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Cross sections of X-ray production induced on Ti, Fe, Zn, Nb and Ta by O, Cl, Cu and Br ions with energies between 4 MeV and 40 MeV



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ABSTRACT

Differential cross section of X-ray production induced by O, Cl, Cu and Br ions with energies between 4 MeV and 40 MeV have been measured for thin targets of Ti, Fe, Zn, Nb and Ta in a direct way. A fully characterized silicon drift diode was used as X-rays detector. Beam currents have been measured by a system of two Faraday cups. Corrections for target thickness effects have been applied to the raw data. Experimental cross sections are compared both with theory and with previously published results. Experimental results from other authors are in reasonable agreement with ours over a wide energy range. Theory produces consistent results in the case of oxygen ions but gives cross sections even orders of magnitude below the experimental ones for heavier ions (ECPSSR-UA) or contrasting results (PWBA) depending on the ion-target combination.

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1. Introduction

The multi-technique character of Ion Beam Analysis (IBA) allows combining Secondary Ion Mass Spectrometry (SIMS) and particle-induced X-ray emission (PIXE) performed with light and medium-mass ions. SIMS, with incident ions at MeV energies (MeV-SIMS), has been recently established as an analytical method, relatively simple and cheap to implement [1-4]. The International Atomic Energy Agency (IAEA) has promoted a Coordinated Research Project (CRP F11019) [5] meant at consolidating, MeV-SIMS as an analytical technique and developing the combined application of quantitative Heavy-Ion PIXE (HI-PIXE) analysis. With this goal, a specific effort has been made by the CRP partners to increase the availability of X-ray production cross sections [6,7], in particular for the ion-target combinations that were identified as the most interesting for a combined MeV-SIMS and HI-PIXE sample characterization. Using the measurement protocols and data analysis procedures of our previous study [7] for C and Si ions, we have extended the investigation to the production of X-rays induced by O, Cl, Cu and Br ions with energies between 4 MeV and 40 MeV on thin targets of TiO₂, Fe₃O₄, ZnO, Nb₂O₅ and Ta₂O₅

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grown on different substrates. This corresponds to a maximum of 1.66 MeV/u in the case of O ions and a minimum of 0.12 MeV/u when Br ions are used. The X-ray production cross sections are presented and discussed.

2. Experimental set-up

The experimental set-up was described in connection with the measurement of cross sections for HI-PIXE induced by C and Si ions at the 5 MV tandem accelerator of the Centro de Micro-Análisis de Materiales (CMAM), Universidad Autónoma de Madrid (UAM), Spain. Full account of the beamline and end station, as well as of the detector performance and target sizes and composition can be found in a previous article [7]. We just recall that a few targets were distributed amongst the CRP partners. They have been produced by atomic layer deposition (ZnO, TiO₂, TiN), sputtering (Ta₂O₅) or reactive sputtering (Nb₂O₅, RuO₂). In addition, the targets TiN-C and Fe₃O₄-C were produced at UAM by ion beam sputtering on a silicon substrate, using a Kaufman-type ion source. Target thicknesses, reported in Table 1, have been extracted from the analysis of elastically backscattered 2 MeV alpha particles. The estimated uncertainty of the target thicknesses is on average around 2.5%.

The charge was measured comparing the data provided by an insertion (IFC) and a transmission Faraday Cup (TFC), both with a secondary electrons suppressor. The IFC is used to determine the

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Table 1Thickness of the used targets as determined by 2 MeV alpha backscattering. Targets TiN-C and Fe₃O₄-C were produced at UAM by ion beam sputtering.

| Targets | Analyte thickness [μg/cm²] | Target thickness [μg/cm²] | Density [g/cm³] |
|---|-------------------------------|------------------------------|--------------------|
| TiN/Si | 11.2 ± 0.4 | 14.5 | 4.82 |
| TiN-C/Si | 19.1 ± 0.5 | 28 | 4.82 |
| TiO ₂ /Si | 11.6 ± 0.2 | 19.4 | 4.23 |
| ZnO/Si | 22.9 ± 0.4 | 29 | 5.61 |
| Fe ₃ O ₄ -C/Si | 64 ± 2 | 86 | 5 |
| Nb ₂ O ₅ /Sigradure | 14.3 ± 0.2 | 21 | 4.6 |
| Ta ₂ O ₅ /Sigradure | 15.9 ± 0.4 | 19.8 | 8.18 |

net charge sent to target immediately before and after the irradiation, while the TFC signal is used to record possible current fluctuations and to correct the values provided by the IFC. The TFC intercepts (7.1 ± 1.5) percent of the beam going to the sample, as determined using a 2 MeV proton beam and the ratios of the integrated charges given by the two FC. The uncertainty of the measured charge depends on the stability of current: we observed an average of 2.7% and an extreme value of 11%, considering the O, Cl, Cu and Br ions cumulatively.

X-rays are detected by a KETEK AXAS-A 10 mm^2 silicon drift detector (SDD), at 120° from the beam direction, protected by an absorber ($500 \mu \text{m}$ polyethylene terephthalate PET) from the impact of backscattered ions and protons. Using a 2 MeV proton beam and the X-ray production cross sections compiled in the GUPIX software package [8], we have built the efficiency curve, $\varepsilon_{abs}(E_X)$, as a function of X-ray energy. Its uncertainty is on average 4% (minimum: 2.8%; maximum: 7.2%); the difference with a third order

polynomial fitting curve is on average 7%. For the K-lines we used experimental efficiencies to extract the cross sections. In the case of the Ta L-series, we used the experimental efficiency for the L_{α} line and fitted values for all other lines.

Currents between 2.5 nA and 130 nA have been used for O ions in charge states ranging from 2 to 4 with an average deadtime of 0.3% (see Table 2). Currents between 4.5 nA and 110 nA have been used for Cl ions in charge states ranging from 3 to 7 with an average deadtime of 2.2%; between 0.87 nA and 2.82 nA for Cu ions in charge states 3 to 7 with an average deadtime of 2.0%; between 1.42 nA and 29.2 nA for Br ions in charge states 3 to 7 with an average deadtime of 1.1%. Preamplifier signals have been processed by an ORTEC 572A amplifier and a Fast Comtec 7072 dual analog-to-digital converter. Spectra have been collected and processed with a Fast Comtec MP3 multi-parameter system.

It is important to note that, when using Cu and Br ions, the K lines produced by excitation of the incoming ion, may mix with the target element characteristic lines. In these cases the spectra (see the example of Fig. 1) have been fitted by a series of Gaussian functions plus a user defined continuous background, leaving the peak centroids, widths and heights as free parameters. Doing so we have been able to account for peak energy shifts and broadening [9] of the target element with respect to proton PIXE and to subtract the contribution of the incoming ion peaks to the HI-PIXE spectrum. We estimated a combined standard uncertainty (including the global statistical and fitting uncertainties) on each individual yield between 1% and 5%, being the highest values observed for the less intense peaks (e.g. Nb K_{β} , Ta $L_{\gamma 1}$) or for the peaks most affected by the superposition with the incoming ion ones (e.g. Zn K_{α} and Zn K_{β} peaks excited by Cu ions).

 Table 2

 Differential cross sections for the ion-target combinations detailed in the text.

| Energy | Charge state | Zn K [b/sr] | Ti K [b/sr] | Fe K [b/sr] | Ta L [b/sr] | Nb K [b/sr] |
|--------------|--------------|-------------------------|-------------------------|-------------------------|-------------------------|---------------------|
| O ion | | | | | | |
| 20 | 4 | 9.58E+00 ± 4.83E-01 | 2.23E+02 ± 1.18E+01 | 5.49E+01 ± 3.47E+00 | 6.43E+01 ± 4.09E+00 | 7.37E-01 ± 4.02E-02 |
| 16 | 4 | 3.74E+00 ± 1.49E-01 | 7.06E+01 ± 3.66E+00 | 1.76E+01 ± 1.11E+00 | 2.82E+01 ± 1.67E+00 | 2.48E-01 ± 1.32E-02 |
| 12 | 4 | 1.24E+00 ± 4.93E-02 | 1.57E+01 ± 8.13E-01 | 4.33E+00 ± 2.73E-01 | 1.51E+01 ± 8.95E-01 | 8.58E-02 ± 4.50E-03 |
| 8 | 3 | 2.51E-01 ± 9.83E-03 | 2.85E+00 ± 1.50E-01 | 8.02E-01 ± 5.06E-02 | 4.48E+00 ± 2.61E-01 | 1.53E-02 ± 9.09E-04 |
| 4 | 2 | $1.13E-02 \pm 5.03E-04$ | $1.10E-01 \pm 5.79E-03$ | 2.88E-02 ± 1.79E-03 | 1.60E-01 ± 9.66E-03 | |
| Energy [MeV] | Charge state | Zn K [b/sr] | Ti K [b/sr] | Fe K [b/sr] | Ta L [b/sr] | Nb Ka [b/sr] |
| Cl ion | | | | | | |
| 40 | 7 | 9.41E+00 ± 4.41E-01 | 1.06E+03 ± 6.42E+01 | 1.40E+02 ± 9.65E+00 | 1.06E+02 ± 6.52E+00 | 1.69E-01 ± 9.93E-03 |
| 33 | 6 | 6.36E+00 ± 2.67E-01 | 7.96E+02 ± 4.42E+01 | 9.74E+01 ± 7.11E+00 | 7.01E+01 ± 5.16E+00 | 1.18E-01 ± 8.68E-03 |
| 26 | 5 | $1.74E+00 \pm 6.87E-02$ | 3.78E+02 ± 2.12E+01 | 2.79E+01 ± 1.99E+00 | 3.25E+01 ± 1.88E+00 | |
| 19 | 4 | 4.07E-01 ± 1.62E-02 | 1.46E+02 ± 7.29E+00 | $7.01E+00 \pm 5.33E-01$ | $1.00E+01 \pm 5.82E-01$ | |
| 12 | 3 | $3.47E-02 \pm 2.66E-03$ | 3.97E+01 ± 2.00E+00 | $8.94E-01 \pm 7.17E-02$ | $1.20E+00 \pm 7.15E-02$ | |
| 9 | 3 | $3.39E-03 \pm 2.36E-04$ | 1.57E+01 ± 8.46E-01 | 1.75E-01 ± 1.50E-02 | 2.95E-01 ± 1.95E-02 | |
| Energy | Charge state | Zn K [b/sr] | Ti K [b/sr] | Fe K [b/sr] | Ta L [b/sr] | |
| Cu ion | | | | | | |
| 40 | 7 | 1.79E+02 ± 1.22E + 01 | 1.06E+03 ± 6.21E+01 | 6.97E+02 ± 7.87E + 01 | 4.27E+02 ± 5.39E+01 | |
| 35 | 6 | 1.28E+02 ± 8.69E+00 | 8.39E+02 ± 4.78E+01 | 5.54E+02 ± 6.65E+01 | 3.18E+02 ± 3.29E+01 | |
| 30 | 5 | 9.30E+01 ± 6.34E+00 | 6.58E+02 ± 3.84E+01 | 4.52E+02 ± 4.85E+01 | 2.02E+02 ± 3.37E+01 | |
| 25 | 4 | 5.73E+01 ± 3.90E+00 | 4.80E+02 ± 2.80E+01 | 3.33E+02 ± 3.65E+01 | 1.21E+02 ±2.45E+01 | |
| 20 | 4E- | $4.05E+01 \pm 2.76E+00$ | 3.33E+02 ± 1.94E+01 | 2.25E+02 ± 2.59E+01 | 6.20E+01 ± 1.72E+01 | |
| 15 | 3 | 2.10E+01 ± 1.52E+00 | 1.85E+02 ± 1.36E+01 | 1.51E+02 ± 2.43E+01 | 2.57E+01 ± 1.02E+01 | |
| 10 | 3 | 1.08E+01 ± 6.79E-01 | 9.39E+01 ± 4.79E+00 | 3.93E+01 ± 7.49E+00 | 1.23E+01 ± 4.18E+00 | |
| Energy | Charge state | Zn K [b/sr] | Ti K [b/sr] | Fe K [b/sr] | Ta L [b/sr] | |
| Br ion | · | | · | · | · | |
| 40 | 7 | 1.72E+02 ± 1.13E+01 | 2.57E+02 ± 1.61E+01 | 2.93E+02 ± 3.03E+01 | 2.32E+02 ± 1.72E+01 | |
| 35 | 7 | 1.39E+02 ± 5.99E+00 | 2.23E+02 ± 1.20E+01 | 2.57E+02 ± 2.34E+01 | 2.04E+02 ± 1.34E+01 | |
| 30 | 7 | 1.13E+02 ± 4.35E+00 | 1.67E+02 ± 1.73E+01 | 1.97E+02 ± 1.35E+01 | 1.41E+02 ± 1.83E+01 | |
| 25 | 5 | $5.22E+01 \pm 3.03E+00$ | 8.01E+01 ± 5.17E+00 | 8.99E+01 ± 9.58E+00 | 6.33E+01 ± 4.51E+00 | |
| 20 | 5 | $4.09E+01 \pm 1.84E+00$ | $4.67E+01 \pm 2.47E+00$ | 5.76E+01 ± 3.61E+00 | $3.73E+01 \pm 2.54E+00$ | |
| 15 | 3 | 2.02E+01 ± 8.52E-01 | 2.13E+01 ± 1.09E+00 | 2.32E+01 ± 1.46E+00 | 2.07E+01 ± 1.36E+00 | |
| 10 | 3 | 9.56E+00 ± 3.66E-01 | $0.00E+00 \pm 0.00E+00$ | 1.02E+01 ± 6.29E-01 | 1.30E+01 ± 8.42E-01 | |

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