



Secondary ions are not monoenergetic and their energy distribution depends upon the energy of the primary beam, the kind of specimen and other factors. The energy broadening of secondary ions makes it necessary to use energy analysis before mass analysis.

Several secondary ion mass spectrometers have been described up till now [6-10]. Quadrupole mass filters were usually used as mass analysers.

In this paper the secondary ion mass analysing arrangement based on the MI-1201 mass spectrometer is described. The apparatus is equipped with a simple electrostatic energy analyser with a capillary type ion gun.

#### ADAPTATION OF THE MI-1201 MASS SPECTROMETER TO SECONDARY ION MASS ANALYSIS

The MI-1201 mass spectrometer, made in USSR, has a medium resolving power, from 600 to 1200, depending on the size of ion source and collector slits. The deviation angle of the ion beam in the magnetic field is  $90^\circ$  and the mean radius of ion trajectory within the magnetic lens is 21 cm. Sufficient pressure in the mass analyser chamber, which is  $2,6 \cdot 10^{-5}$  Pa ( $2 \cdot 10^{-7}$  Tr) for the mercury pumps and  $2,6 \cdot 10^{-6}$  Pa ( $2 \cdot 10^{-8}$  Tr) for the oil pumps, is provided by a system of two diffusion pumps.

Fig. 1 shows the scheme of the deflection chamber and the vacuum system of the MI-1201 mass spectrometer. A complex electronic system makes it possible to pre-program intensity changes of the spectrometer's magnetic field in order to register alternately of selected mass spectrum peaks.

The device registering the intensity of ion beams dispersed in the magnetic field is a D.C. electrometer with input resistance of  $10^{12} \Omega$  or  $10^{10} \Omega$ . The minimal intensity of the registered ion current is  $10^{-14}$  A. A vacuum-tight manipulator makes it possible to aim the ion current either at a collector connected to the electrometer described above or at an ion-electron multiplier which can register single ions.

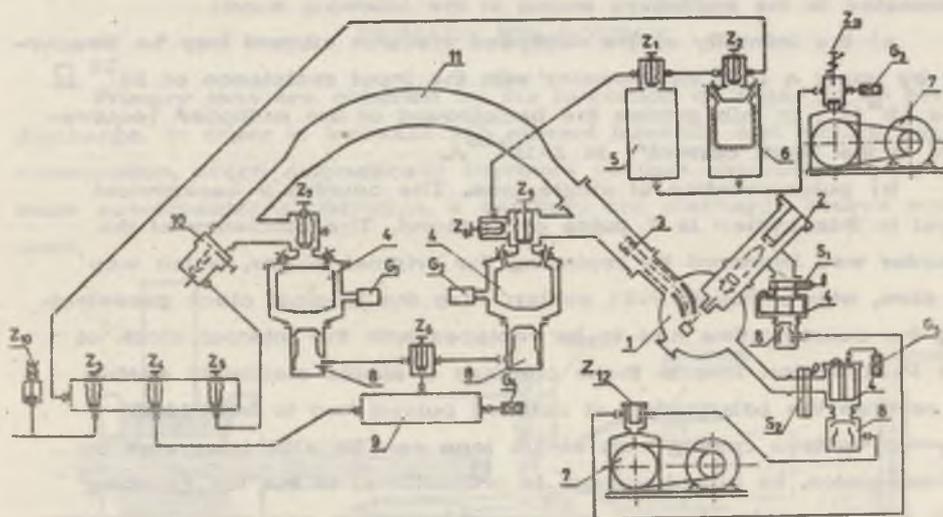


Fig. 1. The vacuum system scheme of modernized mass spectrometer MI-1201, 1-sample chamber with the cylindrical electrostatic analyser of the secondary ions energy, 2-primary ion source ( $\text{Ar}^+$ ), 3-accelerating and focusing system of the secondary ions, 4- L-N cool-traps, 5-absorber, 6-sorption pump, 7-rotary pumps, 8-diffusion pumps, 9-rough vacuum, 10-ion counter SI-03, 11-deflection chamber  $G_1$ - $G_5$ -vacuum-meters,  $Z_1$ - $Z_{12}$ - vacuum valves,  $S_1$ ,  $S_2$ -vacuum locks

The adaptation of the analysing part of the mass spectrometer to secondary ion analysis required the adjustment of the magnetic lens in such a way as to obtain a maximum resolving power with the ion accelerating voltage reduced to 2 kV. When the transmission was 2 %, the obtained resolving power was  $\frac{M}{\Delta M} = 600$  (with an accelerating voltage of 4 kV). When the accelerating voltage was reduced to 2 kV (that was necessitated by the distribution of voltage in the primary ion source), the spectrometer transmission decreased to 1 %, and the resolving power to 400.

In order to increase the spectrometer sensitivity, an SI-03 ion counter, made in USSR, was installed instead of the collector-electrometer system. The counter consists of two measuring systems: a traditional collector-electrometer system and an ion-electron multiplier system whose current amplification factor is  $2,5 \cdot 10^6$  at the supply voltage of the multiplier equal 3,5 kV.

Ion current intensity can be measured by the electronic systems connected to the multipliers output in the following ways:

a) the intensity of the multiplied electron current may be measured by using a D.C. electrometer with the input resistance of  $10^{10} \Omega$  and  $10^8 \Omega$ . In this system the background of the multiplier (equivalent to the "dark current") is  $2 \cdot 10^{-18} \text{ A}$ .

b) pulse-counting of single ions. The counter's background level in this system is 1 pulse per second. The efficiency of the counter was improved by replacing the original scaler, which was too slow, with a Polish P-21 scaler. Also the original clock preselecting the counting time had to be replaced with the internal clock of the P-21 scaler. Due to these changes a simple electronic system to reverse the polarization of counted pulses had to be added.

c) pulses coming from single ions can be also integrated by an integrator. Its output voltage is proportional to the ion counting rate and it can be directed to a recorder which can plot mass-spectrum peaks at the level of  $10^{-18} \text{ A}$ .

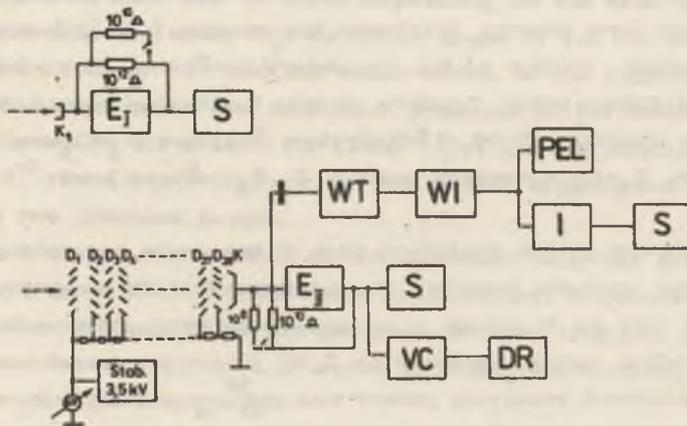


Fig. 2. Block scheme of ion current detection system.  $K_1$ -ion collector with electrometer  $E_1(E_1)$ ,  $D_1$ - $D_{28}$ -ion-electron multiplier, S-recorder, WT-follower, WI-pulse amplifier, PEL-scaler, I-integrator, VC-digital voltmeter, DR-printer

A block scheme of the modified electronic measuring system is shown in Fig. 2. The upper part of this scheme illustrates the possibility of registering an ion current of  $10^{-8}$  to  $10^{-12} \text{ A}$  by using collector  $K_1(K_1)$  and electrometer  $E_1$ .

## PRIMARY ION SOURCE

Primary ions are obtained by the ionization of a gas in an arc discharge. In order to increase ion current intensity and reduce gas consumption, which automatically improves vacuum conditions of the mass spectrometer's operation, a capillary arc discharge source was used.

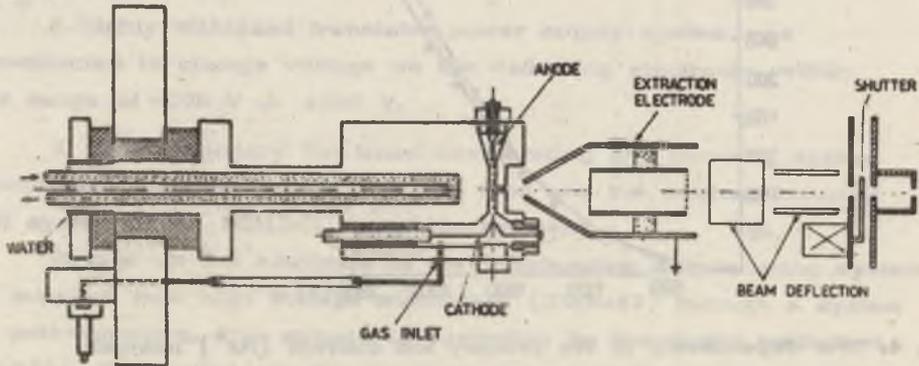


Fig. 3. Schematic diagram of primary ion source construction

The scheme of the ion source is shown in Fig. 3. The discharge chamber of the source was made from an aluminium block and was cooled by water. The low-pressure arc discharge takes place in a capillary between the anode and the cathode. The capillary is 3,5 mm in diameter and 10 mm in length. Half-way between the ends of the capillary there is a hole 2,5 mm in diameter through which positive ions extracted by the extracting electrode are directed to the focusing system. The cathode was made from a tungsten wire 0,5 mm in diameter. The anode in the form of a tungsten rod was placed at the capillary's end. Because the volume of the discharge space was small and the ion source chamber was gas tight, gas consumption was reduced significantly, which in the source described was between 7 and 10 ccm per hour.

Due to plasma condensation caused by the capillary discharge the intensity of the ion current emitted from the described source was much higher than in the case of a simple arc discharge source.

The characteristic of the capillary ion source, i.e. the dependence of the ion current intensity (measured behind the extracting electrode) on the extracting voltage, is shown in Fig. 4.

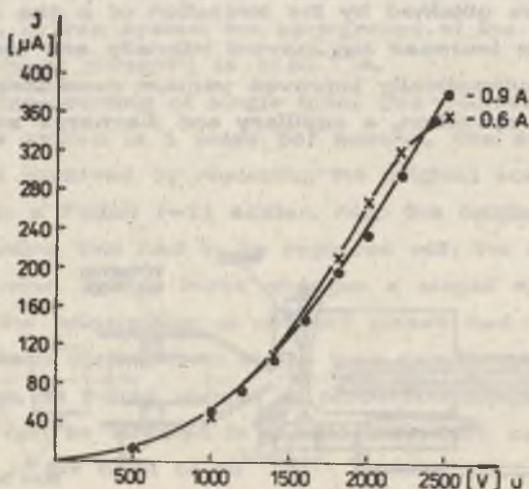


Fig. 4. The dependence of the primary ion current ( $\text{Ar}^+$ ) intensity on the extracting voltage

After connecting the ion source to the chamber of the SIMS spectrometer the density of the ion current was measured on the target and the primary ion beam's diameter was determined. When the voltage accelerating  $\text{Ar}^+$  ions was 3 kV, the obtained ion current density was  $0,5 \text{ mA/cm}^2$ . The bombardment area on the sample's surface was a circle 4 mm in diameter.

#### CONSTRUCTION OF ANALYSING CHAMBER, SAMPLES TABLE, SECONDARY ION ENERGY ANALYSER, ACCELERATING AND FOCUSING SYSTEM

1 The analysing chamber was made from stainless steel (type 1HN9T). Inside the chamber there is a rotating table which allows to examine individually four different samples placed on the table's sides.

The primary ion beam emitted from the source is directed to the examined sample and causes emission of secondary ions from the analysed material.

2. Because of a considerable energy distribution of obtained secondary ions ( $0 \leq E \leq 100$  eV), an energy analyser (a cylindrical energy filter) of secondary ions was placed between the examined sample and the mass spectrometer.

When the accelerating voltage was 200 V, (the potential difference between the sample and the input slit of the energy analyser system) the resolving power of the energy analyser is

$$\frac{E}{\Delta E} = 20.$$

A highly stabilized transistor power supply system was constructed to change voltage on the deflecting electrodes within the range of  $-200$  V  $\div$   $+200$  V.

3. The secondary ion beam accelerating and focusing system comprises of cylindrical electrostatic lens and the original focusing slit system of the MI-1201 mass spectrometer.

Voltage on the electrode of the accelerating and focusing system is supplied from high voltage stabilizers (ZWN-42) through a system of potentiometers. The voltage is controlled by the digital voltmeters (V-628). The voltage on the spectrometer original slits is supplied by the high voltage stabilizer of the MI-1201 mass spectrometer.

## RESULTS

To illustrate the results two mass-spectrums of secondary ions are presented: the silicon monocrystal (Fig. 5a) and the polycrystalline copper sample (Fig. 5b).

When the  $\text{Ar}^+$  primary ion beam intensity was  $70 \mu\text{A}$ , the integral secondary ion beam intensity was  $10^{-9}$  A, ( $10^{-13}$  A for  $^{64}\text{Cu}$ , and  $10^{-12}$  A for  $^{28}\text{Si}$ ). The sputtering rate of the Cu sample at the above  $\text{Ar}^+$  ion intensity was about  $300 \text{ \AA}/\text{min}$ .

The authors hope that by equipping the standard MI-1201 mass spectrometer with a secondary ion apparatus, the applicability of this spectrometer can be extended to investigations of post implantation solid surface phenomena. Moreover, through isotopic investigations one could expect to develop some practical applications in geology and geochronology.

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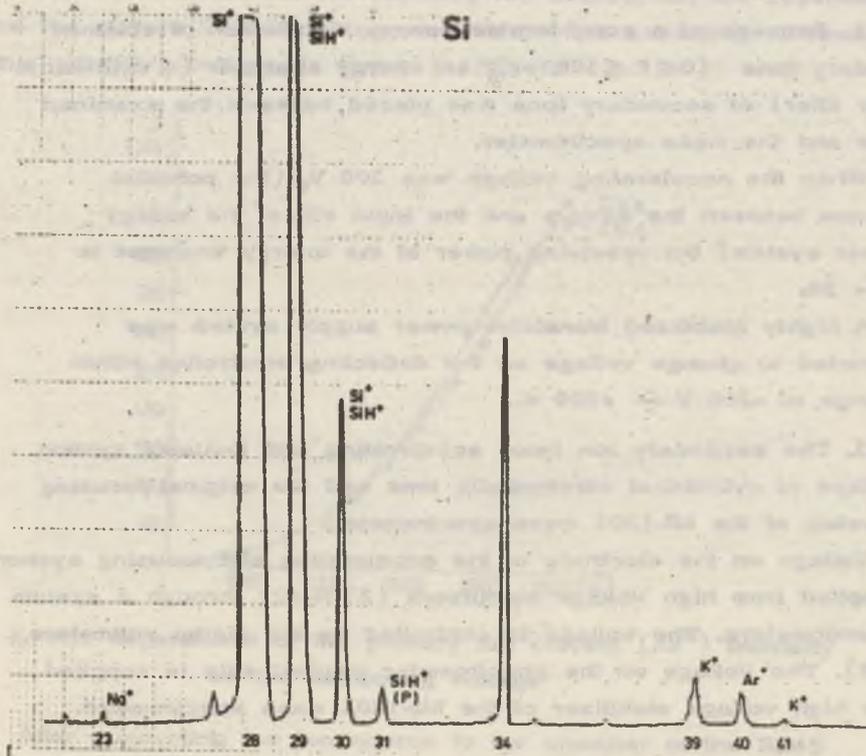


Fig. 5a. Secondary ion mass spectrum of silicon crystal sample

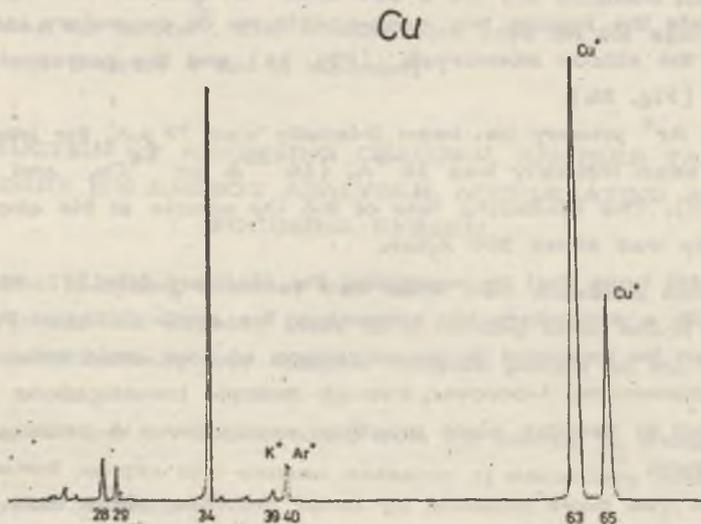


Fig. 5b. Secondary ion mass spectrum of polycrystalline Cu sample

## REFERENCES

1. Benninghoven A., Plog C., Treih N.: Int. J. Mass Spectrom. Ion Phys. 13, 415 (1974).
2. Benninghoven A.: Surf. Sci. 53, 596 (1975).
3. Estel J., Hoinkes H., Kaarmann H., Nahr H., Wilsch H.: Surf. Sci. 54, 393 (1976).
4. Magee C., Chung P. Wu.: Nucl. Instr. Methods, 149, 529 (1978).
5. Degreve F., Figaret R., Laly P.: Int. J. Mass Spectrom. Ion Phys., 29, 351 (1979).
6. Benninghoven A.: Surf. Sci., 28, 541 (1973).
7. Muller A., Benninghoven A.: Surf. Sci., 41, 493 (1974).
8. Schubert R., Tracy J.: Rev. Sci. Instr., 44, 487 (1973).
9. Dawson P.: Int. J. Mass Spectrom. Ion Phys.: 17, 423 (1975).
10. Dawson P., Redhead P.: Rev. Sci. Instr., 48, 159 (1977).

## STRESZCZENIE

W pracy przedstawiono sposób dokonanej modernizacji spektrometru mas typu MI-1201 produkcji ZSRR, w celu umożliwienia dokonywania analiz mas jonów wtórnych, wybijanych w procesie rozpylenia z próbki ciała stałego. Podano uzyskane parametry zmodernizowanej aparatury oraz zamieszczono widma mas jonów wtórnych z monokryształu krzemu i polikrystalicznej próbki miedzi, ilustrujące pracę spektrometru.

## РЕЗЮМЕ

В работе представлен масс-спектрометр вторичной ионной эмиссии построенный на базе масс-спектрометра МИ-1201. Приведены новые параметры спектрометра, а также масс-спектры вторичных ионов из монокристалла кремния и поликристаллической меди.

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