TXRF spectrometry at ion beam excitation

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Abstract. The work presents short discussion of TXRF and PIXE methods peculiarities. Taking into account of these peculiarities we elaborate the experimental scheme for TXRF measurements at ion beam excitation of characteristical fluorescence. The scheme is built on base of the planar X-ray waveguide-resonator with specific design. Features of the new experimental method and possibilities of Sokol-3 ion beam analytical complex were used for the method application in real measurements.

1. Introduction

X-ray fluorescence analysis in conditions of the total external reflection of the exciting radiation flux on the studied material surface (TXRF) is today the most effective method for quantitative elemental diagnostical material objects on base of X-ray fluorescence application [1]. The method realization in the total external reflection conditions provides excitation of a surface layer with thickness 3–5 nm. So, X-ray fluorescence yield excited in these conditions characterizes the element composition of thin surface layer. Moreover, the background deposit into TXRF spectrum has very small value, and the spectrum dispenses with the need for the matrix correction [2].

TXRF application allows decreasing of pollution detection limits on 1.5–2 orders in comparison with XRF conventional measurements. At the same time, TXRF analysis as well as conventional X-ray fluorescence spectrometry at excitation of the fluorescence yield by X-ray fluxes, gamma radiation and electron beams has some difficulties at the light elements diagnostics in materials. It is illustrated by figure 1 presenting cross-sections of K_α and L_α characteristical fluorescence excitation by MoK_α radiation flux for different elements. YK_α and AlK_α cross-section comparison shows that the fluorescence yield of these lines is different on four orders.

Fluorescence difficulties of light element diagnostics in materials can be compensated by ion beam excitation of X-ray fluorescence yield (by PIXE method) [3]. The mechanism of fluorescence excitation by ion beams differs from ones featured to other excitation methods. PIXE bears similarly to the fluorescence yield produced by electron excitation but it is characterized by small value of the bremsstrahlung photon intensity yield. In the result, PIXE spectrometry demonstrates low levels of the background in comparison with the electron beam excitation of the fluorescence yield. But the main preference of the PIXE method is high cross-section of X-ray fluorescence excitation for light elements. Experiments showed that proton and helium ion beams with energy near 1 MeV excite the light element fluorescence more effective in comparison with the fluorescence of heavy elements [4]. Figure 2 allows to compare the cross-section of X-ray fluorescence excitation for AlK_α and YK_α lines

in condition of proton beam (E_0 = 1 MeV) application. It is apparent that AlK$_\alpha$ line excitation is effective on three orders approximately.
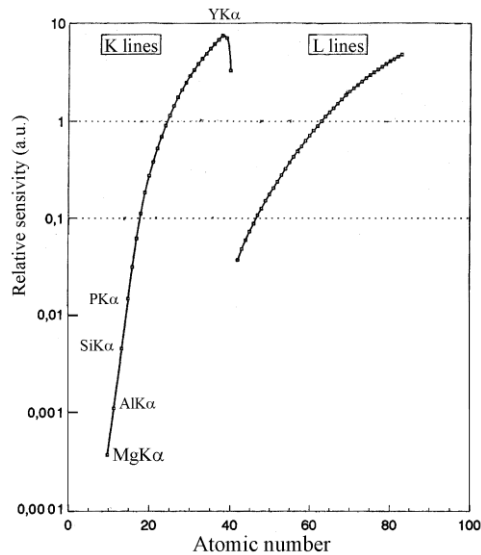

Figure 1. Cross-sections excitation for K$_\alpha$ and K$_\beta$ different elements lines yields in conditions of MoK$_\alpha$ radiation flux using as the exciting agent [1].
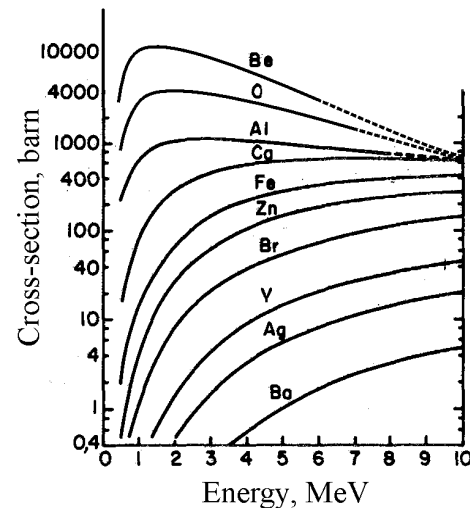
Figure 2. Energy dependences of the cross-section excitation for K$_\alpha$ different elements lines yield in conditions of H$^+$ ion beam using of the exciting agent [4].

2. PIXE method in TXRF condition

TXRF spectrometry registries of X-ray fluorescence yield from thin film surface layer. Unlike TXRF method PIXE spectrometry is characterized by registration of the total fluorescence yield excited in the layer, which thickness is defined by the ion beam penetration into volume of the studied material. Moreover, the real yield of the characteristic fluorescence will be defined by absorption factor featured for the material. So, every lines will characterize the layer with thickness defined by own absorption factor. TXRF method is free from the matrix influence. PIXE measurements must take into account this factor. Suppression of the matrix factor influence is not simple task. At the same time, removal of the fluorescence yield dependence from layer absorption thickness is not difficult problem. This task can be solved by application of the specific X-ray optical scheme for X-ray fluorescence yield registration executed on base of the planar X-ray waveguide-resonator (PXWR) with hole in its central position (figure 3).
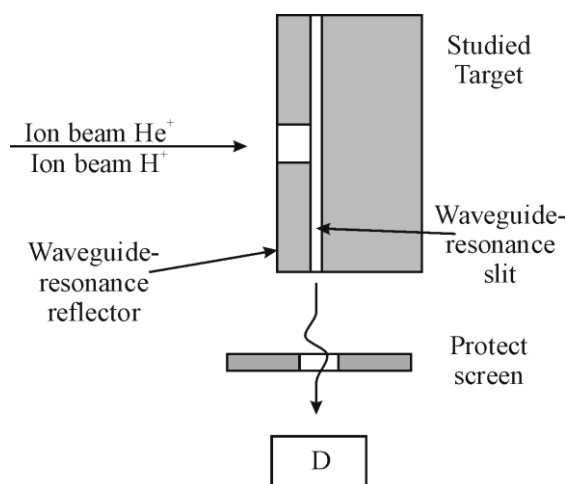

Figure 3. Scheme of X-ray fluorescence yield corresponding to thin surface layer allowed using ion beam excitation in conditions of the planar X-ray waveguide-resonator application. PXWR slit width 0.15 μm.

This scheme allows forming the waveguide-resonance structure by surface of the studied material and the polished surface of Be reflector carried Ti strips on its edges with thickness 0.15 μm. In the result, we have the waveguide-resonance slit clearance for X-ray fluorescence lines of light elements. Proton ion beam incidents on the target through hole in the Be reflector and excites the target characteristic fluorescence. At the same time, X-ray optical scheme provides registration of X-ray fluorescence yield for thin surface layer of the target with thickness 3–5 nm. This registration scheme is hybrid of PIXE and TXRF investigation methods but it does not deliver from the matrix effects influence.

3. TXRF in the PIXE conditions

TXRF spectra collected in conditions of ion beam fluorescence excitation is the best support for RBS spectra approximation for material involving light elements. Figure 4 demonstrates RBS spectrum of H^+ ion beam ($E_0 = 1.18$ MeV) for multielement target containing of the light element set. Spectrum shows some vagueness in Al, Si and P atomic concentration owing to not enough of its energy resolution. In this case the TXRF information obtained at ion beam excitation is very useful. Figure 5 shows X-ray fluorescence yield spectrum of the multielement target collected on condition of excitation by proton beam with energy $E_0 = 1.18$ MeV. The spectrum demonstrates SiK_α, AlK_α and NaK_α intensity lines, envelope of PK_α and ZrL_α composite line and small peak of OK_α. This spectrum approximation allows removing some vagueness at RBS spectrum interpretation and makes it possible to determinate element composition of the multielement target. It is $Na_3Zr_{1.3}Si_{1.9}Al_{0.1}P_{1.0}O_{12}C_2$. It is important to notice that the PIXE spectrum collected in the conventional conditions showed perceptible differences in comparison with spectrum presented in figure 5. It is suggested that the comparison of TXRF spectra collected in conditions of ion beam and X-ray excitations permits to elaborate the objective method allowed to evaluate the matrix effect influence on TXRF spectra ion beam excitation.

4. Conclusion

The work presents short discussion of TXRF and PIXE method and suggest new procedure of TXRF and PIXE hybrid investigation. It is shown that the new method can be used for investigation of multielement targets containing light element set.
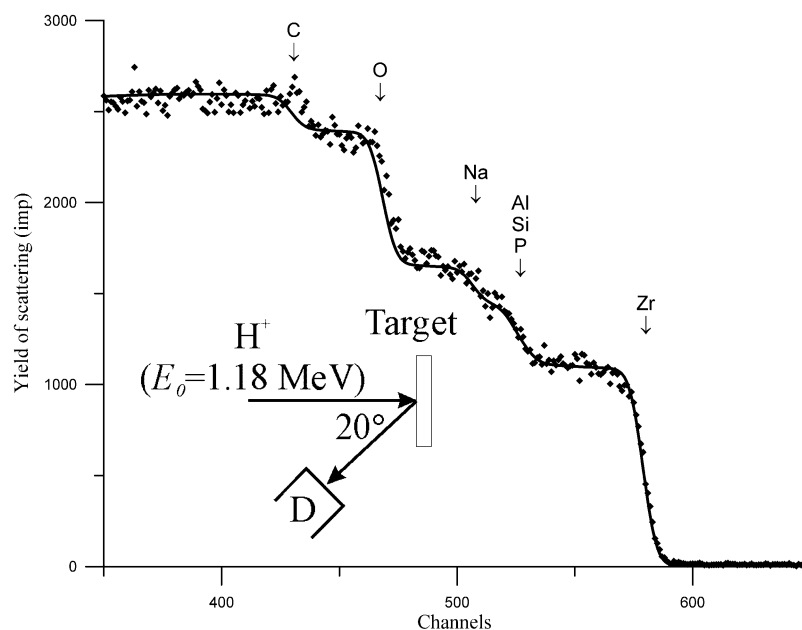

Figure 4. Experimental and theoretical spectra RBS H^+ ion beam ($E_0 = 1.18$ MeV) for multielements target collected for θ = 160° scattering angle. Arrows show energies corresponding to scattering on nuclears of surface atoms. Energy step 1.9 keV/channel.
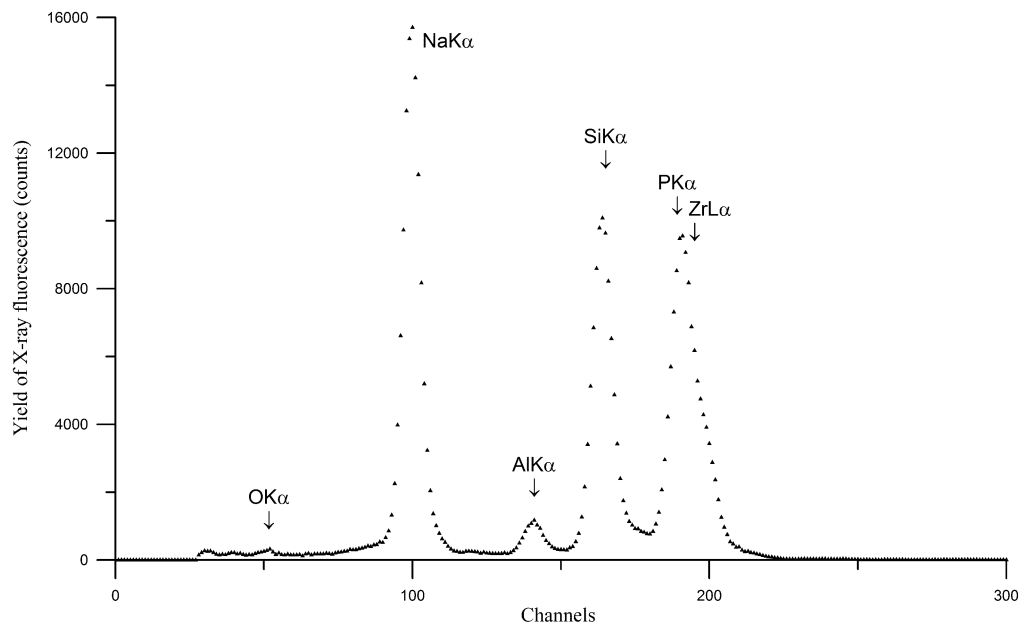
Figure 5. TXRF spectrum collected for multielement target in conditions of excitation by H^+ ion beam (E_0 = 1.18 MeV) at PXWR with specific design application. PXWR slit width was 0.15 μm. SSD registrator was equipped by Be window 8 μm. Energy step 10.2 eV/channel.

References
[1]   Klockenkamper R 2014 *Total reflection X-ray fluorescence analysis* (New York: Wiley)
[2]   Van Grieken R and Markowicz A A 2002 *Handbook of X-ray spectrometry* (New York: Dekker)
[3]   Johanson S A E, Campbell J L and Malqvist K G 1995 *Particle induced X-ray emission spectrometry (PIXE)* (New York: Wiley)
[4]   Cahill T A 1980 *J. Annu. Rev. Nucl. Part. Sci.* **30** 211