

**MAX-PLANCK-INSTITUT FÜR PLASMAPHYSIK**  
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Surface Analysis of a Central Part  
of the JET Graphite Limiter

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## Abstract

A central part (tile 4) of the carbon limiter of JET from octant 4, which was used in the period from July 1983 to December 1983 was analysed with regard to its surface composition and topography by PIXE, RBS, SEM and EIXE.

For reference, a graphite sample from a limiter tile which had not been incorporated in JET and a section of a graphite piece cut from the limiter were also investigated.

The elements found most frequently on the rough ( $\sim 10 \mu\text{m}$ ) graphite surface were Mo ( $10^{20} - 10^{21}$  atoms/m<sup>2</sup>), Ni ( $10^{20} - 10^{21}$  atoms/m<sup>2</sup>), Cr ( $10^{19} - 10^{20}$  atoms/m<sup>2</sup>), Fe ( $10^{19} - 10^{20}$  atoms/m<sup>2</sup>), Ti ( $10^{18} - 10^{19}$  atoms/m<sup>2</sup>), Ca ( $10^{19}$  atoms/m<sup>2</sup>) and K ( $10^{19}$  atoms/m<sup>2</sup>).

Mo, Ni and Cr were also found on tile 1 of the same limiter analysed by Culham Laboratories /1/ by means of RBS and other methods.

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## I. Introduction

With the recent use of graphite limiters in all large tokamaks, the plasma performance and metal impurity concentration in the plasma have been considerably improved. This is partly due to very suitable properties of graphite such as its resistance to high heat loads and the absence of melting. Furthermore, graphite is a low-Z material, and so a much higher concentration of the limiter material can be tolerated in the plasma compared with that of metals. Experiments with collection probes in the plasma boundary have shown that during the plasma discharge considerable amounts of wall material eroded by the plasma are redeposited on surfaces in the torus. These processes also occur on limiter surfaces and may reduce the advantages of graphite limiters. To know the extent of these surface variations, it is very important to investigate the surface of the graphite limiters especially with regard to the deposition of metals.

## II. Experimental Methods

After shut-down of JET in December 1983 the graphite limiter in octant 4 was dismantled and tile 4 (Fig. 1) was sent to IPP for surface analysis. From the bottom edge of this tile 30 samples with a thickness of 1.5 cm were cut (Figs. 1, 2) and analysed by the following methods:

- 1) Proton Induced X-ray Emission (PIXE) analysis with 1.5 MeV protons. This method is sensitive within a depth in graphite of up to 5 to 10  $\mu\text{m}$  but has no depth discrimination. Quantitative results were obtained by calibration with standard targets and are mostly given in units of surface concentration ( $\text{atoms}/\text{m}^2$ ). Quantities of elements which are supposed to be distributed within the bulk of graphite are alternatively given in units of volumetric concentration ( $\text{atoms}/\text{carbon atom}$ ).
- 2) Rutherford Backscattering Spectroscopy (RBS) with 2.6 MeV  $^4\text{He}$  and protons and a backscattering geometry of  $165^\circ$ . This method allows measurement of the depth distributions of concentrations. The results are quantitative and are given in units of surface concentration ( $\text{atoms}/\text{m}^2$ ).
- 3) Scanning Electron Microscope (SEM) observations for investigating the limiter surface topography.
- 4) Electron Induced X-ray Emission (EIXE) analysis, which could be performed in situ with the SEM observations.

### III. Results

#### 1) Limiter tile surface

##### a) PIXE results:

Figure 3 a,b) gives two examples of PIXE spectra measured at position 60 mm a) and 11 mm b) on the limiter surface.

The elements which can be clearly detected are Cl, K, Ca, Ti, Cr, Fe, Ni and Mo. Possibly also present are O, N, Al, Si, P, S, Mn and Cu. But their  $K(\alpha)$  emission lines are either suppressed by the 50  $\mu$ m mylar foil in front of the X-ray detector (O, N, Al) or are covered by lines of other elements (Si, P, S, Mn, Cu). For example, the Mn ( $K_\alpha$ ) line is covered by Cr ( $K_\beta$ ), and the Cu( $K_\alpha$ ) line by Ni( $K_\beta$ ). But a comparison of the line intensities of Cr ( $K_\alpha$ ) with Cr ( $K_\beta$ ) and of Ni( $K_\alpha$ ) with Ni( $K_\beta$ ) at position 11 mm on the tile surface shows that Mn must be present with a concentration of about  $1 \times 10^{19}$  at/m<sup>2</sup> and Cu with about  $4 \times 10^{19}$  at/cm<sup>2</sup>. For Cu, the signal may be partially due to protons scattered from the copper cylinder which surrounds the target. At other positions Mn and Cu might be present with concentrations similar to that of Ti since they are also found in comparable amounts in Inconel 600. For K, Ca, Ti, Cr, Fe, Ni and Mo the distribution of concentrations across the limiter tile surface are given in Fig. 4 a,b). Cl could not be reliably evaluated owing to the presence of the large Mo( $L_\alpha$ ) peak.

Besides the statistical uncertainties (given with error bars in Fig. 4 a,b), the quantities for Ti, Fe, Ni and Mo have a systematic uncertainty of at most + 20 % for the highest concentrations measured owing to self-absorption and energy loss of protons in metallic layers thicker than those used on the standard targets for calibration. From the calibration itself we have an uncertainty of about  $\pm 10$  %.

Cr was not calibrated with a standard target but was evaluated relative to the yield of Fe, giving an uncertainty of  $\pm 20$  %.

K and Ca were also evaluated relative to the yield for Ti, but are only certain within a factor of two owing to the unknown detector efficiency at low X-ray energies ( $< 3$  keV).

The measured ratios of Ti/Ni, Cr/Ni and Fe/Ni are 0.07, 0.21 and 0.09 respectively. Within the uncertainties these ratios correspond to the ratios present in Inconel 600.

b) RBS results

Figure 5 a,b) gives two examples of He RBS spectra at position 60 mm a) and 11 mm b) on the tile surface. Besides the carbon backscattering edge, three elements can be clearly detected: O, Ni and Mo. Figure 5b) shows a small shift of the Mo peak relative to the surface, indicating the presence of a material layer on the Mo. The large tail of the metallic components prevents observation of the elements detected by PIXE and makes it difficult to separate Ni and Mo quantitatively from the background signals.

This makes it unreasonable to calculate depth profiles, which is additionally confirmed by analysis of the surface topography (see c).

A better separation from the background signal can be achieved by means of 2.6 MeV protons, Fig. 6 a,b). The evaluation of

the Ni and Mo quantities in Fig. 5a) gives, within the uncertainties, the same results as the PIXE measurements. For O the peak integral gives a concentration of  $5 \times 10^{20}$  O-atoms/m<sup>2</sup>. Here a cross-section enhancement factor of about 5.6 had to be taken into account.

c) SEM and EIXE results

SEM:

At position 11 mm, 110 mm, 230 mm, 350 mm and 457 mm on the tile surface SEM and EIXE analysis were performed (Fig. 7). At these positions the concentrations of the heavier elements have maxima and minima (see Fig. 4 a,b)). The viewgraphs show a surface covered with droplets at the tile edges, whereas on the rest droplets can only be identified with difficulty (position 110 mm) or cannot be observed at all (diameter smaller than 0.1  $\mu$ m).

Droplets are predominantly found in grooves of the graphite surface, whereas on ridges very few can be seen. The existence of droplets and the very rough surface indicate the difficulties of calculating depth profiles from RBS measurements. The occurrence of a tail of the Ni and the Mo peak in the He-RBS spectra is due to the surface roughness and the existence of droplets (diameters up to 2  $\mu$ m) and possibly of metal implanted or diffused into the graphite. Separation of these different contributions is not possible and thus limits the usefulness of depth profiles.



EIXE:

Three different analysis measurements were made:

- 1) EIXE analysis of a surface area with  $0.02 \text{ mm}^2$  by scanning the electron beam over this area .
- 2) EIXE analysis of a spot at a droplet.
- 3) EIXE analysis of a spot on the surface without droplets.

EIXE measurements lack absolute quantitative results.

Thus only relative quantities can be given.

Figure 8 shows two EIXE spectra made at position 457. Spectrum a) is the result of analysis 1) and spectrum b) that of analysis 2).

A comparison of the signal peak heights with the bremsstrahlung background shows a larger concentration of Mo in b) and similar concentrations of Ni. The ratio of the  $\text{Mo}(L_\alpha)$  peak to the  $\text{Ni}(K_\alpha)$  peak in b) is nearly a factor of 8 larger than in a), which again indicates the larger relative amount of Mo in the droplets. The same result was observed on other droplets and also at position 11 mm. In Fig. 9 a,b) the Mo distribution 9a) is shown on a surface given in Fig. 9b). At droplets, the density of Mo is larger than in between them. The ratio of  $\text{Mo}(L_\alpha)$  to  $\text{Ni}(K_\alpha)$  from analysis 3) is similar to that from analysis 1).

A comparison of the peak heights of  $\text{Cr}(K_\alpha)$ ,  $\text{Fe}(K_\alpha)$ ,  $\text{Ni}(K_\alpha)$  and  $\text{Mo}(L_\alpha)$  for the five positions, measured in analysis 1), is shown in Fig. 10. The shapes of the curves are quite similar to those measured by PIXE (Fig. 4) where the area measured with the ion beam was a factor of 100 larger than in the EIXE measurements.

2) Impurities in the limiter bulk material

PIXE analysis of the section of the graphite piece cut from the limiter tile mainly shows the presence of S, Cl, K, Ca, Cr, Fe and Ni with the following mean concentrations (averages of five measurements):

Element	Surface concentration (At/m <sup>2</sup> )	Bulk concentration (at/c-atom)
S	4.6 x 10 <sup>17</sup>	4.4 x 10 <sup>-7</sup>
Cl	8.1 x 10 <sup>17</sup>	8 x 10 <sup>-7</sup>
K	1 x 10 <sup>17</sup> ± 100 %	1 x 10 <sup>-7</sup>
Ca	1.4 x 10 <sup>17</sup>	1.3 x 10 <sup>-7</sup>
Cr	1.4 x 10 <sup>17</sup>	1.3 x 10 <sup>-7</sup>
Fe	1.3 x 10 <sup>18</sup>	1.3 x 10 <sup>-6</sup>
Ni	1.6 x 10 <sup>17</sup>	1.6 x 10 <sup>-7</sup>

The large mean deviation of ± 100 % is due to large variations of concentration at different locations.

3) Impurities on the surface of a piece of a limiter tile not incorporated in JET

The following impurities and concentrations were found:

Element	Surface concentration (at/m <sup>2</sup> )	Bulk concentration (at/c-atom)
K	1.3 x 10 <sup>17</sup> ± 100 %	1.3 x 10 <sup>-7</sup>
Ca	5.6 x 10 <sup>19</sup> ± 30 %	5.6 x 10 <sup>-5</sup>
V	4.8 x 10 <sup>18</sup> ± 100 %	
Cr	1.4 x 10 <sup>17</sup> ± 100 %	
Fe	1.2 x 10 <sup>18</sup> ± 50 %	
Ni	1.4 x 10 <sup>18</sup> ± 25 %	
Mo	1.0 x 10 <sup>20</sup> ± 4 %	

The results show a larger concentration of Ca compared with 2) and additionally V and Mo. The Mo concentration is lower than found on the limiter tile surface (see 1).

#### IV. Discussion and Conclusion

The presence of metals and other elements on the graphite limiter may be due to three processes:

- 1) Deposition by the plasma
- 2) Segregation from the graphite bulk to the surface
- 3) Deposition during machining, handling and bakeout.

An example of 3) is Mo, which is regarded as being deposited on the limiter during bakeout in the vacuum furnace /1/. However, Mo is a constituent of Inconel 625, out of which the bellows is made. The latter, however, is covered with protection plates of Inconel 600 containing no Mo.

For Ti, Cr, Fe, and Ni the concentration ratios relative to Ni are similar to the ratios in Inconel 600. This indicates that these metals are transferred from the wall to the limiter. Segregation alone, from the bulk to the surface, would give lower concentrations and different ratios.

The similarity in shape of the distributions along the limiter surface of Ti, Cr, Fe and Ni, originating from the torus wall, to that of Mo, probably initially present on the limiter, indicates that the distribution is a result of deposition, erosion and, presumably, redeposition.

For K and Ca, frequently present in graphite, it is assumed that the quantities detected are present owing to segregation processes out of the bulk. This is indicated by the shape of the profiles, which are rather different from those of the heavier elements and which may be a result of segregation and erosion processes on the surface.

For understanding the profiles of the metals (except K and Ca) deposition and erosion have to be regarded. Probe measurements have shown that in most tokamaks metal deposition increases with decreasing distance relative to the plasma. This may explain the small increase of deposits at the very edges of the limiter tile (Fig. a,b), but not the subsequent strong minima. The two characteristic minima correspond very closely to the positions of radiation maxima, i.e. temperature maxima, observed with infrared cameras on limiter surfaces during plasma discharges /2, 3/. The slight asymmetric shape of the concentration profiles at the ion and electron drift sides is also observed in a slightly lower peak surface temperature at the electron side compared with the ion side.

The concentration minima can be explained if we assume that erosion processes are stronger at depositions with higher energy deposition.

This erosion can be due to sputtering by impinging ions and atoms, to evaporation by heat loads or to arcing processes.

The relative maximum at the centre of the tile surface might be due to deposition of gyrating particles with guiding centres parallel to the surface and thus lower energy deposition and lower erosion.

At the edges of the limiter an important result is the observation of droplets (Fig.7), but it cannot be excluded that droplets also exist at positions near the centre. Formation of droplets is attributed to heating of metal films on graphite surfaces. It was shown /4/ that between 830 K and 1090 K Ni films on graphite surfaces change into a droplet structure. The presence of Ti, Cr, Fe, Ni and Mo on droplets may indicate the presence of effective mixture processes of elements on the limiter surface.

The elements found in the limiter bulk are typical of impurities in graphite /4, 5/. The sum of all bulk concentrations is in the range of ppm, which indicates the presence of a rather pure graphite.

A somewhat surprising result of the analysis of the limiter part, not exposed in JET, was the detection of V. The origin of this impurity is not known. In contrast to the other elements found on the surface Mo is rather homogeneously distributed on this sample surface which is indicated by the small relative mean deviation of 4 %. This supports the assumption that vapour deposition of molybdenum oxide occurs in the vacuum furnace during bakeout.

Acknowledgements

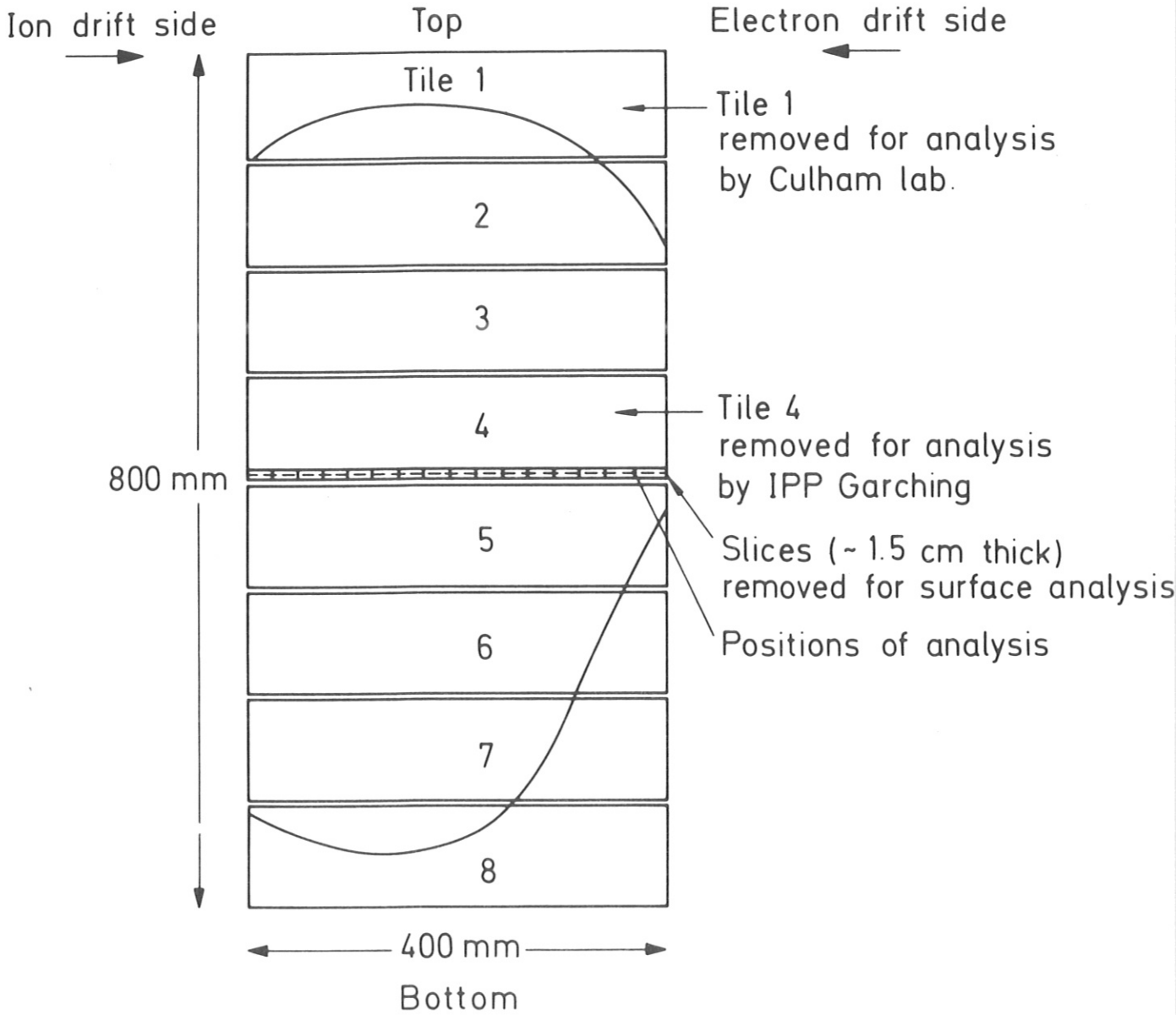
We should like to thank H. Kukral for performing the SEM and EIXE measurements and Dr. A.P. Martinelli for preparing the tile samples and assisting with the measurements.

References:

- /1/ G.M. McCracken, TAN (84) 2-2
- /2/ L. de Kock, private communication,  
measurements performed by M. Pick and D. Summers
- /3/ L. de Kock, private communication
- /4/ J. Ehrenberg and P. Børgesen, to be published
- /5/ P. Børgesen, L. Svendsen and J. Ehrenberg,  
to be published

Figure Captions

- Fig. 1 The graphite limiter of JET, octant 4, seen from the plasma.
- Fig. 2 Cross-section of the limiter tile.
- Fig. 3 PIXE spectra at position 60 mm a), and 11 mm b) on limiter tile surface.
- Fig. 4 a) Distribution of impurities across the limiter tile surface,  
b) same as a) for Ni and Mo, linear scale.
- Fig. 5 RBS spectra of 2.6 MeV  $^4\text{He}$  ions at position 60 mm a) and 11 mm b).
- Fig. 6 RBS spectra of 2.6 MeV H ions at position 60 mm a) and 11 mm b).
- Fig. 7 SEM micrographs of the tile surface at five different positions.
- Fig. 8 EIXE spectra of a surface with an area of  $0.01 \text{ mm}^2$  a) and of a droplet b).
- Fig. 9 Element distribution of Mo a) on a surface region with droplets b).
- Fig. 10 Distribution of signal peak heights of  $\text{Cr}(K_\alpha)$ ,  $\text{Fe}(K_\alpha)$ ,  $\text{Ni}(K_\alpha)$  and  $\text{Mo}(L_\alpha)$  at five different positions measured by EIXE on a surface with an area of  $0.01 \text{ mm}^2$ .  
Peak heights of different elements cannot be quantitatively compared with each other.



Jet carbon limiter octant 4  
View from the plasma

Fig.1)



JET CARBON LIMITER  
OCTANT 4 TILE 4 RECEIVED  
EDGE VIEW, SECTIONS FOR SURFACE ANALYSIS

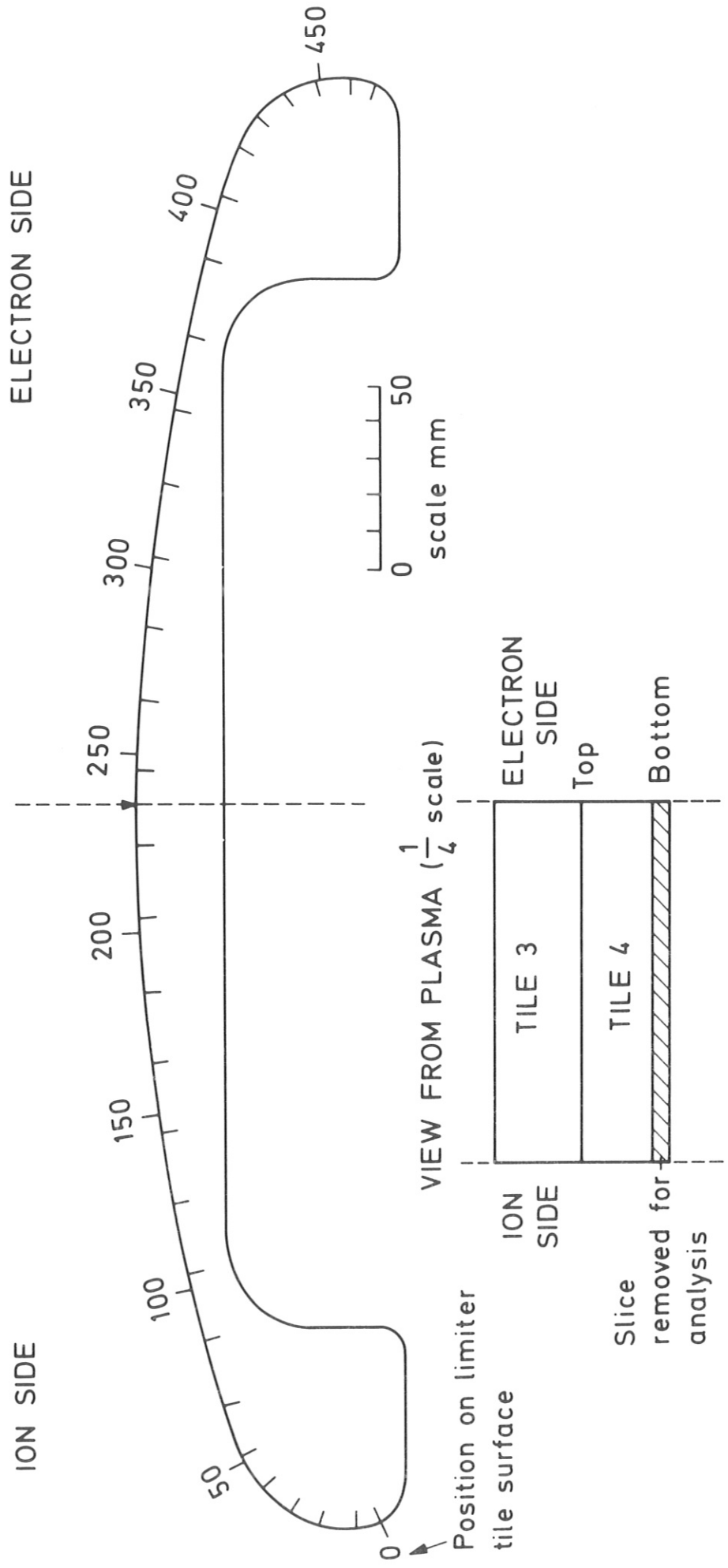


Fig.2)

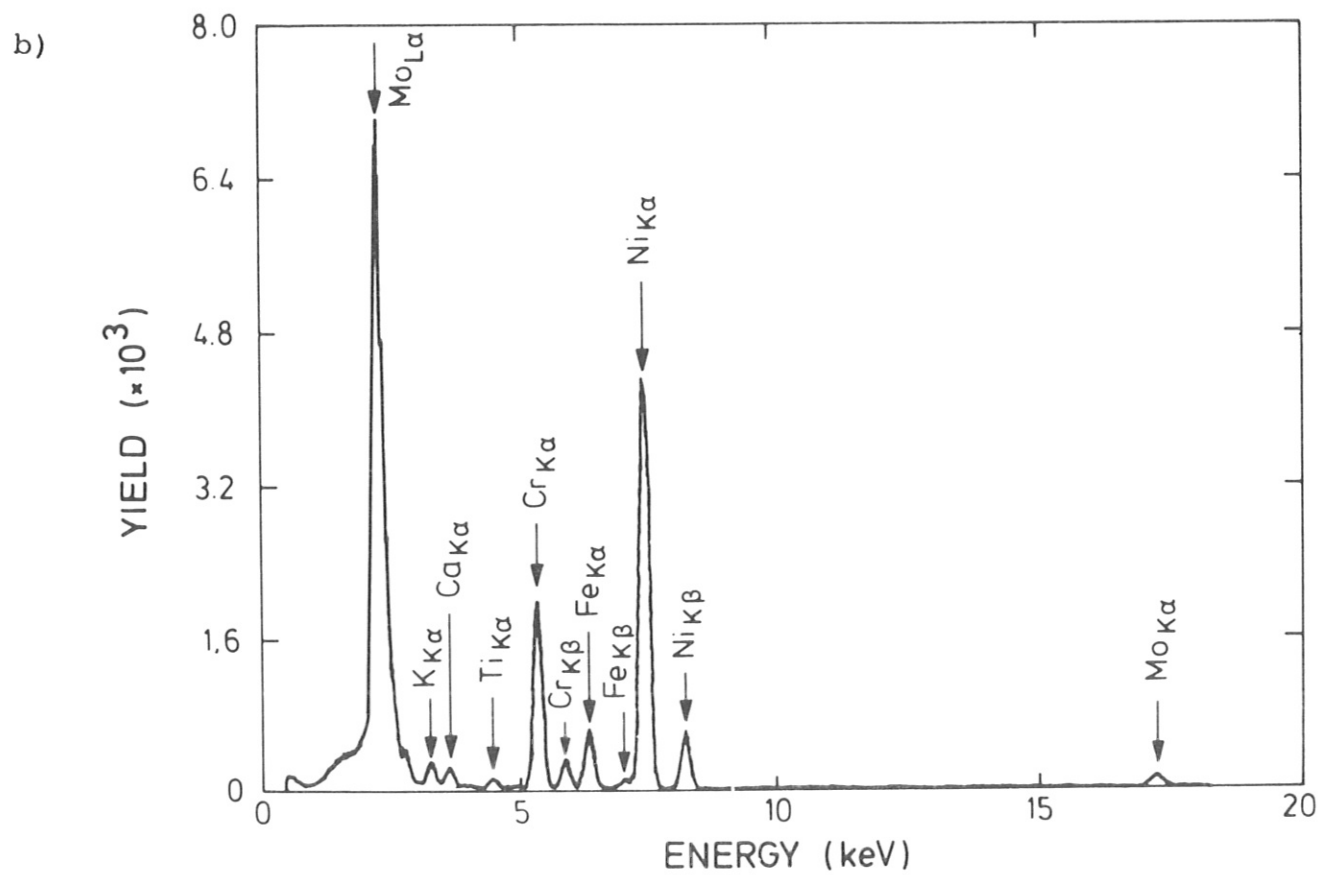
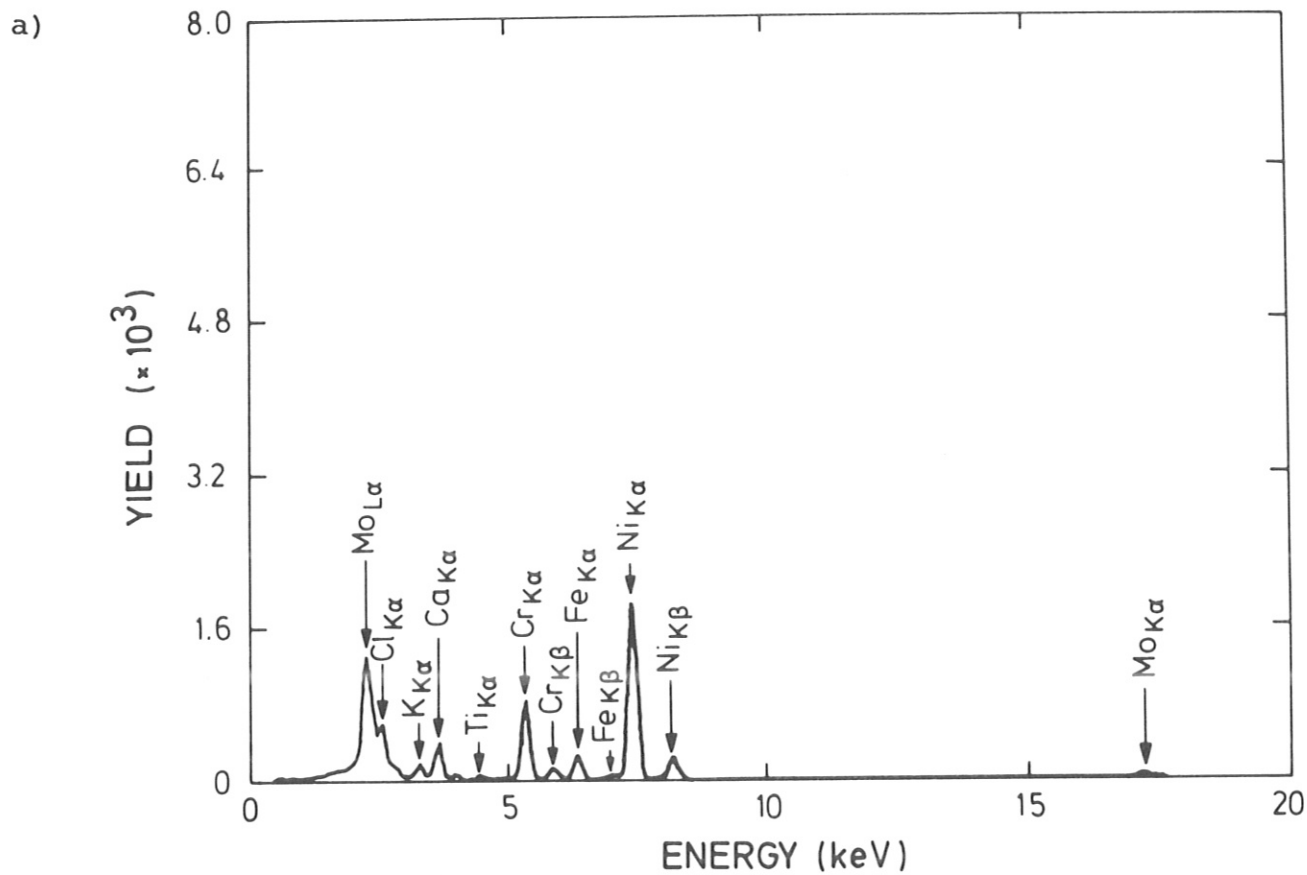


Fig.3)

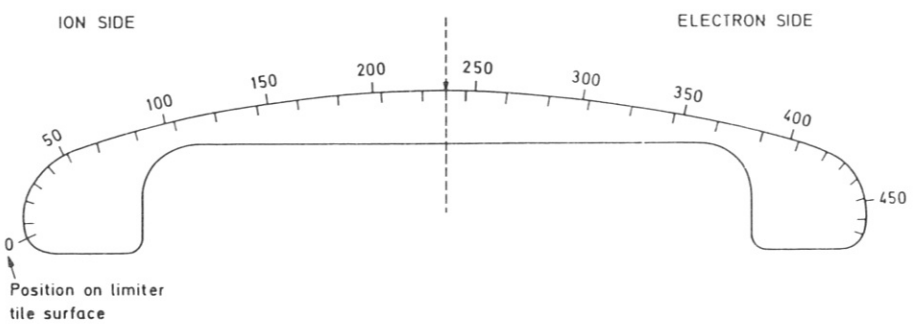
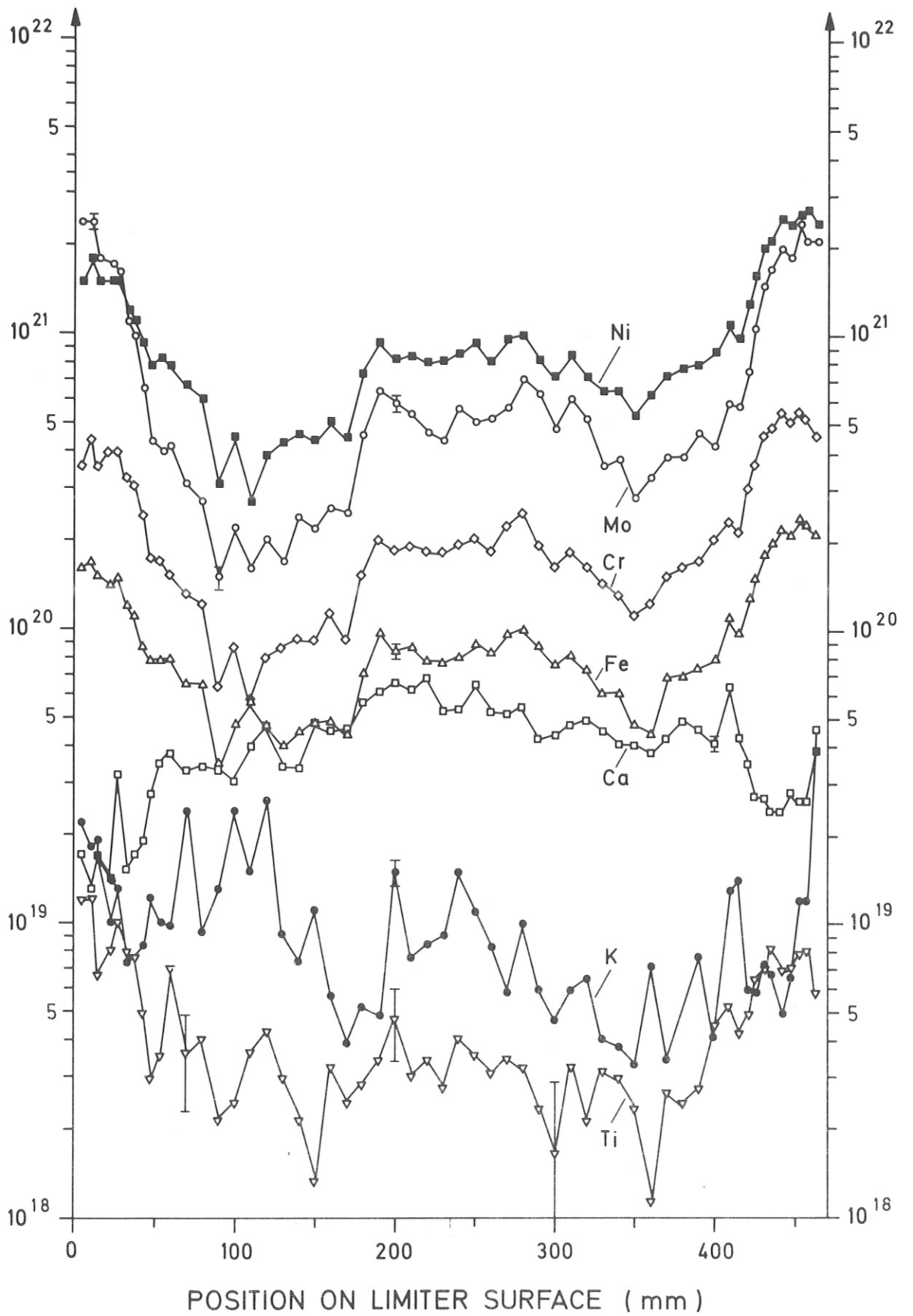


Fig.4a)

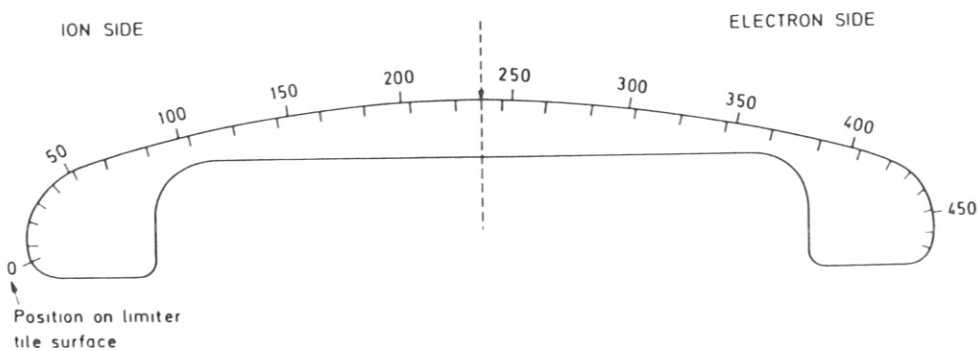
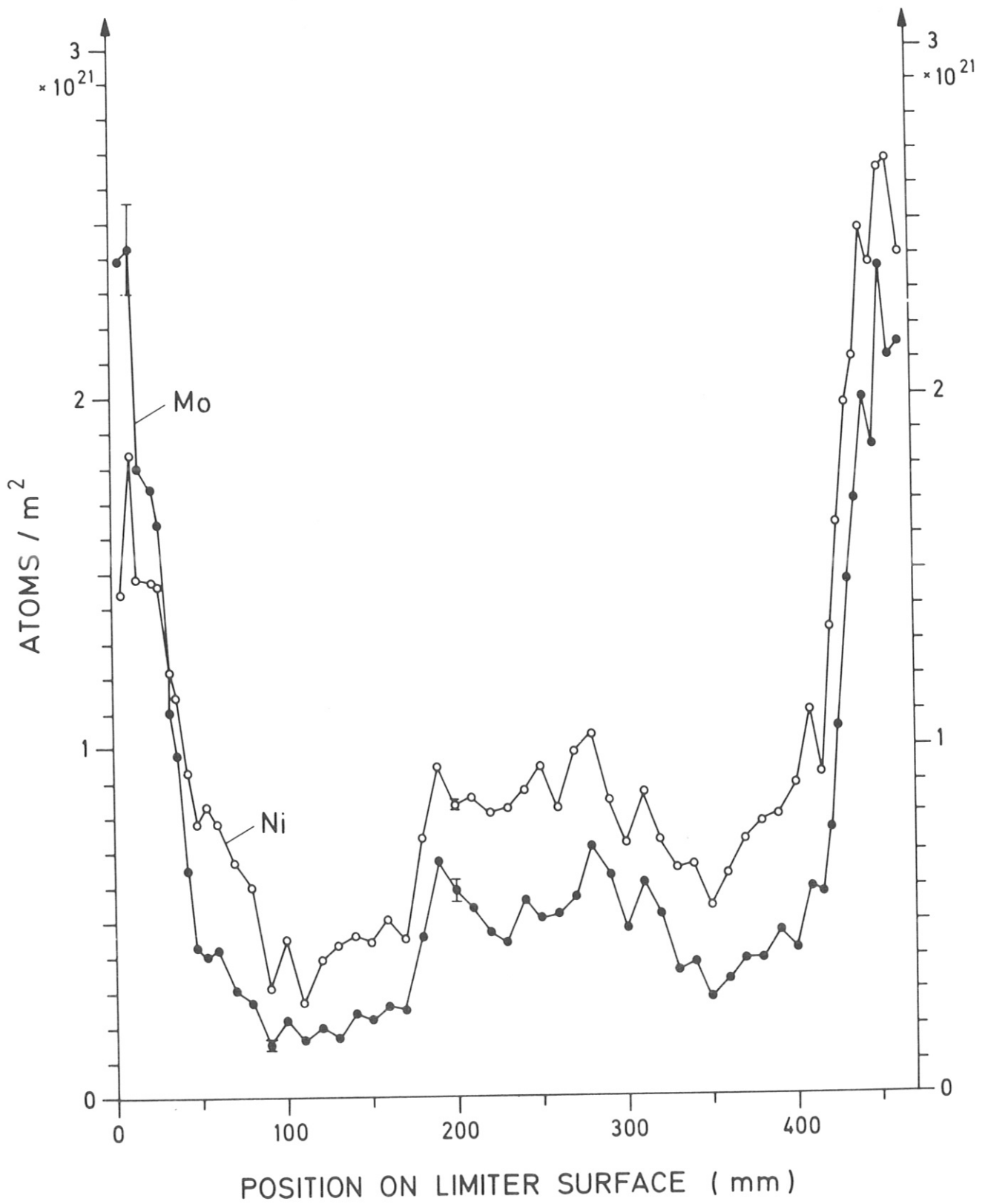


Fig.4b)

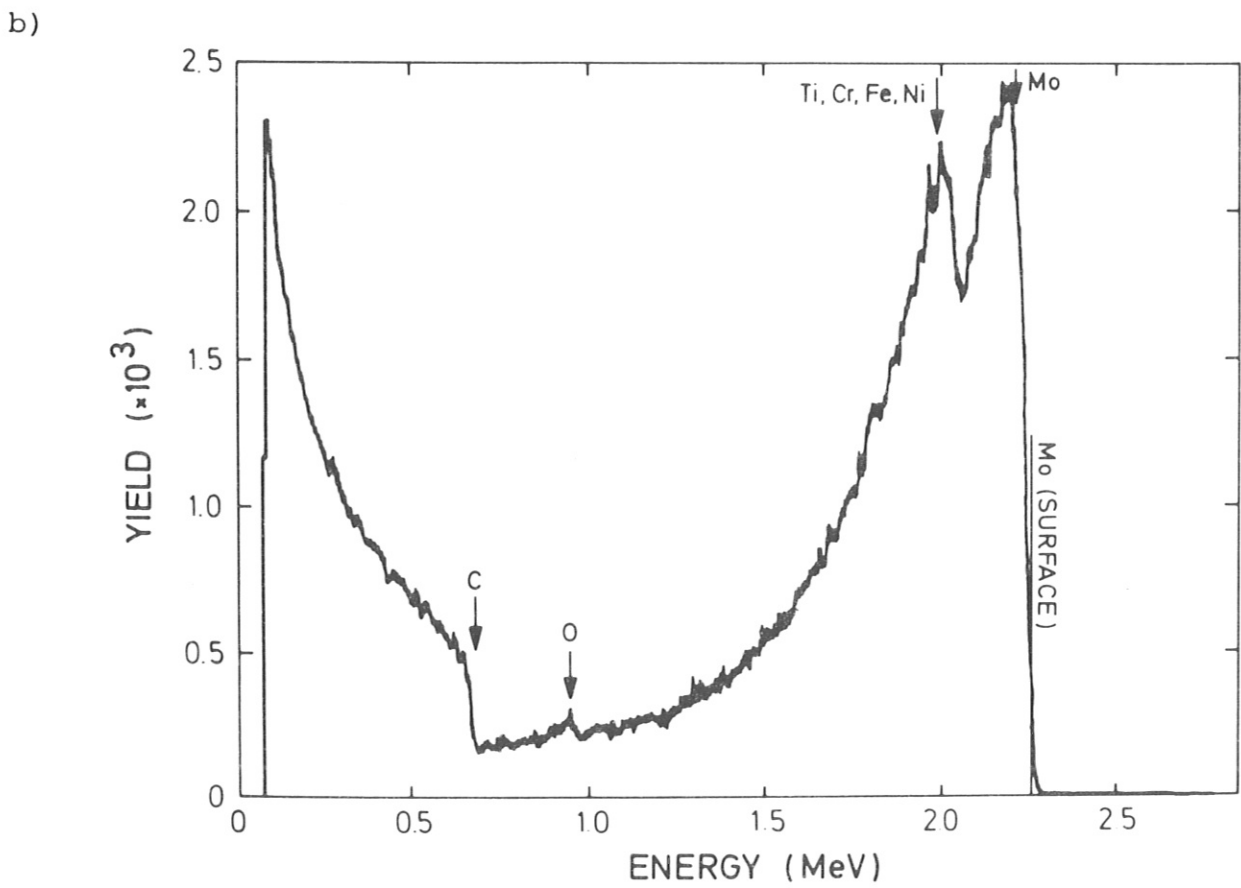
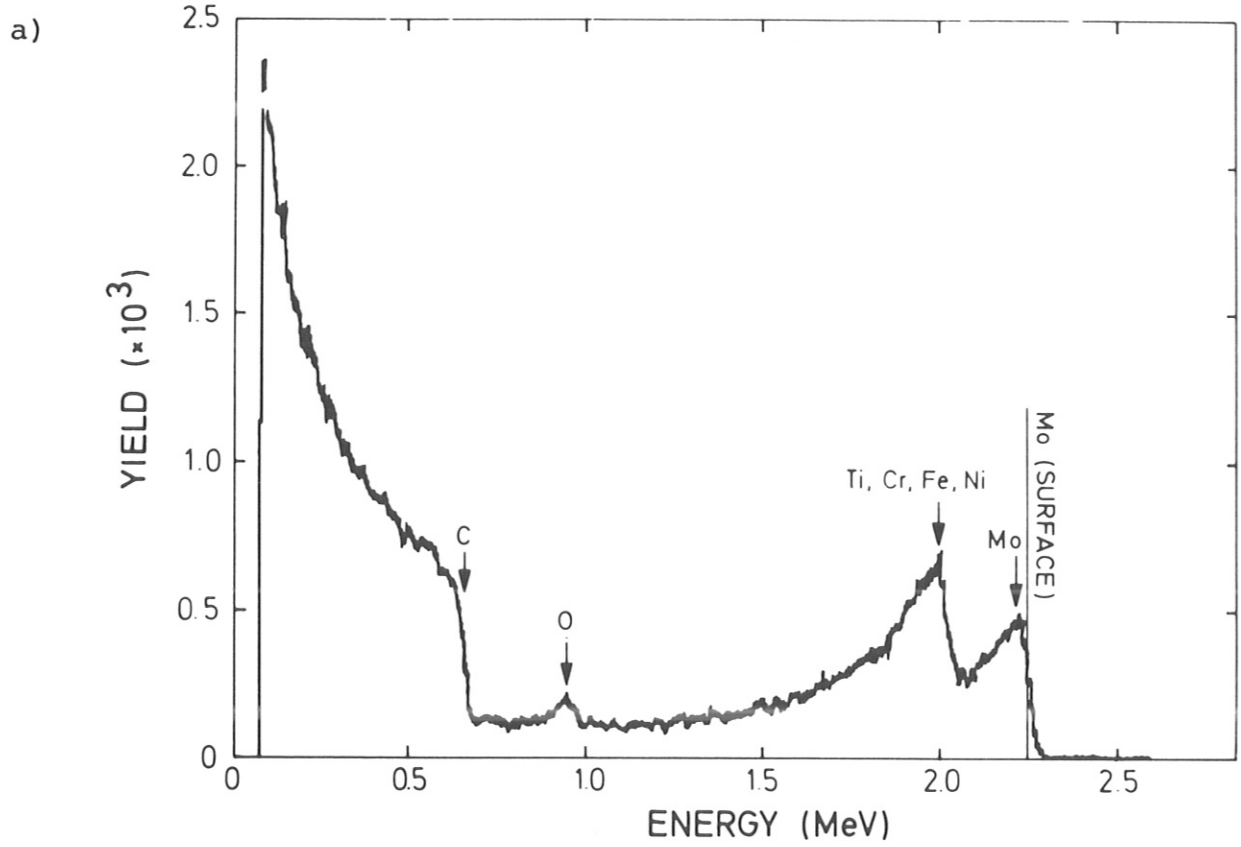


Fig.5)

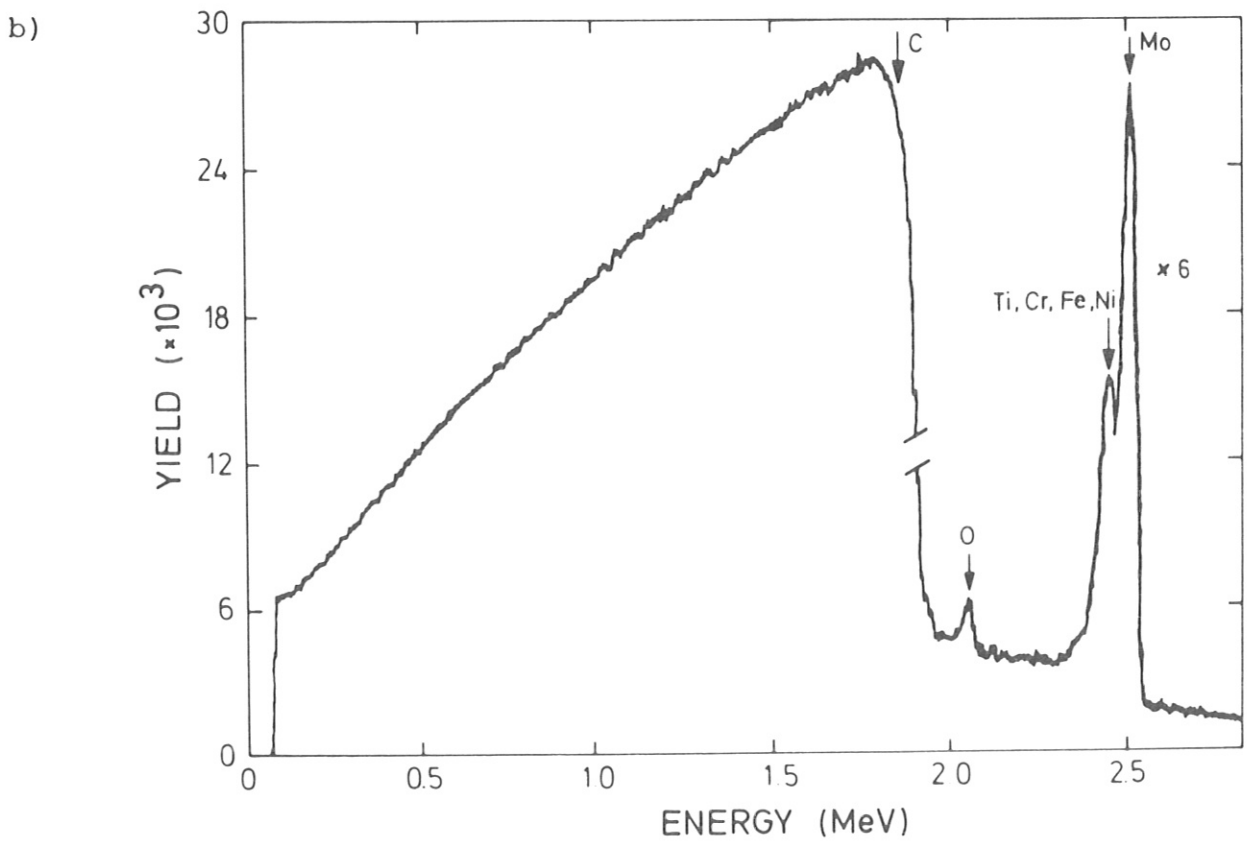
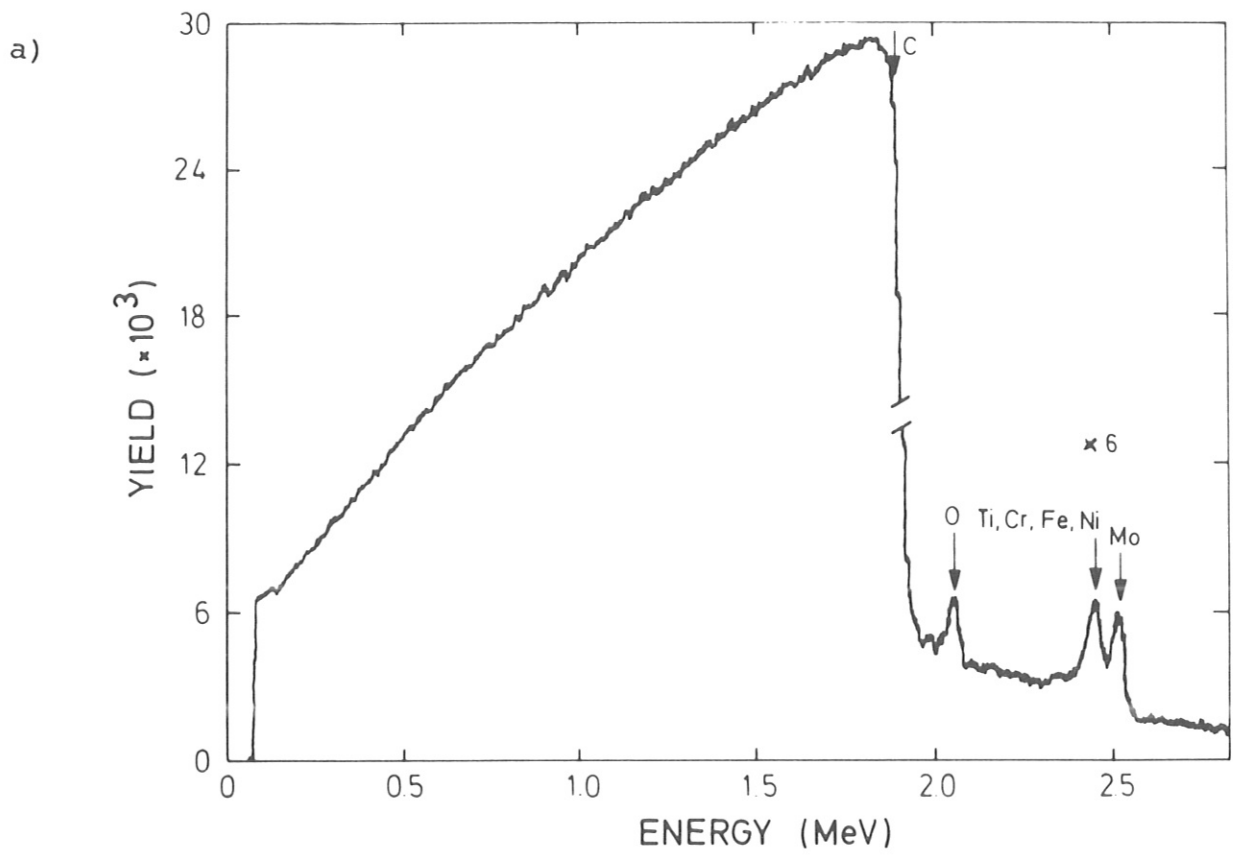
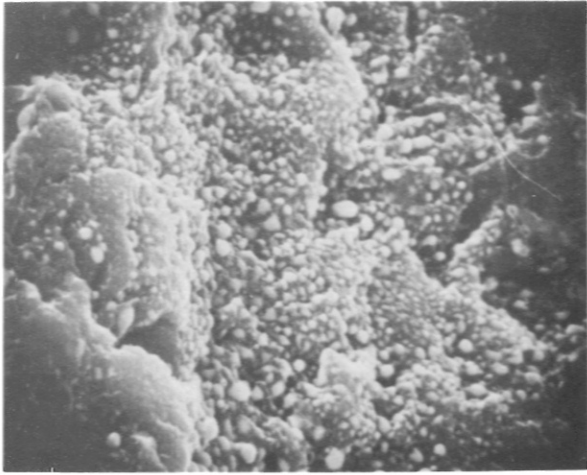


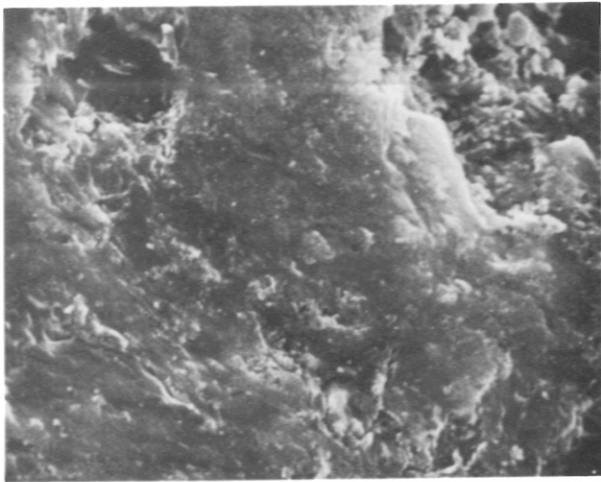
Fig.6)



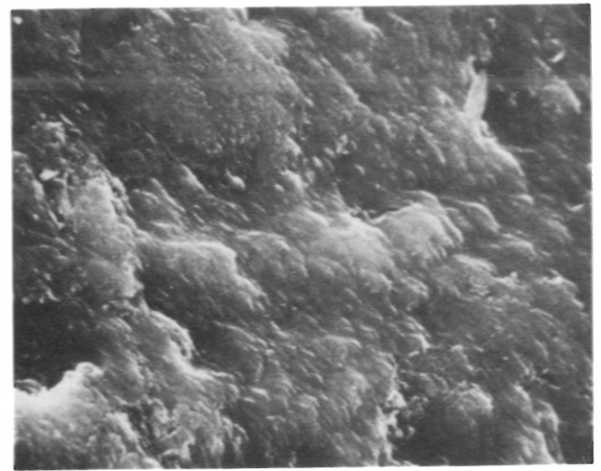
11 mm



110 mm



230 mm



350 mm

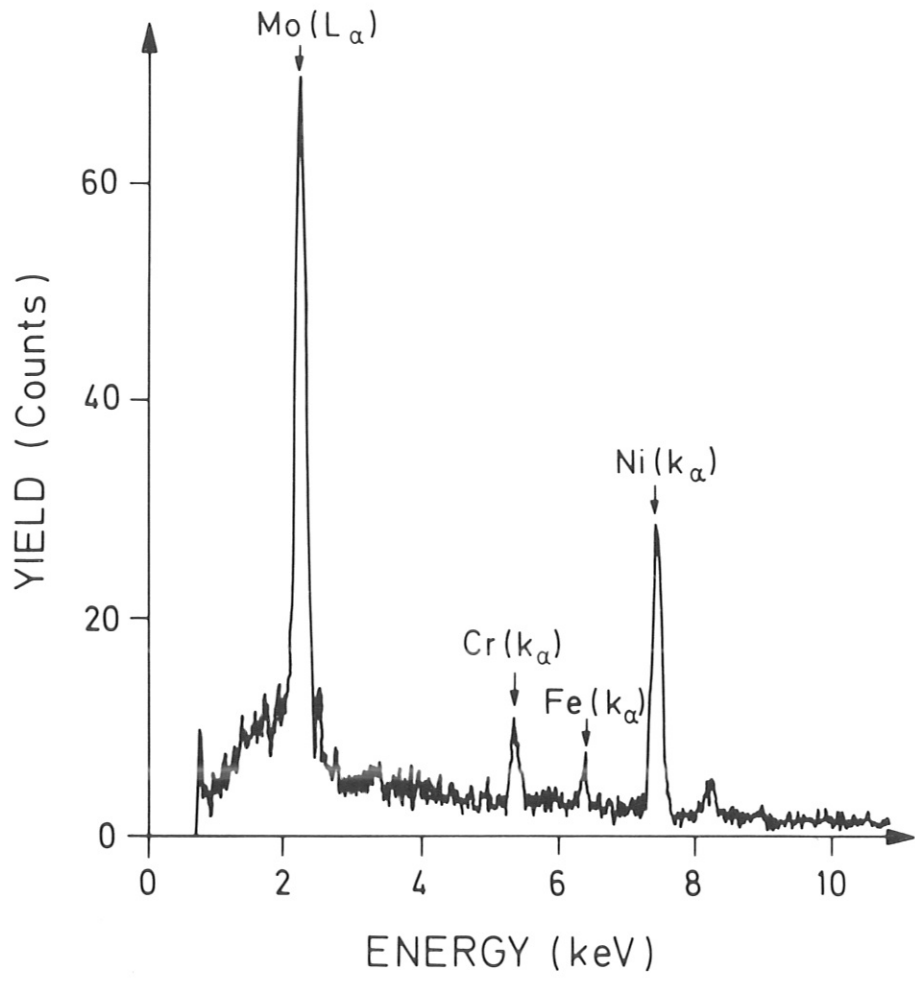


457 mm

20  $\mu$ m

Fig.7)

a)



b)

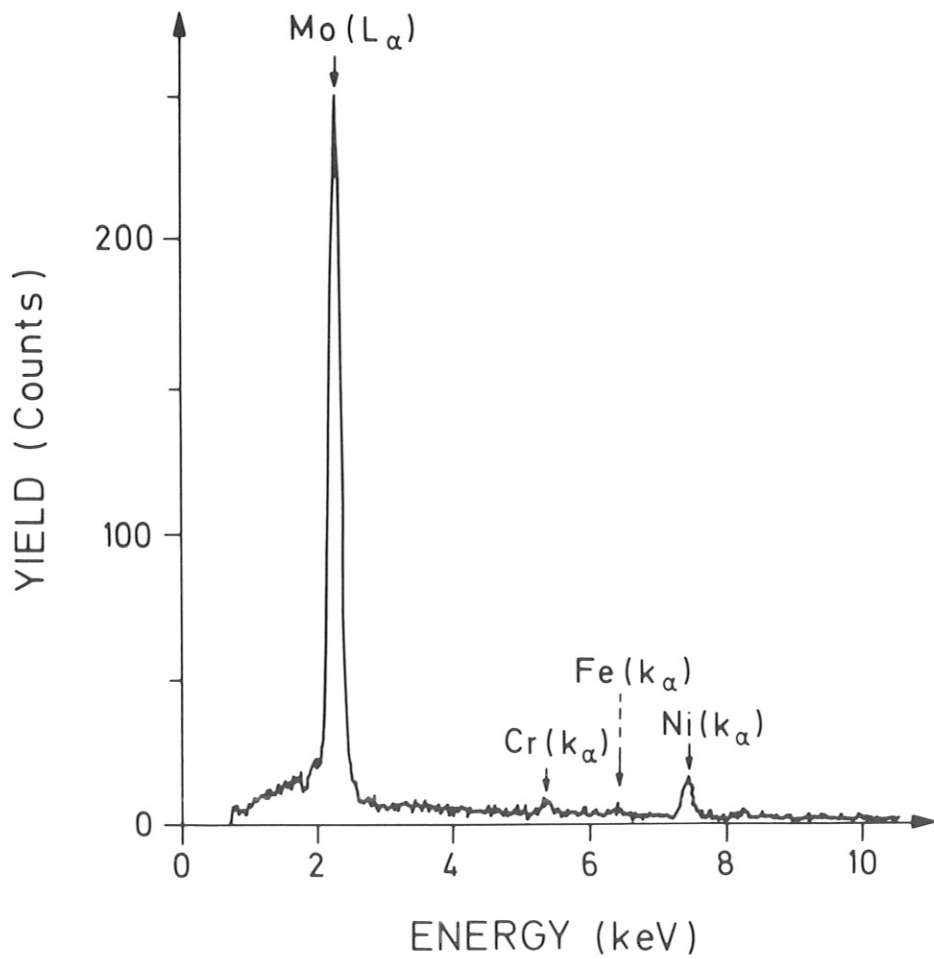
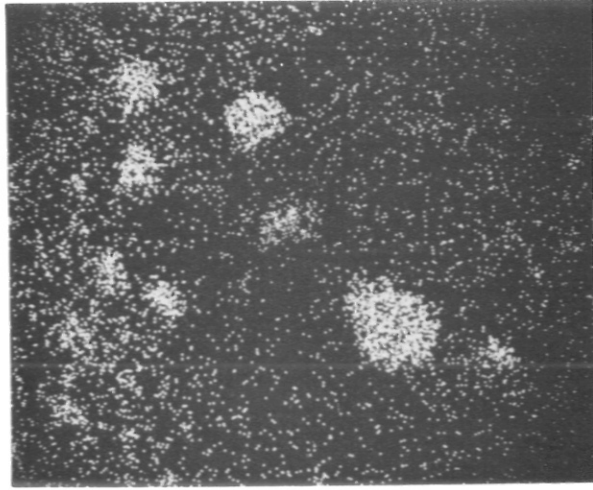
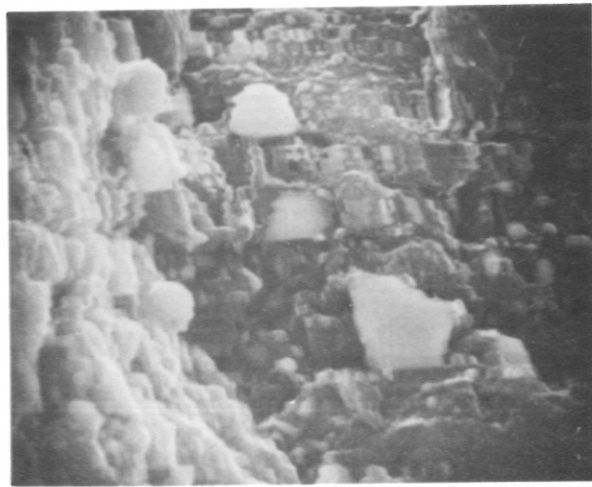


Fig.8)





a)



b)

10  $\mu\text{m}$

Fig.9)

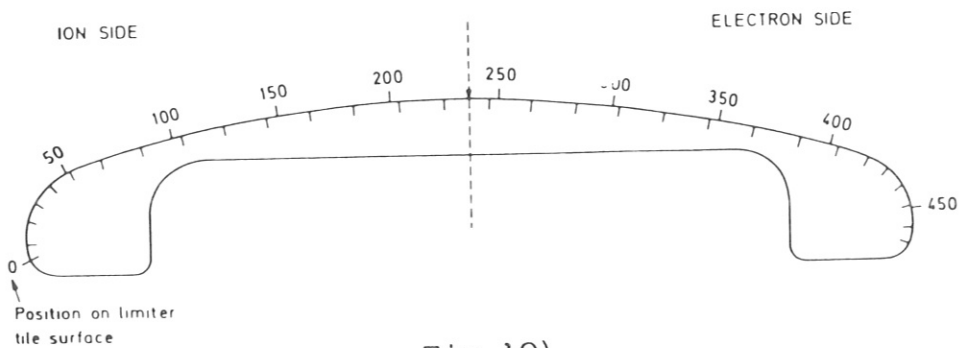
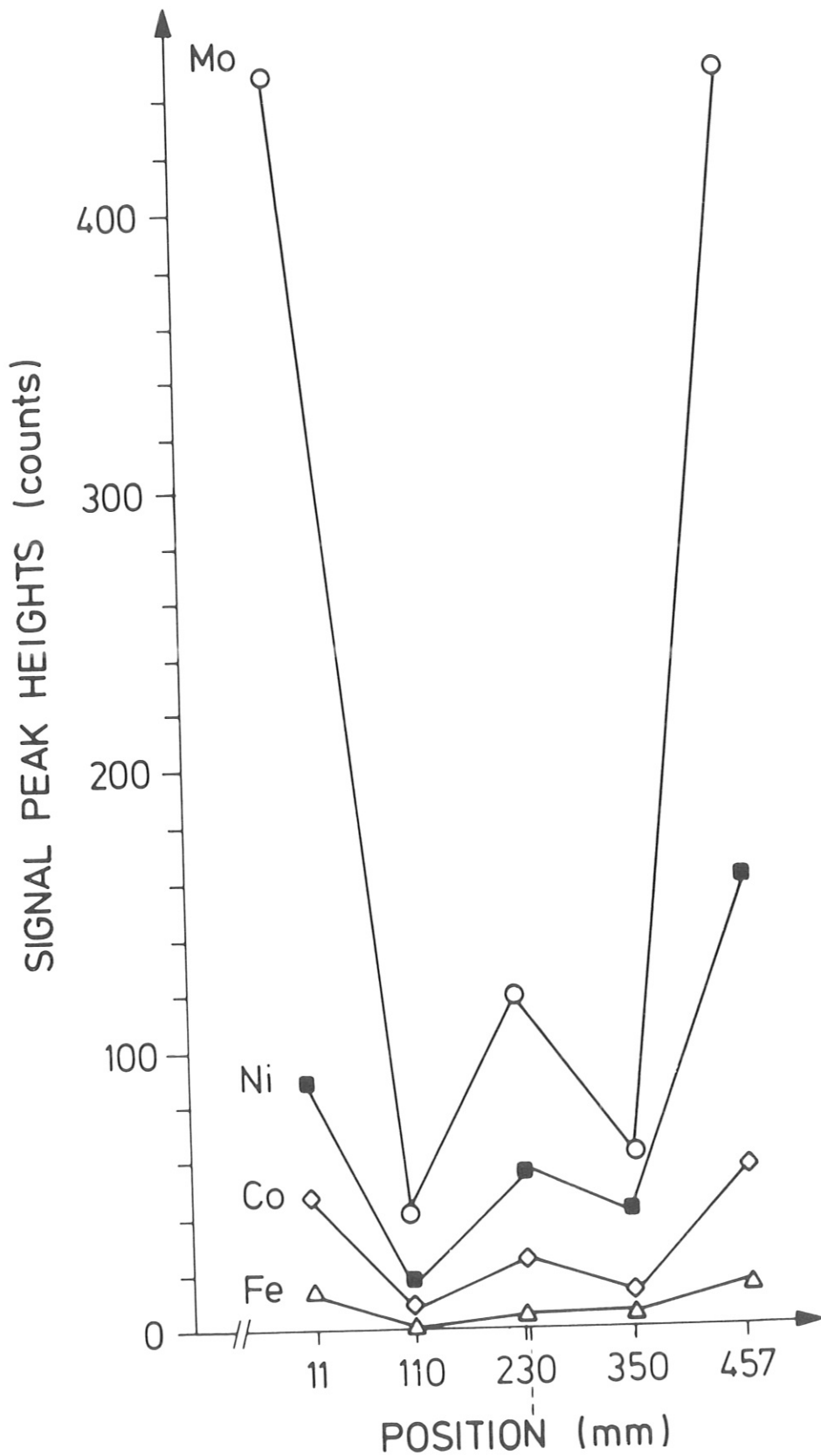


Fig.10)