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A LABORATORY ReflEXAFS SPECTROMETER

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Résumé:

Nous présentons un spectromètre simple, spécialement construit pour les mesures de ReflEXAFS. Il utilise un tube de rayons-X conventionnel associé à un miroir parabolique. Un monochromateur à deux cristaux (LiF) permet d'obtenir une résolution de 8,5 eV à 8 keV. Le faisceau incident est monitoré par un compteur proportionnel et le faisceau réfléchi est mesuré par un compteur à scintillation (NaI) qui permet d'éliminer les harmoniques par discrimination d'énergie. Le spectromètre est utilisable de 7 à 18 keV. Les spectres relatifs à la surface d'un échantillon de nickel avant et après oxydation thermique sont présentés.

Abstract:

A simple spectrometer using a conventional X-ray generator has been constructed for ReflEXAFS measurements at glancing angle. The X-ray source is a sealed X-ray tube with a parabolic mirror. A monochromator with two flat LiF crystals is used. The energy resolution amounts to 8.5 eV at 8 keV. A proportional counter monitors the incident X-ray intensity and a NaI scintillation detector measures the intensity of the reflected beam. This system is free from fluctuations of the incident X-ray intensity and harmonics can be rejected with the energy discrimination. The range of energy is 7 to 18 keV. The ReflEXAFS spectra related to the surface of a nickel sample and a thermally oxided nickel sample are presented.

INTRODUCTION.

The use of a synchrotron radiation source is very attractive to perform EXAFS measurements because of its unique properties of high flux, small beam divergence, structureless spectrum, and polarization. However the difficulty of accessing such facilities has generated a considerable interest in laboratory systems (1), which can be designed for the user oriented conditions and problems. Several such laboratory facilities have been described (2). However, complicated alignement and control procedures or expensive rotating anode x-ray sources are required. In most cases, they are not suitable for ReflEXAFS experiments.

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We report here a relatively simple device we use to test the suitability of samples for further synchrotron experiments, since the ReflEXAFS experiments require samples of good quality and special experimental conditions.

GENERAL TECHNIQUE:

When an X-ray beam impinges on a surface with a glancing angle smaller than a critical angle θc (< 10 mrad for the usual compound in the energy range of interest), it is nearly totally reflected and contains the EXAFS and XANES characteristics of the top of the surface (penetration depth about 3 nm). To perform this so-called ReflEXAFS technique, a very narrow X-ray beam (< 25 µm) with a low divergence (< 0.1 mrad) is needed, and it is not possible to use classical devices with a bent crystal for enhancing the flux.

MONOCHROMATOR.

The monochromator consists of two separated LiF (200) flat crystals in the classical (+ -) arrangement shown in figure 1. Energy scanning is obtained by directly rotating the whole two-crystals assembly, attached to the mechanical axis of an accurate incremental angle encoder. The angular displacements of the monochromator are thus made with a typical resolution of 0.002 degree. Accurate parallelism between the two crystals can be achieved with a dc motor. The spacing between the two crystals is monitored by a microcomputer in order to avoid a displacement of the beam with the energy.

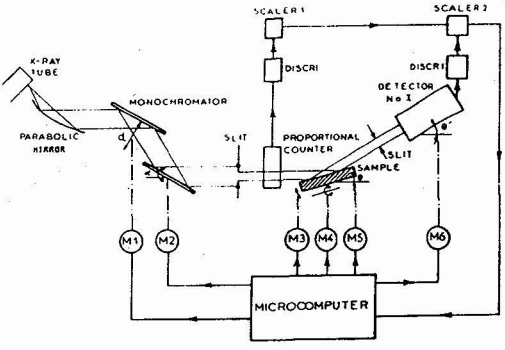


Fig.1. Schematic diagram.

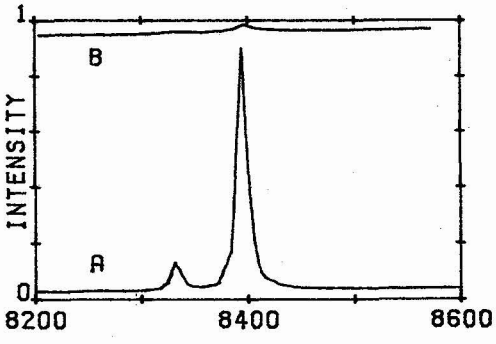


Fig.2. Counting rate of the scintillation counter showing the efficiency of the monitor:
A) without monitor
B) with monitor.

X-RAY SOURCE.

The X-rays are generated by a conventional sealed X-ray tube in its line image position. By choosing anode material such as Mo, characteristic lines which may seriously deform the spectra can be, in principle, avoided in the range 7 to 18 keV. However the intense characteristic $L\alpha$ line of tungsten and other spurious lines cannot be avoided, due to the filament evaporation (as shown further below, this difficulty can be overcome by using a monitor set in the incident beam).

In order to increase the flux without increasing the divergence, a gold-coated parabolic mirror is used. The parabolic form is obtained by bending a glass-plate(160 x 30 x 3 mm^3). Thus the flux is about 7 times higher.

MONITOR AND DETECTOR.

X-rays coming from the first slit (25 μm) pass first through a home-made proportional counter coupled with a fast preamplifier. The counter, equipped with entrance and exit X-ray tranparent windows (kapton), is filled with P10 gas (90% argon and 10% methane) at atmospheric pressure so that it absorbs 10-20% of the X-ray beam depending on the energy range of interest.

The intensity reflected by the sample is measured by a NaI(Tl) scintillation detector chosen for its good noise characteristics. It cannot handle count rates much above 10^5 counts s^{-1} ; but this count rate capability is sufficient for ReflEXAFS measurements where the flux behind the slit never rises above this limit. Harmonics can be rejected with the energy discrimination. All thesignals are counted until the number of photons detected by the first counter reaches the preset number of the dual-scaler. The ratio between the two detectors, without any sample, is shown in figure 2; on account of the lack of detector linearity, the W-Lα caracteristic line is divided by 300.

SAMPLE HOLDER.

In order to make very fine adjustment to the position and the orientation of the sample, a precise goniostat was employed. It allows the alignment of the sample parallel to the beam and in the middle of the beam. Then the angular dependence of the reflectivity can be performed (fig. 3).

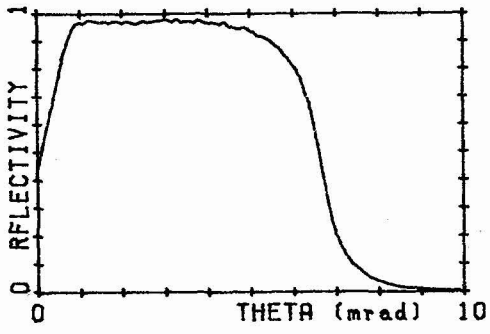


Fig.3. Angular dependence of the reflectivity from Ni at 8200 eV

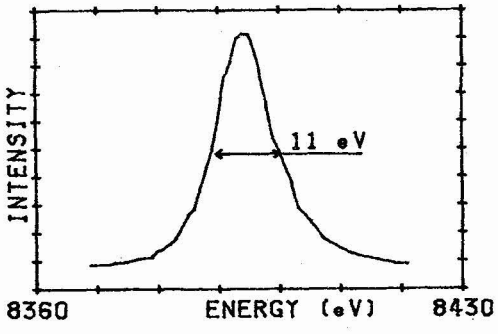


Fig.4. Experimentaly measured W-Lα caracteristic line.

PERFORMANCE AND APPLICATION.

The experimental resolution was deduced from the measured intensity profile of the characteristic emission line (Lα) of the tungsten impurity (fig. 4). Taking the natural line width of about 7.2 eV, the actual energy resolution is evaluated to be 8.5 eV. With a slit width set to 25 μm, the X-ray source operating at 20 kV and 40 mA gives an X-ray flux of 5000 cps at 8 keV.

With this system, samples used for studies of protective oxide formation on metal surface (3) can be tested before the final experiments using the synchrotron radiation source. As an example, figure 5 and 6 show the ReflEXAFS spectra from a nickel sample before and after oxidation (the duration of the data collection per set was about 6 hours). Indeed the spectra are obtained with relatively poor statistics, but it is sufficient to assess the quality of the samples.

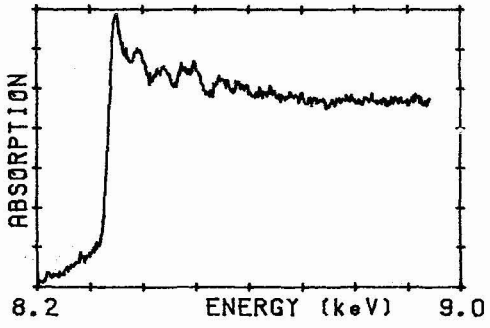


Fig.5. ReflEXAFS spectrum from Ni sample before oxidation. (θ = 5.5 mrad)

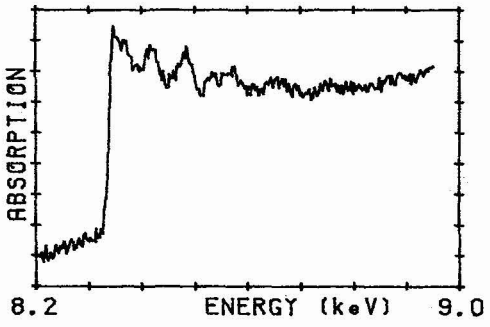


Fig.6. ReflEXAFS spectrum from Ni sample after oxidation. (θ = 5.5 mrad)

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