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VIBRATING SAMPLE HOLDER FOR X-RAY ABSORPTION MEASUREMENTS ON SINGLE CRYSTALS

V. Tullio[1], F. D'Anca[2], F. Campolungo[1], F. D'Acapito[2], F. Boscherini[1,3], S. Mobilio[1,4].

[1)] *INFN, Laboratori Nazionali di Frascati.*
[2)] *INFM, OGG Operative Group in Grenoble.*
[3)] *Università di Bologna.*
[4)] *Università Roma Tre.*

I. Introduction

This technical note reports on the design, construction and performance of a vibrating sample holder in use on the GILDA - CRG beamline at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. It permits to minimize the effects of coherent scattering (Bragg Scattering) when measuring the x-ray absorption coefficient of single crystals.

II. Physics of the problem

The measurement of the energy dependence of the X-ray absorption coefficient of a given atom embedded in a single crystal matrix can be disturbed by Bragg scattering. When the wavelength of the incident radiation λ and the incidence angle on a given set of crystal planes θ verify the Bragg condition:

$$2d \sin\theta = \lambda$$

the beam may interact with the sample both via the expected photoelectric absorption process and also via coherent scattering. The final effect in the measured absorption spectrum are unwanted peaks that, depending on the geometry and on the detector response, may be positive or negative.

The effect can be minimized effectively by making the sample oscillate: in this way the angle θ becomes time-dependent so the Bragg condition is fulfilled only for a short time δt:

$$\delta t = T * (\delta\theta/\Delta\theta)$$

where T is the oscillation period $\delta\theta$ is the angular width of the peak and $\Delta\theta$ the amplitude of the sample oscillation. The reduction of the signal due to diffraction f will then be of the order of

$$f = \delta\theta/\Delta\theta$$

Since $\delta\theta \cong 10$ μrad an oscillation with $\Delta\theta \cong$ a few degrees produces a reduction factor $f \cong 10^{-4}$ that is sufficient to cancel the effect.

III. Description of the apparatus

The system is shown in Fig.1. It consists of:

P1. A copper base needed to fix the apparatus on the cold finger of the cryostat installed on the EXAFS chamber of the GILDA beamline.

P2. An aluminum support bearing at its end an electro-magnet and an optical coupler.

P3. A vibrating metallic plate with holes: samples can be mounted on its front-side and measured both in transmission and fluorescence mode. The plate is realized in Cu-Be, an alloy exhibiting excellent elastic properties and high thermal conduction.

P4. A permanent magnet fixed on the rear side of the vibrating plate

P5. An electro-magnet, used to induce oscillations in the plate

P6. An optical coupler (photodiode + phototransistor) used to measure the oscillation amplitude.

The system is driven and controlled by an electronic circuit in a NIM module. It provides a frequency- and amplitude-variable current for the electro-magnet, amplifies the signal from the optical coupler and supplies alarm signals when the oscillation exceeds high or low oscillation amplitude limits (Fig. 2).

IV. Working principle

The oscillation of the elastic plate is generated by the action of the fixed electromagnet on the permanent magnet. The current in the electromagnet is supplied by a square wave oscillator at variable frequency, contained in the driver circuit. The resonance frequency of the free plate is about 37 Hz and can vary with the plate geometrical dimensions or operating temperature. The amplitude of the oscillations can be easily changed by adjusting the oscillator frequency to approach the free plate resonance value or by varying the amplitude of the oscillating signal in the electromagnet. A value of about 10° of deviation of the elastic plate is normally used for data collection.

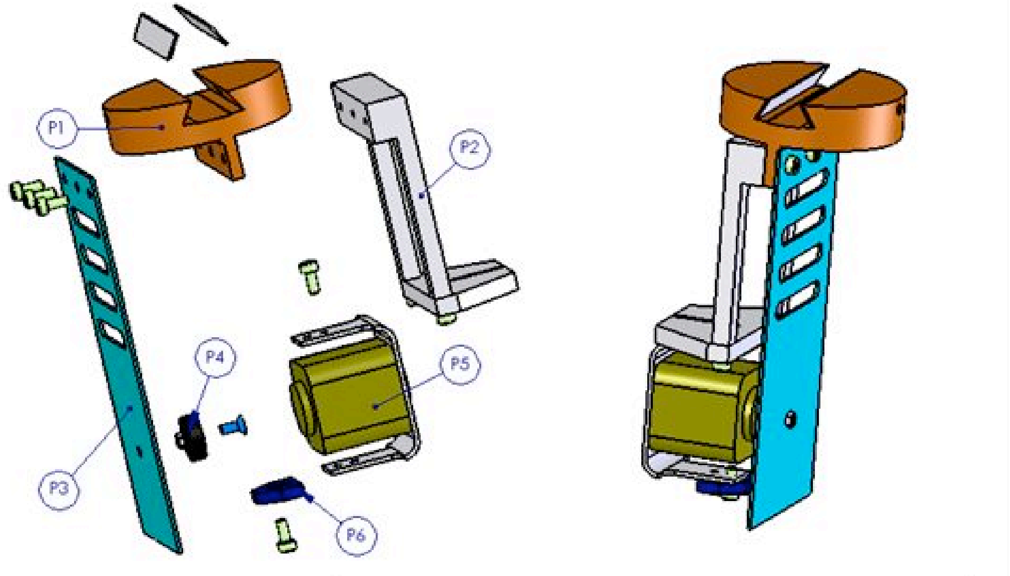
From preliminary tests we have observed that the useful regulating range is ±3Hz off resonance. In working conditions it is preferable to operate below resonance where the plate moves in phase with the magnetic field and the oscillation is stable with time. Working above the resonance resulted in a slowly modulated oscillation amplitude at a typical frequency of a few Hz.

The plate oscillation is detected by an optical coupler consisting in a Light Emitting Diode (LED) plus a Phototransistor. The light emitted by the LED is reflected by the plate and detected by the phototransistor. Depending on the position of the plate a variable level of light will shine the phototransistor. This signal is successively amplified in the driver circuit and is used as oscillation amplitude monitor: in this way the user can easily detect oscillation failure (due namely to a change of the resonanfce frequency due to temperature) and set the system back to the optimal conditions.

The signal from the optical coupler is analyzed by a pair of Schmitt triggers that detect the "too low' or "too wide" amplitude oscillations and set an alarm by means of two LEDs.

V. Conclusion

The Vibrating Sample Holder has been widely tested and with different plates we always obtained stable oscillating conditions. The method of using s vibrating sample has been found to be highly effective in minimizing the effects of Bragg peaks on the XAS spectra collected in fluorescence mode. Fig 3 shows the XAS spectrum of a sample of 10^{16} Sn atoms/cm^2 implanted in SiO_2 measured at the Sn-K edge; when the sample is fixed spurious peaks are evident in the spectrum while they disappear in the spectrum collected with the vibrating sample.



Qty	Description	Part N.	Drawing Number	Material
1	Metallic joint of anchorange	1	03-02-02-01-01	Cu ETP
1	Support for electromagnet end optical sensor	2	03-02-02-01-02	Al/7075
1	Elastic metallic plate	3	03-02-02-01-03	Cu Be
1	Permanent magnet	4		
1	Electromagnet	5		
1	Optical sensor	6		

Fig. 1: Overview of the mechanic parts.

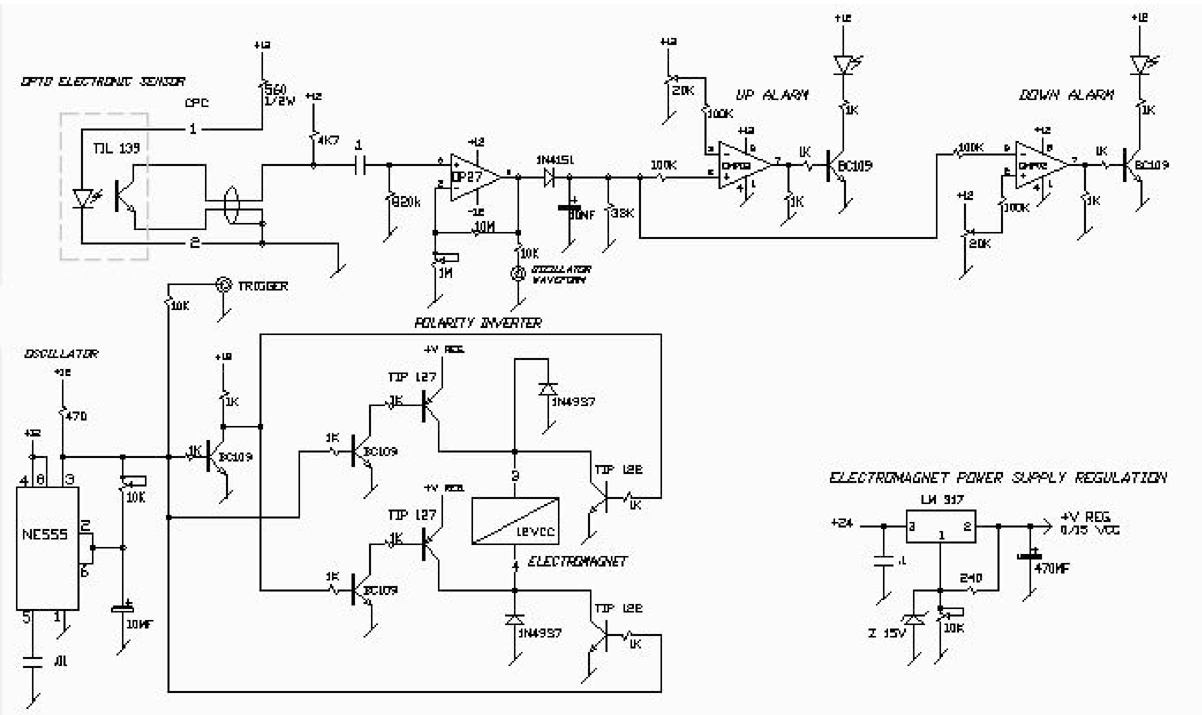


Fig. 2: Electrical circuit of the driver module.

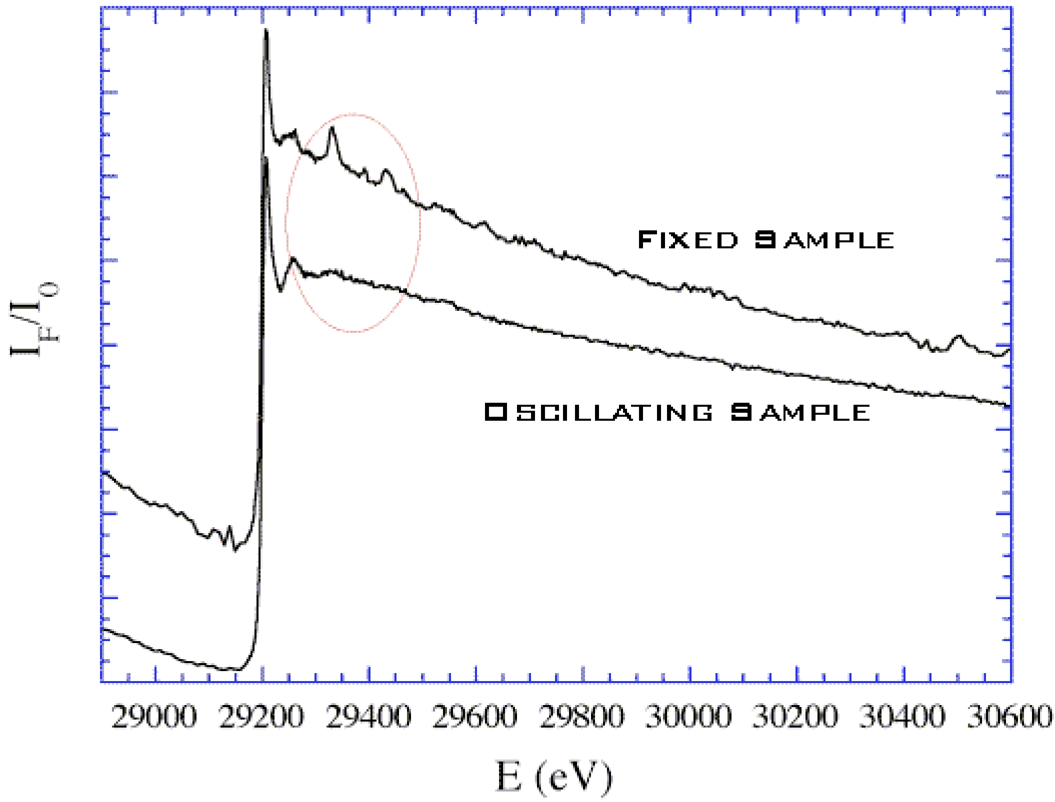


Fig. 3: Absorption spectrum collected on a sample of 10^{16} Sn atoms/cm^2 implanted in SiO_2 measured with static and vibrating sample.