[IV~2] [초청]

Development of a new XIEES system for materials analysis

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Introduction

X-ray induced electron emission (XIEE) is the phenomenon which is observed in the whole range of x-rays from soft to hard region. A couple of well-known techniques for characterization of solids such as XPS, AES, SWT and (S)XAFS are based on it. However each of them has its own theoretical and experimental principles which can not be cross-applied and moreover can not be used for simulation of the phenomenon in general. This is mostly because these independently developed techniques utilize narrow and almost non-overlapping regions of x-ray photon energies. The similar situation is in the hardware developments. Thus a task of creating a united general basis of the XIEE has arisen inevitably. The effect which occurs in the whole range of x-rays and therefore may be taken as the fundamental base for solving the stated task is defined. This is the jump-like increase of the emission at the electron binding energies (absorption edges). On this basis we developed a new X-ray Induced Electron Emission Spectrometer(XIEES) as follows.

Instrumentation

Because of the necessity of changing simultaneously several experimental parameters (x-ray photon energy, sample irradiation angle, electron emission take-off angle, etc.) none of the experimental devices available on market could satisfy these demands. This caused the development of the original vacuum x-ray electron spectrometer. Here we report the XIEES device called STAR (Samsung's Test and Analysis of Radiations). Its specific design features and technical characteristics allow to realize more than 10 original and well known techniques for characterization of solids, for example, (a) EXAFS & XANES at transmission, electron yield, fluorescence and total reflection modes, (b) SWT by electron registration, (c) double crystal x-ray diffraction, (d) nondestructive depth profiling of surface thin layers.

Experimental device is based on the 18 kW MAC Science rotating anode x-ray generator M18XHF[22] as the source of x-rays. Spectrometer is of Johansson type with Rowland radius of 600 mm. One of the original features is the direct connection of the spectrometer chamber to the generator vacuum chamber (the standard Be foil window is removed and both generator and spectrometer are connected by the bellow with a gate valve). Together with the use of the drum system with a 6-crystals monochromator and computer-controlled stepping motor exchanger of the crystals during experiment this design allows to go down to 480 eV over the x-ray photon energy. Upper limit is determined by the maximal voltage and the actual geometry and is of 41 keV. Thus, having the range from 41 keV down to 480 eV in one experiment, the STAR system is believed to have the widest x-ray photon energy range and therefore the largest experimental opportunities in the world.

X-ray beam from generator anode (usually Mo) passes through the bellow into the spectrometer chamber. Inlet collimator is the substitutable slit that limits the beam divergence. For diminishing the vertical divergence a Soller type slit may be installed as well. Beam passes along the main linear drive on which one of the ends of the solid Rowland sector is located. Second end is sliding freely along the guide that is fixed at the angle of 80^0 to the main linear drive (that determines the maximal Bragg angle). Sector is mounted on the three sapphire supports for smooth sliding over the bottom of the chamber and in order to minimize the driving power. Crystal-monochromator system is fixed on the sector that allows to scan over the Bragg angle by the same linear movement. This system contains a drum with six true focusing crystals (basic set is: LiF(422), LiF(200), Ge(111), Ge(111)flat, PET(002) and KAP(100)) and a stepping motor based drive for exchanging the working crystal during experiment together with adjustment rocking. The sample-detector unit is moving along the sector by means of stepping motor in accordance with Bragg law. This unit contains the receiving slit, sample holder, monitoring shoot-through gas-filled ionisation chambers, the channeltron and the x-ray gas-filled proportional detector. All detectors except monitoring ones are turned coaxial with the sample that allows to choose the take-off and irradiation angles as necessary.

The servo system is consisted of 10 stepping motors that can control independently all the movements (including alignment and adjustment movements) in the vacuum chamber (1×10^{-6} torr) of special design.

Computer control system is made on the base of PC. It is propelled by the STAR program which was developed completely new with Borland C++. As well as full automatic operation, the program includes to itself the main control panel for "manual" operation with direct access to each device, experiment planning panels, alignment utilities, statistical utility, etc. Special compiler is developed and implemented. Software allows the external programming and remote control via networks.

Technical Characteristics

Technical characteristics of the STAR system had been measured and the most important are as follows:

- Range of the x-ray photon energy: 480 eV - 41 keV
- Resolution at 10 keV: 2.3×10^{-4}
- Energy set reproducibility: ± 0.08 eV at 10 keV
- Energy scanning step: 0.001 eV at 10 keV
- Sample irradiation angle: any angle (0^0 -360^0)
- Irradiation angle scanning step: 1 arcsec
- Electron emission registration: any take-off angle in respect to the sample surface.

Conclusion

The creation of an original experimental equipment together with development of theoretical basis allows to consider XIEE as the new independent branch in electron emission field.

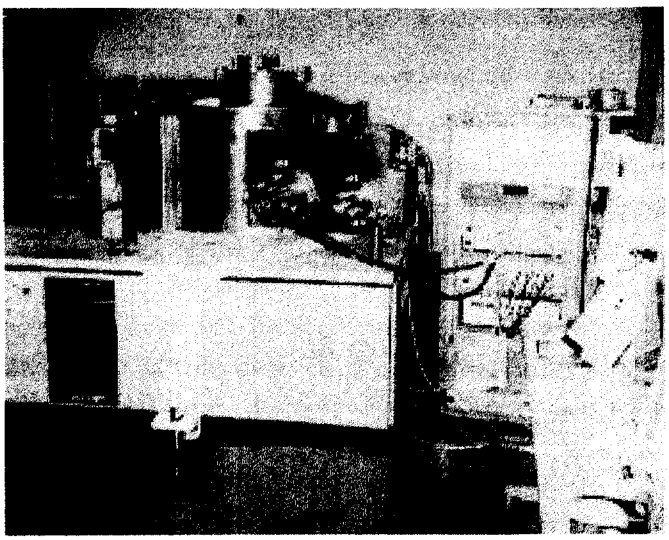

Fig. 1. The XIEES system which was developed at SAIT.