the ion probing beam with an accuracy of $\pm 10\mu\text{m}$; quite sufficient to identify the optically observed features (e.g., electrode or pixel) with features in the RBS image.

The MgO surface thickness was determined by observing the apparent energy change of the He projectiles recoiling of the PbO front edge. ^{Fig.} Figure 2 illustrates an example of the shift in the lead front edge in the RBS spectrum for different He strike locations. The spectra depicted were taken on the front pane of panel #1 in the panel region operated in mode "A". The curve labeled "pixel pit" represents the RBS spectrum for ions striking the crossing points of two electrodes within panel region "A". All the events within this

10x12 μm^2 large area is summed up to improve statistics. The curve labeled "non eroded surface" corresponds to an 250 x 160 μm^2 area more than 20 μm away from any pixel location. Label "re-deposition area @ pixel" corresponds to a 10 x 10 μm^2 region immediately above or below the pixel area.

Even though the shift of the front edge is distinctive, the statistics is poor for small surface areas and will limit the accuracy of the front edge determination. The ideal RBS spectrum front edge shape for a thick (thickness $\gg \delta_x$), single element target sample is that of an error function. δ_x is the depth resolution and is defined as :

$$\delta_x := \delta E_{\text{total}} / (dE/dx) = [(\delta E_B)^2 + (\delta E_D)^2 + (\delta E_{St})^2 + (\delta E_{SA})^2]^{1/2} / (dE/dx)$$

Here, δE_{total} is the total energy resolution of the RBS system (in our case 30 keV), dE/dx is the rate of energy loss. Under the assumption all the individual uncertainties are normally distributed, the contributions to the overall system resolution add in quadrature. δE_B is the beam energy uncertainty, δE_D the detector resolution, δE_{St} the energy straggling, δE_{SA} the solid angle contribution and δE_R the surface roughness. In our case, the only significant contributions to δ_x are the detector resolution and the energy spread associated with the solid angle. The beam energy resolution is better than 10^{-4} , the surface roughness in an area comparable to the beam spot size of $\sim 5\mu\text{m}$ is in most cases negligible.

It is implied, that the rate of energy loss change is much smaller than the energy resolution ($dE^2/dx^2 \ll \delta_{\text{total}}$) as is the case for most RBS studies. Then, the location of the surface is at the half-height point of the step and can be determined exactly for an error function. The accuracy of determining the surface location in a measured spectrum, however, is limited by the acquired statistics N and the position resolution δ_x . To estimate

how well we can locate the surface position as a function of the acquisition statistics N (N is the 90% height of the front edge step), we will differentiate the error function. The material front edge is going to be at the maximum or centroid of the resulting normal distribution. The problem of calculating the surface position has now been transformed into how well we can determine the centroid of this distribution. The standard way to see if normal distribution 1 with a centroid C_1 and a width of δ_{x1} is significantly different from distribution 2 with a centroid C_2 and a width of δ_{x2} can be written as:
$$z = \frac{C_1 - C_2}{\sqrt{\delta_{x1}^2 / N + \delta_{x2}^2 / N}}$$

If we need to test if two positions C_1 and C_2 from the same normal distribution are equally qualified to be the centroid of the distribution, the formula simplifies to:

$$z = \Delta C / \delta_x \sqrt{N/2} \quad ; \quad \Delta C = |C_1 - C_2|$$

The confidence level α , that the hypothesis $H_0: C_1 = C_2$ is given by: $1 - 2\phi(z) = \alpha$

with:
$$\phi(z) = \frac{1}{\sqrt{2\pi}} \int_0^z e^{-\frac{1}{2}x^2} dx.$$

We have used the estimates outlined above to ascertain the depth uncertainties reported in table 1 are for a 95% confidence level.

Closer examination of all the RBS spectra collected (see Figure 2) reveals a shoulder for the spectra of the non eroded and re-deposition areas that extends from the respective lead edges to the energy of surface lead. The shape, energy range and location of the shoulder strongly suggest an elemental contamination of Tl, Pb or Bi throughout the MgO film. Within our energy resolution, we can exclude all other elemental contributions. Experimental errors, such as pile-up or backscattering from surfaces other than the sample can also be excluded. This MgO contamination can not be seen with broad beam RBS

because the surface roughness term δE_R would have masked this very low but distinctive step. If we assume, that the MgO film is more or less uniformly contaminated with Pb (about 2% of the PbO substrate concentration) the reported MgO thickness has to be reduced accordingly by about 3.5% to 5%.

3 - Results and discussion

a) Top panels

Figure 3 shows the MgO thickness for panel region "A" through "D" of panel 1 calculated from the 2D RBS spectra and subsequently smoothed over an area equivalent to the lateral position resolution ($8\mu\text{m}$ or 2 lateral grid points) to reduce noise. Each of these four panel regions correspond to different operational conditions and are expected to have varying erosion patterns. The right side of Figure 3 shows optical microscope photographs of areas similar to the corresponding RBS area. While the flat panel region photographed is not identical to the RBS analysis area, it is from the same operational region and of the same size as the RBS analysis area.

The pixel area in panel 1 "A" and "B" as seen under the microscope, have very pronounced edges and ridges that appear "wrinkled" and elevated. While not visible in the images, sharp lines (they appear to be cracks) formed around the periphery of each pixel and extend along the axis of the electrodes from one pixel to the next. These features suggest strong sputter erosion and/or thermal stress. Panel regions "C" and "D" exhibit less pronounced ridges around the pixels than panel regions "A" or "B" and no crack like lines are visible.

As can be seen best from the color bar at the bottom of Figure 3, the surface areas for panel regions "A" through "D" in the MgO thickness plot (excluding the immediate pixel

area) are between 540 nm and 715 nm thick. The accuracy of the depth determination for a single grid point in the RBS spectra is in most areas about ± 17 nm or better. The numbers indicated on the MgO thickness plots correspond to areas that have been identified as self similar Regions Of Interests (ROI). For example, panel 1 panel region "A" ROI 1 is identified as a largely featureless surface that is distant to any pixel erosion area. All the events in this ROI are summed for better statistics and subsequently used to determine the average MgO surface thickness in this area. ^{Table I} Table 1 lists all the ROIs, the calculated average MgO thickness for this ROI and the area in μm used for this ROI. The number of channels is an indication of the relative statistics. The error cited is the 95% confidence value for the uncertainty in the depth calculation. The surface thickness of panel region that has not been operated ("unaged") was determined to be 474 nm (see ^{Table I} table 1). The very pronounced and almost rectangular depressions in the MgO thickness are located exactly at an optically determined pixel position. The entire MgO film has been eroded away from the pixel panel regions "A", "C" and "D". Panel region "B" has eroded all but 200 nm of the MgO film. Also, note that the scanned area for panel region "B" was only $125 \mu\text{m} \times 125 \mu\text{m}$. This result was not expected as the optical images suggest a stronger erosion from areas "A" and "B" than for "C" and "D". On average a MgO film 100 nm thicker than the surface was measured just above and below the pixel areas for all four panel regions. This elevation is interpreted as a redeposition area for the sputtered MgO. The optical images identified these areas as "wrinkled" and elevated ridges surrounding the pixel. The measured ridge thickness does not correlate well with the optical images and suggests that the strong wrinkles may be formed in the underlying PbO.

The lead concentration within the MgO film in this ridge area is similar to the concentration found in the overall MgO film ($< 5\%$).

V Figure 4a shows on the left side the MgO thickness plot for panel 3, panel region "A" and on the right side the corresponding microscope photograph. The MgO erosion patterns are very similar to panel 1. The MgO thickness outside the pixel area is 525 nm and only 20 nm in the center of the pixel. The area above and below the electrode is elevated by about 100 to 150 nm. The only observable difference to panel 1 is the broader electrode ($\sim 80 \mu\text{m}$ instead of $60 \mu\text{m}$). The patterns visible in the optical micrograph are comparable to the patterns observed for panel 1 "C" and "D" (little wrinkling and no crack lines). The optical photographs of panel 3 B (not shown) show clearly visible patterns similar to panel 1 C, D and panel A. However, the RBS spectra did not exhibit any structure.

b) Bottom panels

The convoluted topographical structure and the varied layering of MgO and Phosphors of the bottom panes make the interpretation of the RBS spectra difficult at best. Hence, in contrast to the top panel images, Figure 4b shows on the left side only the relative energy shift of the lead front edge in arbitrary units. The primary purpose of the 2D RBS image is to identify topographical areas such as ridges or troughs and to obtain location and a qualitative thickness measure of the phosphorous layers and MgO in the center of a trough. Again, the micrographs shown immediately to the right of each 2D RBS image show a similar panel area of approximately the same size.

The optical image of the bottom pane of panel 3, panel region A clearly shows 3 phosphor patches (diffuse white areas) at the bottom and the edges of 3 more patches at

the top. The 2 speckled, lightly colored, lines running from the top to the bottom through the entire image are the ridges separating the troughs filled with phosphor. The bright white line connecting the phosphor patches is the subsurface electrode. Note, that the electrode is offset from the center of the trough by roughly 20 μm . There is no visible evidence of damage or erosion in any of the optical images. Careful optical inspection showed, that all the phosphor appears to be concentrated on the bottom of the trough. Using the depth of focus of the microscope, we tentatively determined a phosphor thickness of $\sim 5\mu\text{m}$.

The $\sim 100\mu\text{m}$ wide and reddish colored area in the 2D plot of the PbO front edge energy, clearly identifies the phosphor free trough area. The deep blue and purple areas extending perpendicular from the wide reddish area can be associated with phosphor filled troughs. These troughs are separated by ~ 10 to $15\mu\text{m}$ wide yellow/red lines depicting the ridges between the troughs. The ridges between the troughs are most visible between the phosphor covered troughs. As the ion beam moves away from the top of a ridge, the beam incidence becomes oblique as it strikes the sides of the ridges and results in an overestimate of the MgO film thickness. Ions striking the bottom of a trough are of normal incidence and provide meaningful MgO thicknesses. Ions scattered from the phosphor free troughs are of highest energy (red) and may show a thinning of the MgO. The energy of the ions scattered off the PbO at the phosphor filled troughs is lower (purple) because the ions lose energy passing through both the MgO film and the phosphor layer. The collection statistics for this bottom panel 2D images is much reduced (by 1/4) over the top panel spectra and the depth resolution is reduced accordingly.

Once we were able to identify the different regions in the RBS image, we stopped the beam raster and moved the ion beam to a selected and fixed point in that image (positioning accuracy is better than $1\mu\text{m}$) to take a spectrum with high statistics (dubbed point & shoot). The size of the beam spot is again roughly $8\mu\text{m}$. The numbers in the 2D RBS image depict the various point & shoot locations. We attempted to measure the relative elemental concentration in a phosphor covered area (e.g., Location 1 of panel 3 region A). Unfortunately, the large number of different elements and the presence of the very strong lead signal prohibited even a qualitative determination. This made it impossible to calculate a meaningful MgO thickness for this particular point. Point & shoot positions 2 and 3 are at the bottom of the phosphor free troughs. Point 2 has a 30nm and point 3 a 90 nm thick MgO layer indicating sputter erosion of the MgO.

The same 2D RBS spectra were taken for panel 3 region A' (MgO on top of a phosphor layer on top of MgO) and for panel 1 region A. Again, there seems to be at the center of the phosphor free troughs an area that is thinner than the ridges that separate the troughs. Unfortunately, a software problem prevented us from targeting this apparently thinner MgO area. The MgO thickness for panel 3 A' at location 1 is 365 nm and 320 nm for location 2. In order to calculate the MgO thickness it was assumed that between the phosphor patches there is no Phosphor sandwiched between the MgO films.

The micrograph of bottom panel 1, panel region A reveals that in contrast to panel 3, the electrode is well aligned with the troughs. The phosphor seems to fill the bottom of the troughs. The center of the phosphor free area once again seems to suggest a thinner MgO film. The point & shoot location 2 indicates a thickness of 52nm.

Conclusion:

We have demonstrated the utility of microbeam Rutherford Back Scattering (μ -RBS) for spatially resolved studies of contamination and operational plasma effects on the interior surfaces of plasma flat panel displays. Changes in the surface MgO film thickness due to plasma erosion and redeposition were measured as accurately as ± 1.5 nm. An estimate for the thickness accuracy as a function of the acquired statistics was presented.

References:

Figure captions:

Figure 1: Sketch of generic flat panel cross section

Figure 2: Sample RBS spectra for different areas of panel 1. The shift in the front edge of PbO is clearly visible. The step extending from the surface lead position to the various subsurface lead edges indicates a probable lead contamination.

Figure 3: 2D maps of MgO thickness determined with 2.8MeV He RBS for 4 different operational conditions "A","B","C" and "D" of panel 1. Optical images are from panel regions corresponding the measured RBS regions. Numbers indicate ROI's that are tabulated in table 1.

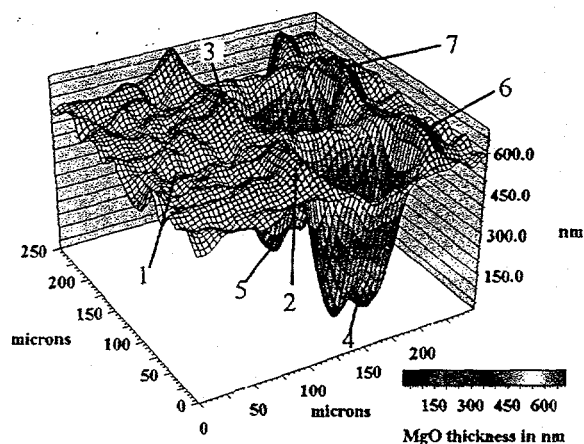
Figure 4: a) 2D map of MgO thickness determined with 2.8MeV He RBS for one section of panel 3. Optical image of Panel region corresponds to measured RBS region. b) Left side shows the relative energy shift of the lead front edge in arbitrary units for the bottom panes of panel 3 region "A", "A' " and panel 1 region "A". Optical microscope images of the corresponding region are shown on the right hand side.

Panel # 1, top pane							
area	ROI	upper left corner [μm]	lower right corner [μm]	# of channel	Comment	MgO [nm]	error [nm]
A	1	0,160	250,250	1536	area to right of electrode	571	± 1.1
A	2	59,129	90,137	27	bottom right pixel hump	640	± 8.5
A	3	164,133	200,140	30	top right pixel hump	606	± 8.0
A	4	74,47	98,55	21	top left pixel hump	658	± 9.5
A	5	176,59	200,63	14	bottom left pixel hump	667	± 11.3
A	6	59,78	90,98	54	bottom pixel pit	29	± 8.0
A	7	164,90	200,109	60	top pixel pit	20	± 5.6
A	8	0,0	250,35	576	above electrode	571	± 1.8
B	1	0,156	250,250	1600	area to right of electrode	715	± 0.7
B	2	117,121	168,129	182	right pixel hump	814	± 2.1
B	3	137,59	160,90	63	pixel pit	223	± 3.6
C	1	0,176	250,250	1280	area to right of electrode	553	± 1.2
C	2	59,94	39,109	35	bottom pixel pit	30	± 7.4
C	3	125,106	156,117	36	center pixel pit	34	± 7.3
C	4	230,109	238,121	12	top pixel pit	82	± 12.6
C	5	16,55	35,63	18	bottom pixel hump	645	± 10.3
C	6	133,59	164,74	45	center pixel hump	658	± 6.5
C	7	0,0	250,39	640	area to left of electrode	544	± 1.7
D	1	0,0	250,78	1280	area left of electrode	546	± 1.9
D	2	35,94	74,102	33	bottom left pixel hump	667	± 11.6
D	3	138,94	180,105	36	center left pixel hump	640	± 11.1
D	4	47,125	74,137	32	bottom pixel pit	1	± 11.8
D	5	145,133	176,141	27	center pixel pit	10	± 12.8
D	6	39,160	66,168	54	lower right pixel hump	624	± 9.1
D	7	145,160	176,168	18	center right pixel hump	615	± 15.7
D	8	0,195	250,250	960	area right of electrode	537	± 2.1
unaged		0,0	250,250	4096	no structure, entire surface	474	± 1.6
Panel # 2, top pane							
A	1	0,0	250,70	1152	area to left of electrodes	526	± 1.9
A	2	35,105	63,113	24	lower left pixel hump	631	± 13.4
A	3	145,105	168,113	21	center left pixel hump	624	± 14.3
A	4	43,145	55,180	40	bottom pixel pit	25	± 10.4
A	5	148,145	164,168	35	center pixel pit	20	± 11.1
A	6	27,207	63,215	30	lower right pixel hump	674	± 12.0
A	7	148,211	176,219	24	center right pixel hump	683	± 13.4
unaged		0,0	250,250	4096	no structure, entire surface	495	± 1.4
B	-	0,0	250,250	4096	no structure, entire surface	528	± 1.1

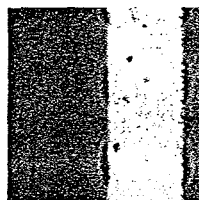
Table 1: Summary of MgO thickness measurements shown in figure 4 and 5a with 95% confidence value for the error in the depth measurement.

2D RBS maps using 2.8 MeV He

MgO thickness

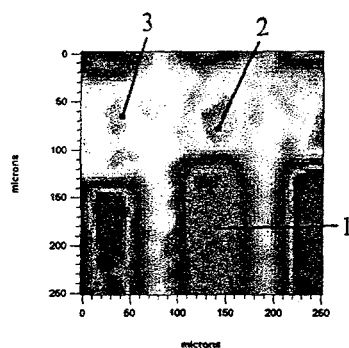


optical image
of RBS area

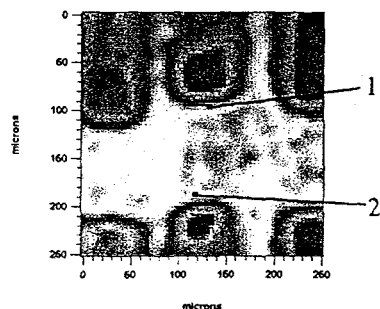


Panel 2
Region A
top pane

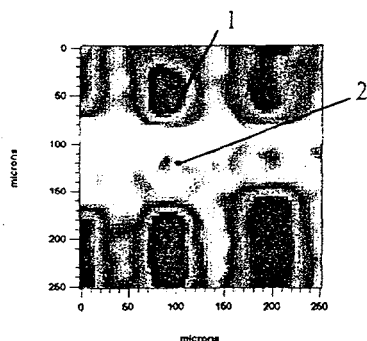
a)



Panel 3
Region A
bottom pane



Panel 3
Region A'
bottom pane



Panel 1
Region A
bottom pane

b)

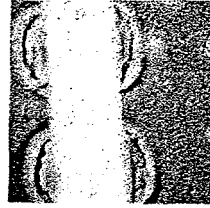
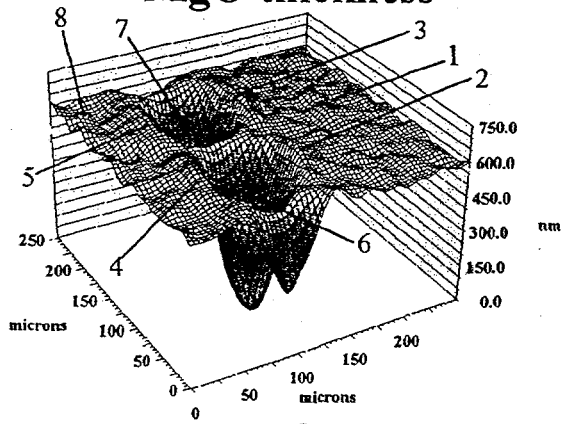
Fig 6

Fig 5

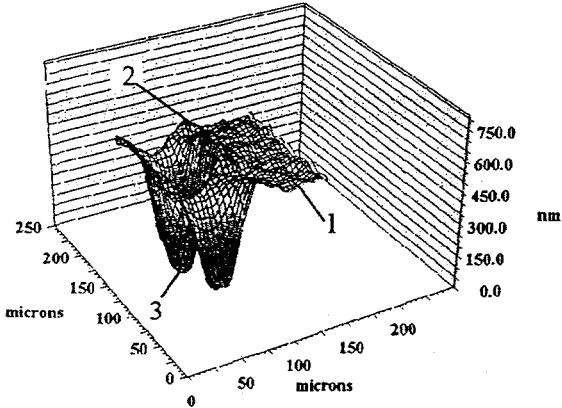
2D RBS maps using 2.8 MeV He

MgO thickness

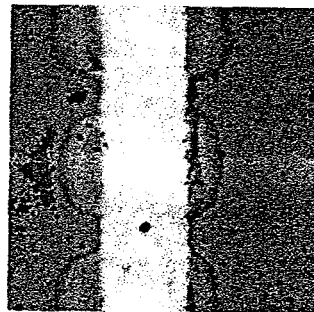
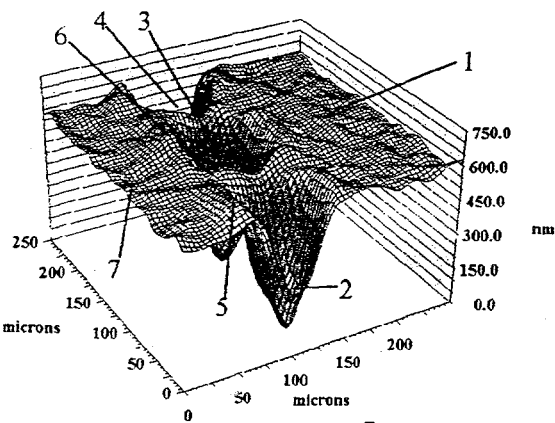
optical image
of RBS area



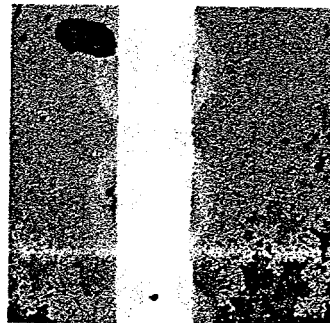
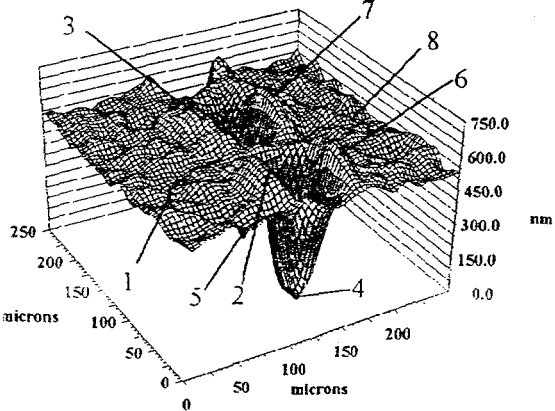
Panel 1
Region A
top pane



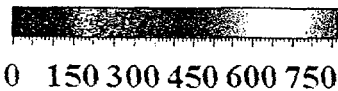
Panel 1
Region B
top pane



Panel 1
Region C
top pane



Panel 1
Region D
top pane



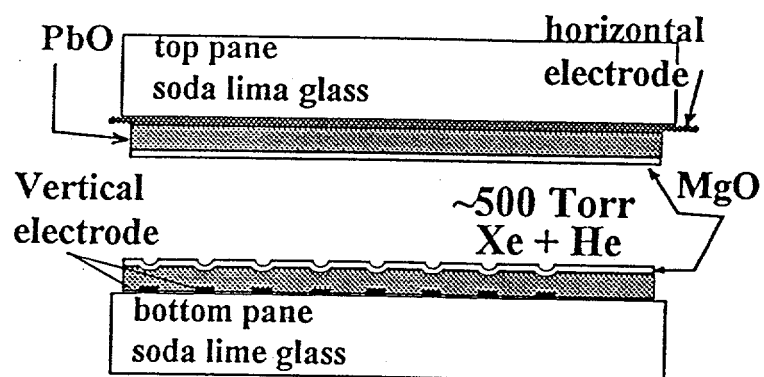


Figure 1

