



# Metal oxide collectors for storing matter technique applied in secondary ion mass spectrometry



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## ABSTRACT

We present results of the use of metal and metal oxide substrates that serve as collectors in 'storing matter', the quantitative technique of secondary ion mass spectrometry (SIMS). This technique allows separating the two base processes of secondary ion formation in SIMS. Namely, the process of ion sputtering is separated from the process of ionisation. The technique allows sputtering of the analysed sample and storing the sputtered material, with sub-monolayer coverage, onto a collector surface. Such deposits can be then analysed by SIMS, and as a result, the so called 'matrix effects' are significantly reduced. We perform deposition of the sputtered material onto Ti and Cu substrates and also onto metal oxide substrates as molybdenum, titanium, tin and indium oxides. The process of sputtering is carried within the same vacuum chamber where the SIMS analysis of the collected material is performed. For sputtering and SIMS analysis of the deposited material we use 5 keV Ar<sup>+</sup> beam of 500 nA. The presented results are obtained with the use of stationary collectors. Here we present a case study of chromium. The obtained results show that the molybdenum and titanium oxide substrates used as collectors increase useful yield by two orders, with respect to such pure elemental collectors as Cu and Ti. Here we define useful yield as a ratio of the number of detected secondary ions during SIMS analysis and the number of atoms sputtered during the deposition process.

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## 1. Introduction

Secondary ion mass spectrometry SIMS is a very sensitive technique to perform depth profile analysis of semiconductor layered structures. The main problem of the SIMS analysis is so-called matrix effect [1]. Such effect arises due to dependence of the ionization efficiency of analyzed elements of their immediate atomic neighbourhood.

The measure of the matrix effect is useful yield (UY) of the individual components of the analyzed sample. UY is defined as a ratio of the secondary ion counts divided by the quantity of sputtered matter per unit time. During the sputtering process, besides neutral species, secondary ions are formed and their ionization is highly dependent on adjacent surface atoms.

Neutral particle emission is much less influenced by matrix effects [2]. Basing on this Slodzian [3] proposed separation of sputtering and ionization processes. According to his concept, analysed material should be sputtered and deposited on the collector,

forming a trace of a sub monolayer thickness. Thus only the collector should determine the observed matrix effect. This technique has been called "storing matter" (SM) [4]. It has been applied to study several inorganic and organic materials, using rotating collectors made of pure materials such as silicon, germanium, gold, silver, copper, tantalum, etc. [5,6]. Geometry of the applied deposition setup causes that the amount of material deposited on the collector surface is small. Namely only 1% [7] or 0.5–2% [8] of the sputtered material reaches the collector surface.

The process of sputter deposition of analysed material and the process of analysis of the deposit usually are conducted in two different vacuum chambers. Between these chambers the collector is transported in a vacuum suitcase [9]. In our experimental setup the process of sputter deposit and the process of analysis of the deposit are performed in single analytical chamber [10]. This allows reducing the time of analysis as well as reducing possible contamination effects.

Our experimental setup allows the use of rotating collectors at a rate of one rev./hour. In this study, however, we use stationary collectors, and the purpose of this study is to select a suitable collector of a relatively high UY coefficient. In this work, we focus on an

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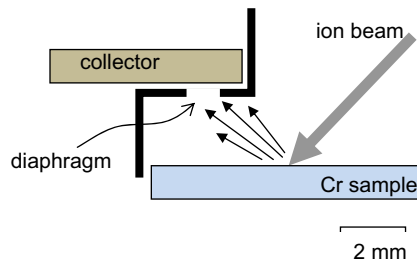


Fig. 1. Schematic view of experimental system up to scale.

analysis of positive secondary ions. This prompted us to apply oxygen-rich substrates such as molybdenum oxide, indium oxide, indium-tin-oxide, titanium-molybdenum oxide and titanium oxide. For comparison we used also two pure elemental collectors: copper and titanium. SM technique is applied with the above collectors and chromium is chosen as deposited material.

## 2. Experimental

The measurements were carried out in the ultra-high vacuum chamber of SIMS analytical equipment SAJW-05 [11]. The system is equipped with quadrupole mass spectrometer QMG422 Balzers and Physical Electronics ion gun. The base pressure in the chamber is  $1 \cdot 10^{-9}$  mbar. Incidence angle of primary ion beam ( $\text{Ar}^+$ , 5 keV) is  $45^\circ$ . The experimental setup is shown in Fig. 1. The system is equipped also with two stage vacuum lock, which is used to introduce samples, in this case chromium (analytical grade 99.99%), and also collectors.

SM procedure consists of sputter deposition of chromium onto the collector through a 1 mm diameter tantalum diaphragm. The

distance between the diaphragm and the sample surface is 2 mm. After deposition of chromium, the collector is inverted up side down, with the use of special fork [10], and then analyzed. The time required for inverting and positioning of the collector is approximately one minute. For the analysis of the collector and sputter deposition of chromium we use 500 nA, 5 keV,  $\text{Ar}^+$  ion beam of a 200  $\mu\text{m}$  diameter. Scanning area of the beam is  $2 \text{ mm} \times 2 \text{ mm}$ . Beam position, when it is in the center of the eroded crater, is presented schematically in Fig. 1.

Collectors are made from 1 mm thick titanium sheet (99.8% purity). The collectors were etched in a mixture of hydrochloric, nitric and hydrofluoric acid (12:4:1 ratio), then rinsed in distilled water and finally washed in an analytically pure isopropyl alcohol. Further processing of collector substrate includes vacuum deposition of several analytically pure oxides as  $\text{MoO}_3$ ,  $\text{TiO}_2$  and  $\text{In}_2\text{O}_3$  from molybdenum boat. Initial pressure in the vacuum coater is below  $3 \cdot 10^{-6}$  mbar. During the process of vacuum deposition, pressure is not higher than  $1 \cdot 10^{-5}$  mbar in the case of  $\text{MoO}_3$  and  $\text{In}_2\text{O}_3$ . Due to the higher evaporation temperature of  $\text{TiO}_2$ , in this case the pressure in the chamber is lower than  $8 \cdot 10^{-5}$  mbar. Also we deposited indium tin oxide by RF plasma from a target composed of In (82%) and Sn (18%). During evaporation of oxides in some cases, chemical reduction occurs and therefore obtaining deposit of exact stoichiometry is difficult. The deposits are amorphous since XRD analysis revealed no crystallization of the deposited layers. During evaporation of  $\text{TiO}_2$ , molybdenum oxides were deposited additionally by co-evaporation. Also  $\text{TiO}_2$  coverage was obtained by thermal oxidation of collectors made of 1 mm thick titanium. Fig. 2 shows secondary ion mass spectra of the four selected collectors: pure titanium substrate,  $\text{TiO}_2$ ,  $\text{Mo}_x\text{O}_y$  and  $\text{Ti}_x\text{Mo}_y\text{O}_z$ . The lowest ion current of  $m/z = 52$  Da ( $^{52}\text{Cr}^+$  background) is registered for  $\text{Mo}_x\text{O}_y$  collector.

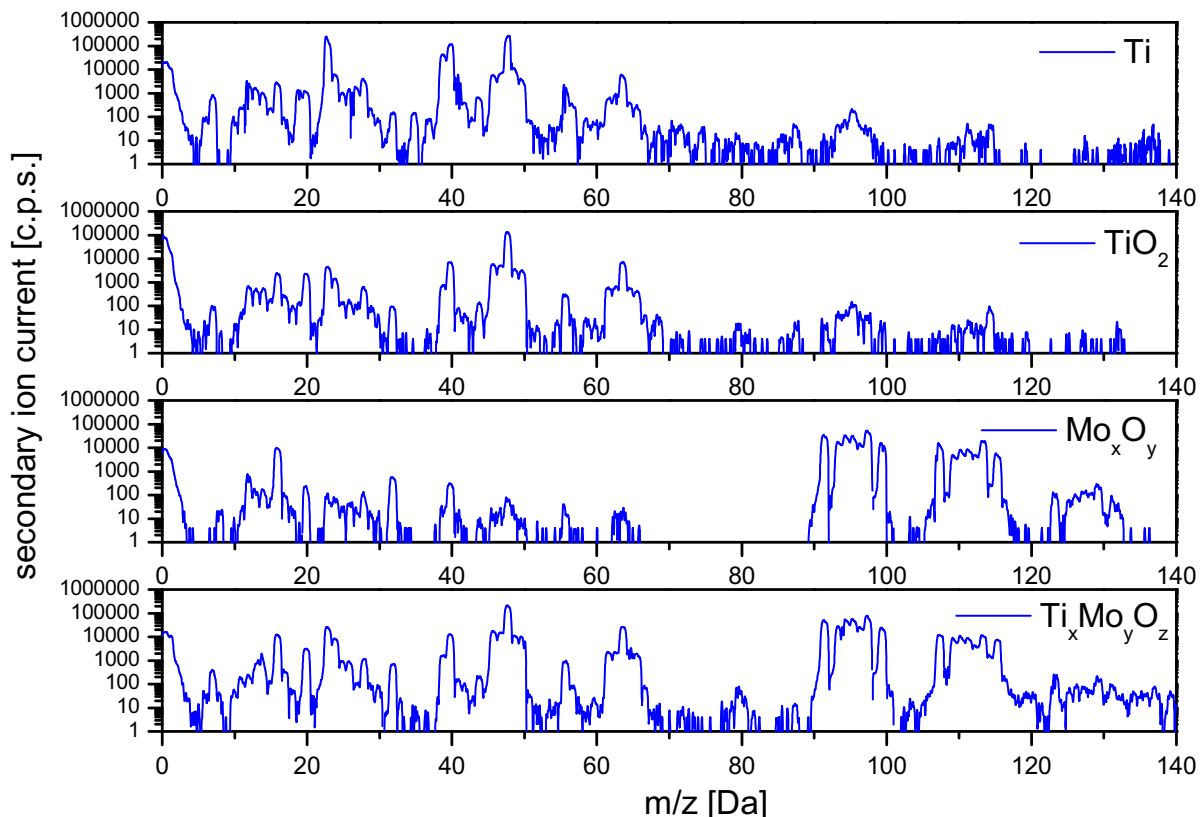


Fig. 2. Mass spectra in 0–140 Da range of three selected collectors: pure titanium,  $\text{TiO}_2$ ,  $\text{Mo}_x\text{O}_y$  and  $\text{Ti}_x\text{Mo}_y\text{O}_z$ .

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