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(3.2) *UP TO DATE SURFACE ANALYSIS TECHNIQUE FOR COMPOSITION DETERMINATION OF SURFACES : QUADRUPOLE IONIC MICROPROBE*

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An original device for surface analysis and profile studies has been realized by the combination of a duoplasmatron type ion gun and a high performance quadrupole analyser. After description of the instrument, some results are presented : traces detection, ionic images, profile studies.

The increasing needs in qualitative and quantitative analysis of solid surfaces, have resulted in the development of two kinds of equipments based on the principle of secondary ionic emission :

- *the first kind uses the conventionnal magnetic filtering and high density primary beams. They are generally known as ion microscopes. These apparatuses have a very high sensitivity to elements at traces concentration, increasing of 2 or 3 decades the detection limits of other analysis technics.*

  More over, recent developments in semi-quantitative analysis have allowed to achieve concentration measurements with a precision in the range of $\pm$ 20 %.

- *the second kind are used for more fundamental studies called static SIMS, involving very low primary ion density. This kind of experiment is mainly directed towards characterisation of superficial adsorption and desorption ; these phenomena being very fast, magnetic mass spectrometers are not suitable.*

QUADRUPOLE FILTER Q 156

The use of a quadrupole analyser in SIMS technique requires very high performances for a given mass range in :

- *sensitivity*
- *signal/noise ratio*
- *resolution*
- *mass descrimination*

The performances of a quadrupole analyser are mainly determined by :

- *The input and output optics of the filter*
- *The geometrical dimensions of the analyser*
- *The high frequency applied to the rods*

In the Q 156, the ions formed at the surface of the sample pass through a 45° selector which suppress from the analysed beam all parasit particles (neutral, photons, high energy ions), this increases signal/noise ratio.

The quadrupole analyser itself is a high precision assembly of 4 molybdenum rods, 15.6 mm diameter each.

The size of the rods is the same for any mass range (300, 600 or 1000 according to the analysis requests).

The very powerful supply permits to work at 2, 1.5 or 1.2 MHZ on the 3 ranges.

At the output of the filter, an off axis 21 dynodes electron multiplier amplifies the signal on an electrometer, a digital counting line and recorder or on CRT display in order to realize ionic images.

In the case of profile analysis or fast phenomenum, a peak selector multiplexer permits to follow only some elements.

These specifications permit to keep excellent results on SIMS analysis on the 4 main characteristics :

- *Sensitivity : detection of 10^{15} Boron atom/cm3*
- *Signal/noise ratio : 10^{8} for a great number of elements*
- *Resolution : $R > 18$ M*
- *Mass discrimination: low reduction of signal at high masses.*

For this reason, new equipments, faster and also less expensive and less bulky have been developed, they are based on quadrupole analysers.

Nevertheless, in order to keep the intrinsic high sensitivity of the SIMS technique, the quadrupoles used in this aim must have very high performances in transmission, resolution, signal/noise ratio and mass discrimination.

The NEW RIBER MIQ 156, QUADRUPOLE IONIC MICROPROBE, has been developed in order to realize the best compromise between these two main applications, by combining the possibilities of ionic microscopy and quadrupole filtering.

This quadrupole ionic microprobe is composed of :

- *Ion gun*
- *Quadrupole filter*
- *Vacuum system and accessories.*

ION GUN MODEL CID 15

The ion gun used in this equipment is duoplasmatron type, producing negative or positive ions at an energy reaching 15 KeV.

A hot cathode and a cold cathode permit to obtain ions from noble gases (Argon, Xenon, Krypton) necessary in some particular applications.

The ion beam is focussed and alined on the input slit of a 90° magnetic sector. This sector is foreseen to suppress any impurity from primary beam, as neutral particles emitted by the gun, as well as desorption ions.

The beam is then refocussed and its density and diameter are independently adjustable.

The minimum diameter obtained is 2 microns.
Defection plates permit to scan the ion beam in a 4 mm square.

The visualisation is achieved by amplification of the current absorbed by the sample or by detection of the secondary electrons emitted under bombardment.

The dimension of the scanned area is adjustable by modification of scanning voltage. Without scanning, the maximum density is 20 mA/cm2 which permits to realize the concentration profile in a very short time.

VACUUM SYSTEM AND ACCESSORIES

The vacuum system is completely bakeable, as realized in ultra-high vacuum technology.

The combination of ionic and titanium sublimation pumping permits to obtain an ultimate vacuum of 5×10^{-11} Torr.

During the bombardment, a differential pumping of the ion gun permits to keep the pressure in the range of 10^{-9} Torr. The sample is mounted on a high precision manipulator. After analysis, it can be taken in a sample loading lock and a new sample can be introduced.

The operation takes 5 mn maximum. The main advantage of such a system in comparison of carrousel is that the analysis chamber is never put to air, and the sample can be heated, cooled, polarized without difficulty.

EXAMPLES OF APPLICATIONS

- Lateral resolution (picture 1)

  The image is made in absorded current mode.
  The tracks of this integrated circuit are of 10 microns.
  The image has been realized with a 15 KeV argon bombardment.

- Ionic image (picture 2 and 3)

  The mass spectrum obtained by bombarding an integrated circuit presents :

  Si^{+} (28) Si^{++} (14) Si^{+++} (9 1/3) SiO^{+} (44)

  Al^{+} (27) Al^{++} (13,5) K^{+} (39) Na^{+} (23)

  After the realization of an absorded current image, the analyser adjusted on these masses, permitted to realize mapping of these elements.

  Thus, carbon is only located on the bounding disc, as well as sodium and potassium which give also some traces on bounding wire.

It is interesting to note that the image of peak 27 (Al^{+}) is different of the one of peak 13.5 (Al^{++}).

This shows that an impurity type $C2H3^{+}$ participates to mass 27.

At least, we can constat oxyde Silicon (SiO^{+}) in the vicinity of the tracks.

This presence perturbs the emission of Si^{+} (28) creating intensity variations in the image, which are not detected in Si^{++} and Si^{+++}

The realisation of these images took only a few seconds.

- *Concentration profiles*

An example of concentration profile of Boron in Silicon, shows the excellent sensitivity of Q 156, the rapidity of the analysis and the good resolution in depth obtained by the combination of scanning of primary beam and detection by electronic window associated to scanning.

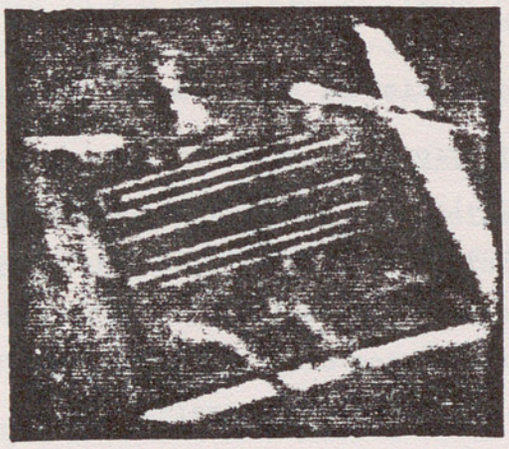
Such a system is very versatile as the user can choose the size of bombarded area and the analysed area.

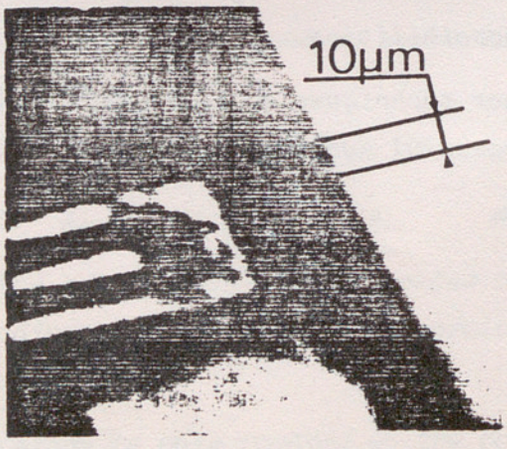
These few examples show that the use of a quadrupole analyser and a scanning ion gun, bring a lot of answers to surface or in depth analysis problems.

Such an equipment binds the two systems presently used and increases the utilisation possibilities.

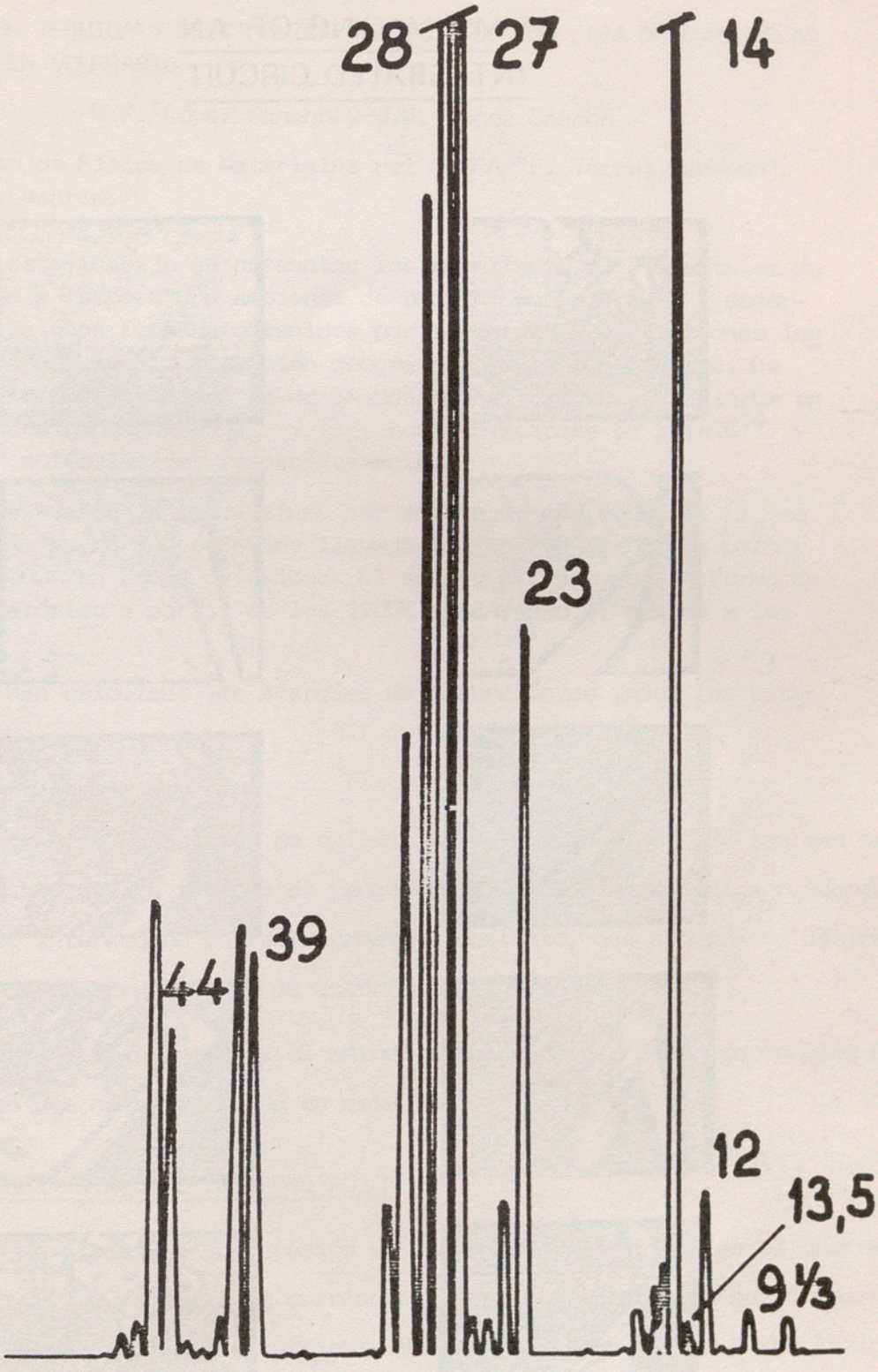
Associated to other techniques as AUGER Microscopy, the MIQ 156 is now one of the most powerful system for solid surface characterization.

INTEGRATED CIRCUIT VISUALISATION



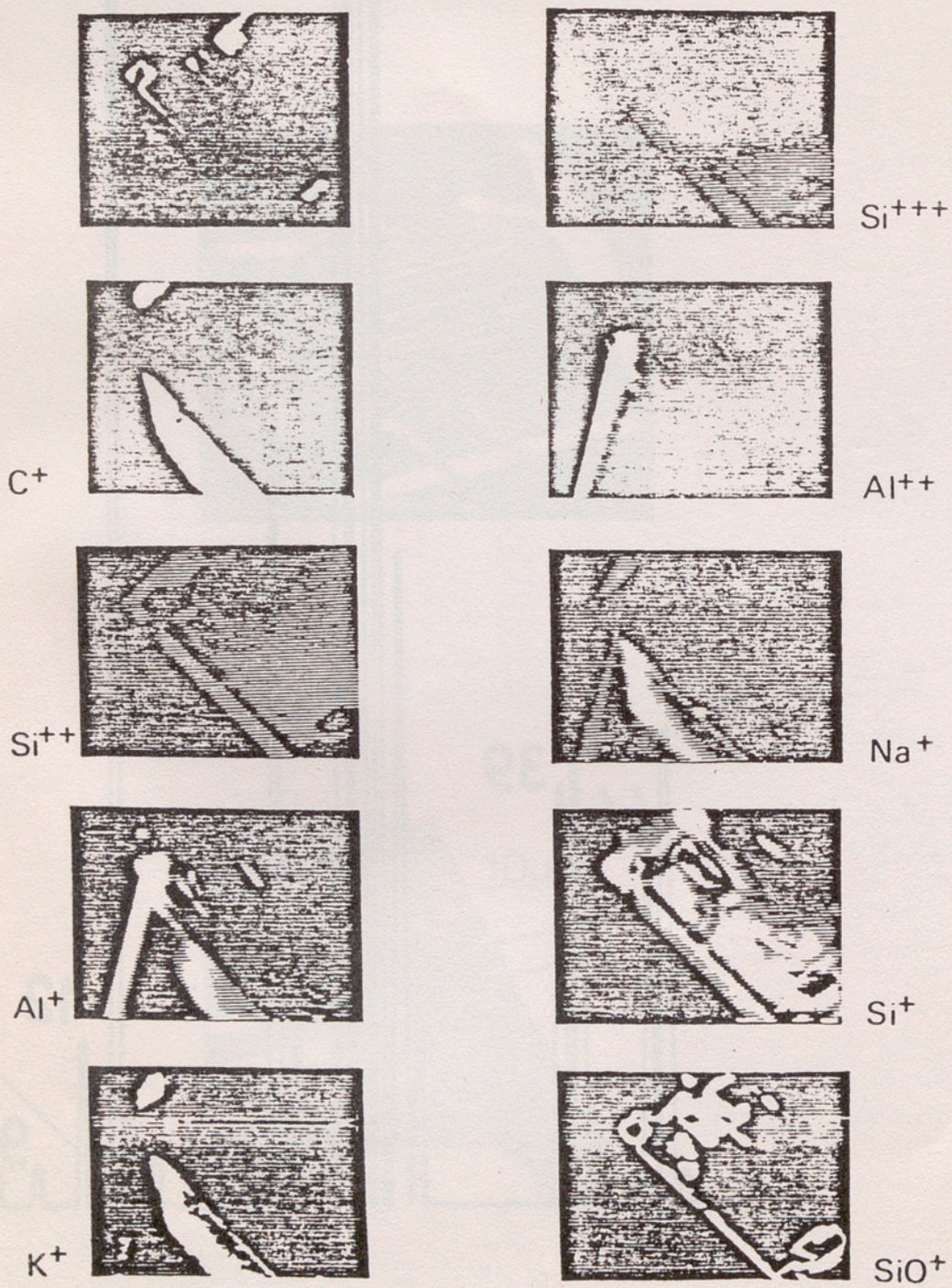


PICTURE 1



PICTURE 2

SIMS IMAGING OF AN INTEGRATED CIRCUIT



PICTURE 3