Element analysis of thin films and liquid dry residue by X-ray and ion beam methods

IOP Conf. Series: Journal of Physics: Conf. Series **1281** (2019) 012011 doi:10.1088/1742-6596/1281/1/012011

Element analysis of thin films and liquid dry residue by X-ray and ion beam methods

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Abstract. The work discusses procedure peculiarities of thin films, surface layers and liquid dry residue elements diagnostics. There are showed that the ion beam analysis embellished by TXRF method is necessary and sufficient for element analysis of material surface layers. Experimental data of thin film surface layers obtained by TXRF, RBS and PIXE methods are presented.

It is known that the element analysis of thin film coatings, material surface layers and liquid dry residue films deposited on Be substrates can be realized by different surface sensitive instrumental methods [1–4]. There are emission methods, surface probing by fast and slow beams of electrons, Auger, infrared, Raman, laser and photoelectron spectrometries, mass-spectroscopy different types as well as ion beam material analysis and X-ray fluorescence spectrometry in conditions of exciting beam total external reflection (TXRF). All surface investigation methods are characterized by own advantages and shortages and every diagnostical procedure has own boundaries of experimental applicability. Furthermore, it is a need to take into account a possible destruction of the experimental procedure. A very important characteristic of the experimental method is a degree of the experimental measurements absoluteness. The designated factors comparison showed that the best efficiency of surface layers element diagnostics can be achieved at the joint application of Rutherford backscattering spectrometry (RBS) with PIXE and TXRF methods.

X-ray and ion beam analytical methods are not destructive procedures. But the main diagnostical feature of RBS spectrometry is the absoluteness of obtained experimental data [5]. This method feature is connected with possibility to calculate of ions energy losses in result of its interaction with material electron subsystem before and after scattering by nuclei of atoms, which forms the studied object. RBS spectra can be approximated by computer programs taking into consideration high energy ions braking and scattering peculiarities [6, 7].

Sample of similar approximation is presented on figure 1 showed experimental and theoretical RBS spectra of He^+ ions (E_0=1.24 MeV) obtained for $SrTiO_3$//SiO_2//Si target. The theoretical model approximating experimental data allowed to define thickness of perovskite coating (t=23 nm) and silicon oxide film (t=430 nm) and to classify both films as stoichiometri composition. The approximation accuracy of theoretical adjustment is not better as 1% owing to the experimental base about ion beam braking in material structures was systematized with similar accuracy [8]. High energy ions scattering cross-section defines of element detection limits of material pollutions. This parameter is usually not better as 0.1% at. But in specific cases we can get improving results.

Figure 2 shows experimental and theoretical spectra RBS of He^+ and H^+ ions obtained for the Be polished plate prepared by powder hot pressing. Spectra approximation demonstrated that the plate element composition is $Be_{0.93}O_{0.07}$. Main pollutions of the material are Fe (0.1% at) and W (0.02% at).

The detection limit for W atoms is connected with very great mass in comparison with the host atoms. Plate surface has the adsorbed film with thickness t=10 nm and CO_2 composition. RBS is the nanoanalytical instrument and it characterizes by depth resolution Δt=10 nm in case of He^+ ion beam application (in specific conditions Δt=2 nm) and Δt=50 nm in case of H^+ ion beam use. Material testing depth penetration by He^+ ion beams correlates with range 2–5 micrometers, and in case of H^+ ion beams use the testing depth range can reach up to 25 micrometers.

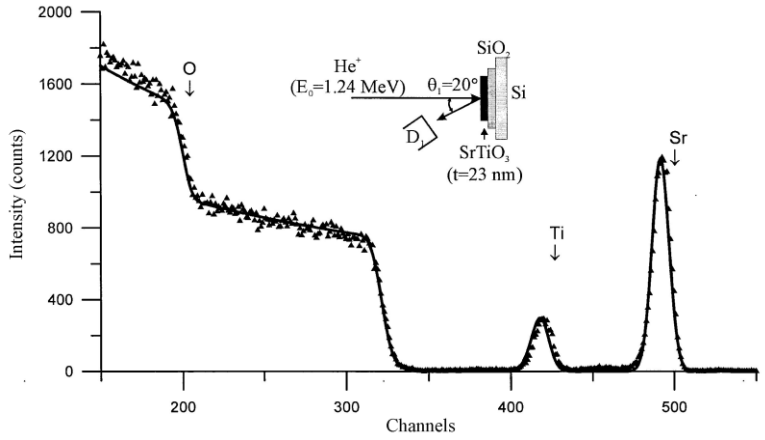

Figure 1. Experimental and theoretical RBS He^+ ions (E_0=1.24 MeV) spectra for the $SrTiO_3/SiO_2/Si$ thin film structure. Arrows show the ion scattering energies on nuclei of atoms located on the target surface. Measurement geometry is shown on insert. Energy step 1.9 keV/channel.

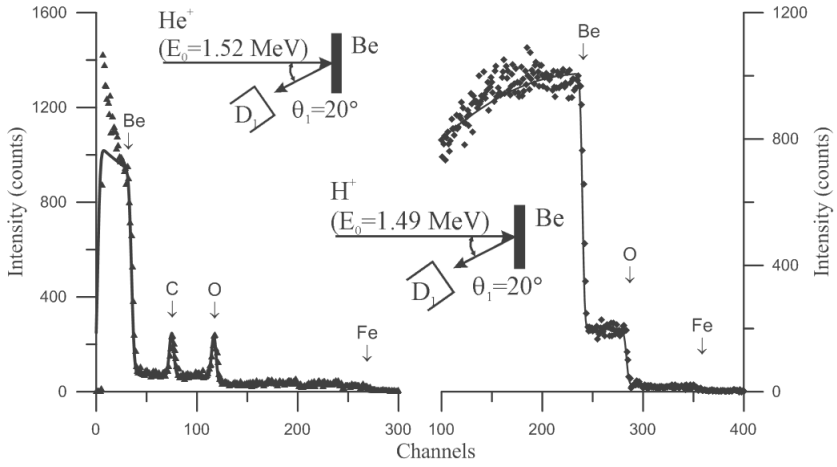

Figure 2. Experimental and theoretical RBS spectra of He^+ and H^+ obtained for Be polished plate. Arrows show ion scattering energies on nuclei of atoms located on target surface. Energy step 1.9 keV/channel.

Be polished plates can be used with success for liquid dry residue element analysis. Figure 3 shows experimental and theoretical RBS spectra of H^+ ions (E_0=0.953 MeV) obtained for the petroleum film deposited on Be polished substrate. The film composition is $H_{0.098}C_{0.900}S_{0.019}N_{0.001}$. Hydrogen concentration was determinated by the nuclear elastic recoil method [9]. The figure contains PIXE and TXRF spectra of the petroleum sample, too. These methods use allows to define the main pollution concentration (table 1). It is interesting that the petroleum sample contains vanadium and lanthanum atoms.

In the last time a specific interest is to sewing fabric with metal coating. This interest is connected with the fabric ability for defence from electromagnetic radiation. Figure 4 shows experimental and theoretical RBS spectra of H^+ ions (E_0=1.24 MeV) obtained for the sewing fabric sample covered by

copper film and TXRF and PIXE spectra of it. RBS spectrum approximation allows to define the copper coating thickness (t=52 nm) and sewing fabric host element composition ($H_xC_{1.5}O_{1.0}$). In this case the hydrogen element concentration did not determinate.

Table 1. Pollutions concentrations detected in the petroleum sample by use TXRF and PIXE methods. Concentration data is related to the sulphur atoms content.

S	Na	Cl	Ca	V	Fe	Ni	La
1	0.005	0.005	0.005	0.015	0.001	0.005	0.0005

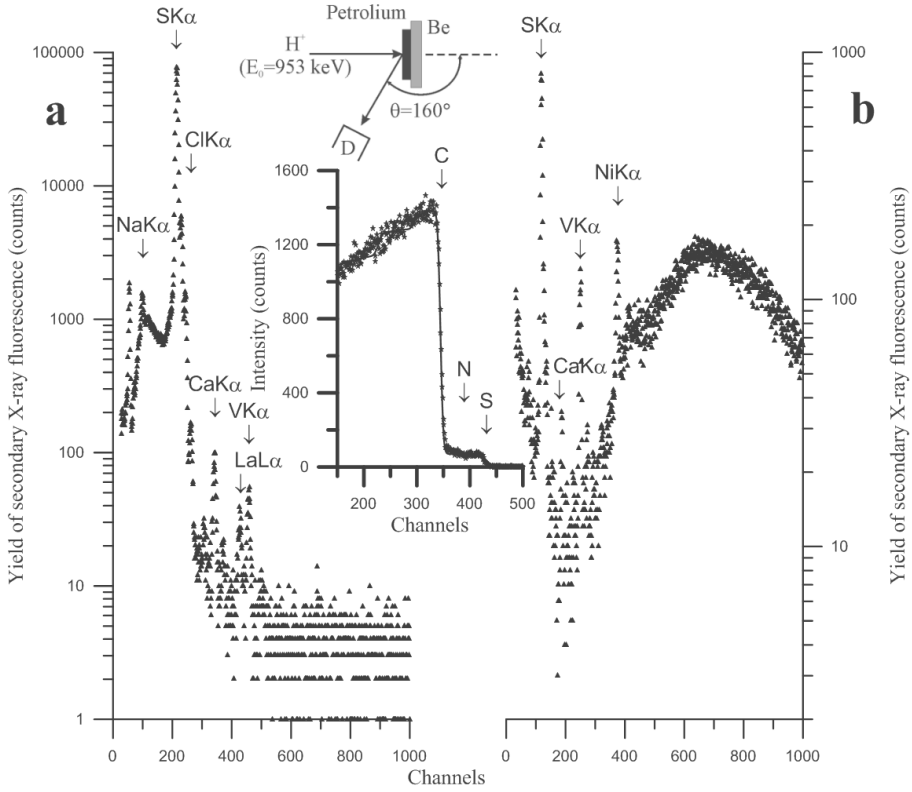

Figure 3. X-ray fluorescence spectra obtained for petroleum film deposited on Be plate excited by H^+ ion beam (a), and in the total external reflection conditions with MoKα radiation flux use (b). Energy steps 10 eV/channel and 20 eV/channel, accordingly. RBS spectrum of H^+ ion beam for the target is presented on insertion. Energy step 1.9 keV/channel.

TXRF measurements showed considerable quantity of Zn atoms content and small concentration of Ca and Fe. Owing to that TXRF measurements characterize an element composition of thin surface layer with thickness near 5 nanometers one can maintain that additional elements are present at the copper coating. Zn atoms concentration is near 7% at and Fe and Ca quantities are not exceeded 1% at. PIXE spectrum of the sewing fabric sample demonstrates pollutions great variety. Its concentrations are not great but its detection in the fluorescence yield spectrum is connected with very high effective excitation [10]. There is important that the PIXE spectrum does not show ZnKα and ZnKβ fluorescence lines. This discrepancy is explained by difference in the measurement geometry of TXRF and PIXE methods. TXRF spectrometry presents data about average element composition of all target surface layer with very small thickness. PIXE spectrum reflects average atomic composition of the target microvolume defined by ion beam cross-section and depth of its penetration into the target. In the result, we can conclude that Zn atoms are distributed inn the coating irregularly. Other X-ray fluorescence lines except CuKαβ ones characterize the pollutions set of the fabric. Main element of

the set is Ti atoms. Additional investigations displayed that Ti atoms distribute in the fabric volume is uniform with content near 0.1% at.

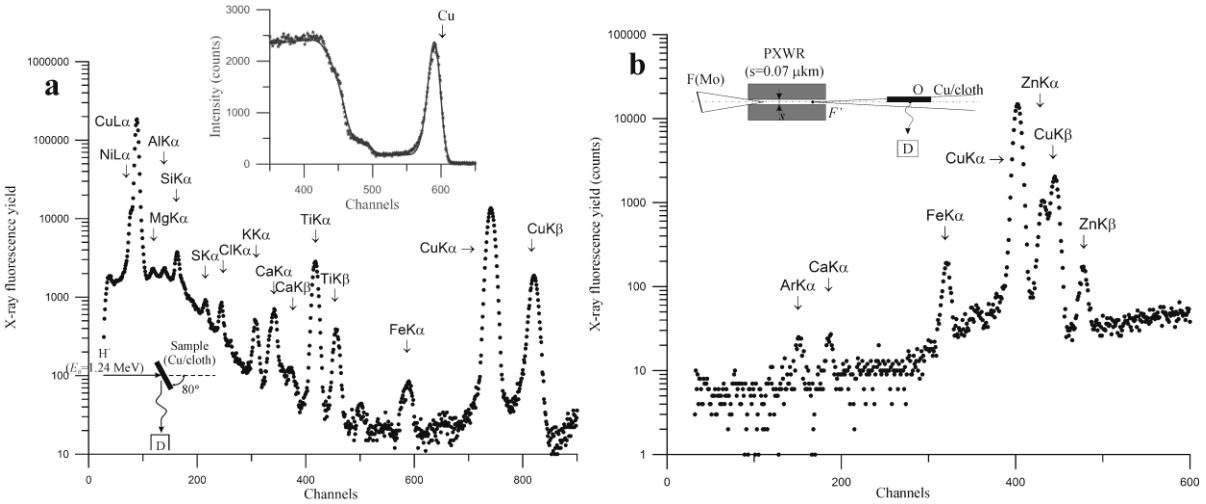

Figure 4. X-ray fluorescence spectra obtained for the sewing fabric sample with Cu coating excited by H^+ ion beam (a) and in the total reflection conditions with use of the planar X-ray waveguide-resonator for MoKα exciting beam formation (b). Energy steps 10 eV/channel and 20 eV/channel, accordingly. RBS spectrum of H^+ ion beam for the target is presented on insertion. Energy step 1.9 keV/channel.

The investigation as a whole showed that the element diagnostics of thin film surface layers can be executed with high efficiency at target complex study by RBS, TXRF and PIXE methods.

Acknowledgments
This work produced by State task #075-00475-19-00 and has been partially financial supported by RFFI (grant #19-07-00271 and #19-08-00019).

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