Developments of the methodology of fluorescence induced by x-ray standing wave

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ELETTRA EXPERIMENTAL REPORT
Proposal number: 20130049
Title: Developments of the methodology of fluorescence induced by x-ray standing wave

Proposer: Philippe JONNARD
Beamline(s): BEAR
Required shifts: 15
Achievements: We performed x-ray fluorescence measurements induced by x-ray standing waves (XSW) in Co/Mg based periodic multilayers. Two different experiments were done when recording the intensity of the CoLa and MgKa emissions as a function of the glancing angle: (i) the glancing angle was varied in the range of the 1st Bragg peak of the incident radiation, i.e. rather grazing (between 0 to 10°); (ii) the glancing angle was varied in the range of the 1st Bragg peak of the fluorescence radiation, i.e. at a large glancing angle (between 110 and 120°). Indeed, in this case it is necessary to put the sample surface close to the detection direction. Two-layer (Co/Mg) and tri-layer (Co/Mg/B4C, Co/Mg/Zr) have been studied for a total of four samples. First the photon energy scale was calibrated through TEY measurements. Then reflectivity measurements were performed at the photon energy of interest for the XSW experiments: 776eV (Co La emission energy), 808 and 998eV (to ionize the Co 2p level and generate the Co La emission), 1301eV (Mg Ka emission energy), 1310, 1332 and 1492 eV (to ionize the Mg 1s level generate the MgKa emission). Then XSW experiments (i) and (ii) were performed.
The achievements are the following:
(1) We observed the modulation of the detected fluorescence emissions in the standing wave mode when the angular range is explored around the Bragg peak of the fluorescence radiation. That is to say the intensity of the characteristics emission produced within the multilayer is modulated owing to the periodic structure of the multilayer. To our knowledge this is the first that such an experiment with synchrotron radiation is reported.
(2) We observed the modulation of the fluorescence emissions in the standing wave mode for both bi-layer and tri-layer systems when working in both angular ranges to diffract the incident radiation or the fluorescence emission. That is to say, fluorescence excited by standing wave can also be applied to tri-layer systems.
(3) We have to run our simulation codes, to interpret the data from the material science point of view to determine if interdiffusion can be evidenced this way. This will be important particularly for the two systems with Zr, which we know from previous studies that they present different behaviors with respect to interdiffusion, depending on the position of the Zr layer within the period.

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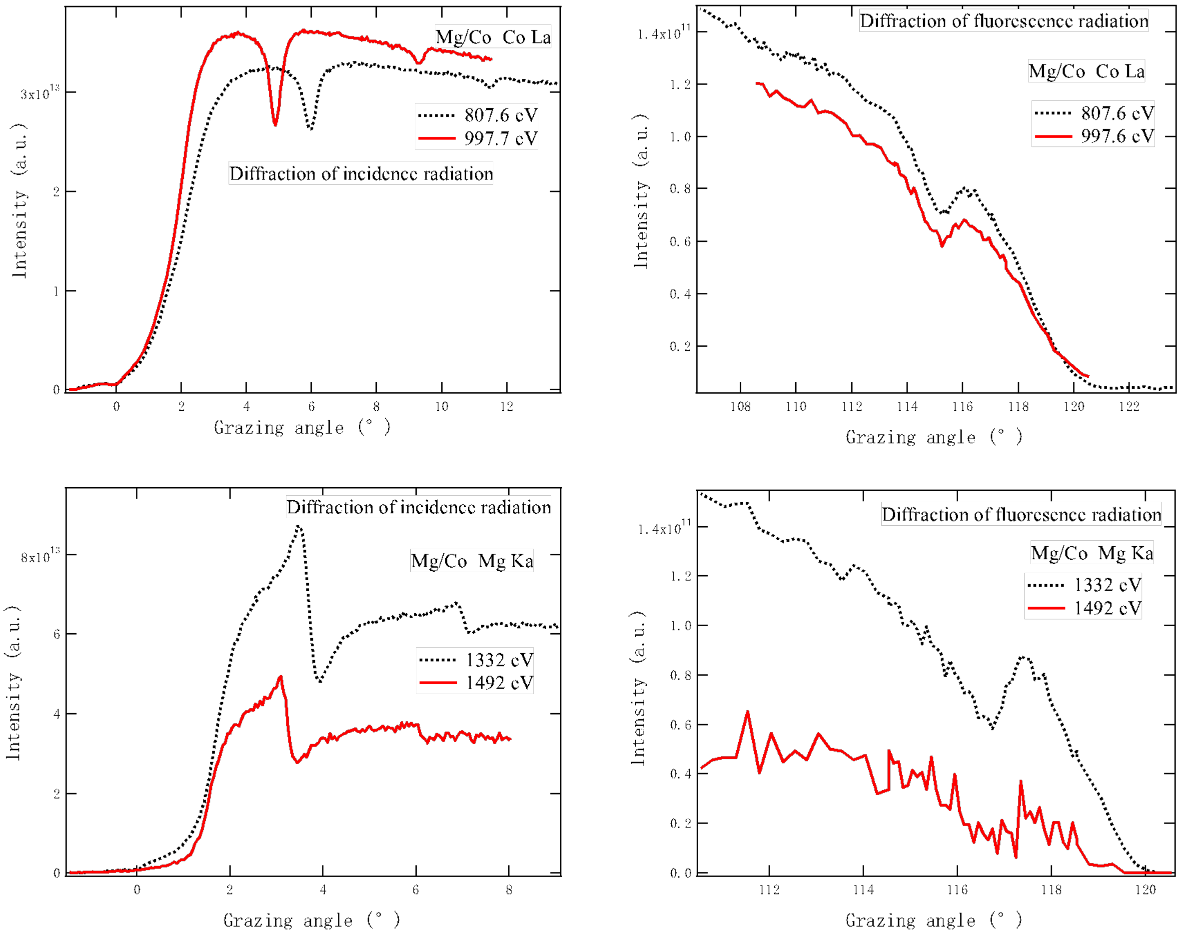
We performed x-ray fluorescence measurements induced by x-ray standing waves (XSW) in Co/Mg based periodic multilayers in the Elettra synchrotron radiation facility, on the BEAR beam line. There are 60° between the direction of the synchrotron beam and the direction of the fluorescence detection. Two different experiments were done when recording the intensity of the CoLa and MgKa emissions as a function of the glancing angle:

(1) the glancing angle was varied in the range of the 1st Bragg peak of the ***incident*** radiation, i.e. rather grazing (between 0 to 10°);
(2) the glancing angle was varied in the range of the 1st Bragg peak of the ***fluorescence*** radiation, i.e. at a large glancing angle (between 110 and 120°). Indeed, in this case it is necessary to put the sample surface close to the detection direction (see the figure in the proposal).

The detailed informations on the sample structures are the following:
(1) Si/$Cr_{2.7\ nm}$/[$Mg_{5.2\ nm}$/$Co_{3.5\ nm}$/$B4C_{0.1\ nm}$]$_{30}$/$B4C_{3.4\ nm}$
(2) Si/[$Mg_{5.45\ nm}$/$Co_{2.55\ nm}$]$_{30}$/$B4C_{3.5\ nm}$
(3) Si/[$Mg_{5.45\ nm}$/$Zr_{1.5\ nm}$/$Co_{2.55\ nm}$]$_{10}$/$B4C_{3.5\ nm}$
(4) Si/[$Mg_{5.45\ nm}$ /$Co_{2.55\ nm}$/$Zr_{1.5\ nm}$]$_{10}$/$B4C_{3.5\ nm}$

We worked with *s*-polarized radiation. The photon energy was calibrated by measuring the Co L3 and Mg K absorption edges obtained in the TEY mode. The detection of the characteristic emissions was done by an SDD. Then reflectivity measurements were performed at the photon energy of interest for the XSW experiments: 776eV (Co La emission energy), 808 and 998eV (to ionize the Co 2p level and generate the Co La emission), 1301eV (Mg Ka emission energy), 1310, 1332 and 1492 eV (to ionize the Mg 1s level generate the MgKa emission). Then XSW experiments (1) and (2) were performed.

In the figure, we show as an example the results regarding the Co/Mg multilayer for both (1) and (2) experiments, for different incident photon energies and for the two monitored characteristic emissions. In experiments (1), the incident photon emission is involved and thus we observe that the positions of the structures depend on the glancing angle (through the Bragg law). In experiments (2), characteristic emissions, whose photon energy are fixed, are involved and thus we observe that the positions of the structures do not depend on the glancing angle. Let us note that owing to angular resolution differences, in experiment (1) both 1st and 2nd Bragg peaks are observed, while in experiment (2) only one is clearly observed. This is due to the fact that, in the first case the resolution is determined by the aperture of the slit for the incoming beam (0.01°), whereas in the second case it is determined from the angular aperture of the SDD detector (0.9°).



Fluorescence XSW experiments for the Co/Mg sample. The glancing angle is scanned in two ranges: small angles (left figures) to diffract the incident radiation, experience (1); large angles (right figures) to diffract the characteristic emissions, experience (2). The intensity of the Co Lalpha (top figures) and Mg Kalpha emissions (bottom figures) are monitored.

As a conclusion:

(1) We observed the modulation of the detected fluorescence emissions in the standing wave mode when the angular range is explored around the Bragg peak of the fluorescence radiation. That is to say the intensity of the characteristics emission produced within the multilayer is modulated owing to the periodic structure of the multilayer. To our knowledge this is the first time that such an experiment with synchrotron radiation is reported.

(2) We observed the modulation of the fluorescence emissions in the standing wave mode for both bi-layer and tri-layer systems when working in both angular ranges to diffract the incident radiation or the fluorescence emission. That is to say, fluorescence excited by standing wave can also be applied to tri-layer systems.

(3) We have to run our simulation codes, to interpret the data from the material science point of view to determine if interdiffusion can be evidenced this way. This will be important particularly for the two systems with Zr, which we know from previous studies that they present different behaviors with respect to interdiffusion, depending on the position of the Zr layer within the period.