

# Microdiffraction FOX2D CU 25\_25P



Application Note n° AN-2D12

## A b s t r a c t

A microdiffraction instrument has been developed at the Laboratoire de Physique des Solides (LPS), Université Paris-Sud for combined micro-XRD and micro-XRF analysis with very high spatial resolution. The instrument is combining a fine point focus rotating anode generator and a FOX2D 25\_25P optic with small dimension pinhole apertures placed at the tip of the collimator. [1]

We report micro-XRD analysis of rubber material filled with exfoliated clay under strain application. It was possible to confirm the orientational correlation between clay and rubber crystallites at a local state.

[1] Rouzière et al. *J. Appl. Cryst.* (2010). **43**, 1131-1133

## Microdiffraction on elastomer filled with exfoliated clay

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## I n t r o d u c t i o n

It is well-known that the outstanding mechanical properties of natural rubber are due to its ability to partially crystallize under high strain. The resistance of the material to wear and abrasion is usually further increased by the addition of a filler; common fillers are carbon black or particulate silica. Exfoliated clay is another potential candidate. X-ray diffraction patterns taken with sub-millimetric beams indicate that natural rubber crystallites growing under strain, not only align along the stretching direction but also display some preferential orientation with respect to the platelet surfaces. Micro-diffraction is used to confirm the orientational correlation between clay and crystallites at a local scale.

## E x p e r i m e n t a l s e t - u p

A rotating anode generator with an apparent focus of  $100 \times 100 \mu\text{m}$  is equipped with Xenocs FOX2D CU 25\_25P mirror and operated at 40kV-20mA. The optic is coupled to a collimator with a  $10 \mu\text{m}$  pinhole placed at the tip defining the beam size onto the sample. The minimal spot size was measured to be  $15 \mu\text{m}$  (knife-edge method) within the range of 4 mm from the exit of the pinhole. The flux measured with a calibrated photodiode at the exit of the pinhole is  $1.5 \times 10^7$  ph/s. The detector is a direct-illumination CCD camera.

Mapping is possible with two motorized translation stages that allow to move the sample in the vertical and lateral directions. Additionally, SDD detector can be available in the experimental set-up in order to combine x-ray fluorescence and diffraction measurements.

## S a m p l e

The orientation of crystallites with respect to the platelet surfaces is best revealed when the incident x-ray beam is coaxial with the stretching direction. For this purpose, the sample is maintained in a strained state as depicted below. The remaining pieces of rubber outside the metallic holder are non-stretched and only contribute to the anisotropic or amorphous part of the x-ray diagram; this gives an additional difficulty for the measurement as they also absorb part of the diffracted beam.



Fig. 2 : Experimental set-up : X-ray beam is coming from the right on the picture. The CCD camera is on the left. Collimator of the SDD detector is seen on the first plan.

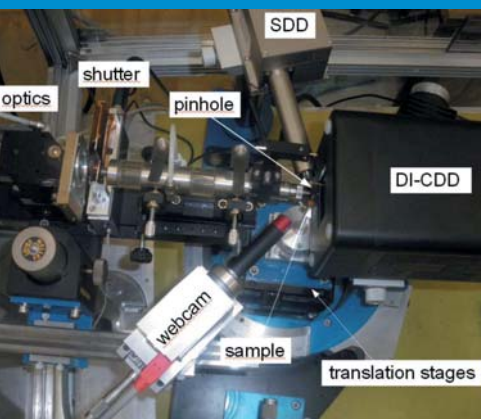


Fig. 1 : Photograph of the micro XRD/ micro XRF instrument at LPS.

## Experimental results

The x-ray beam diameter is 15 $\mu$ m and the sample is moved vertically with a 20 $\mu$ m-step. Some of the diffraction diagrams obtained are displayed in Figure 4 (5 minutes exposures). The patterns show the diffraction signal by the partially exfoliated clay (green arrow) and the diffraction ring at wider angle (red arrow) associated to the (200) reflection of natural rubber crystallites. It clearly appears that the two diffraction signals are correlated both in orientation and in azimuthal modulation of the intensity. For instance, close to the surface of the sample ( $z=1.50$  mm) where clay is most oriented due to surface effect, the (200) ring is well-modulated in intensity whereas it is continuous at  $z=1.90$  mm where clay platelets do not display any preferential orientation.

## Conclusion

A new micro-XRF/micro-XRD instrument was built at the LPS Orsay that enables to capture x-ray diffraction and x-ray fluorescence information with an X-ray microbeam diameter as small as 15  $\mu$ m by using a FOX 2D 25\_25 optic coupled to a fine focus source. The instrument has been used to study the dispersion of clay in natural rubber under strain application. The good spatial resolution and intensity level achieved with the instrument enables to identify the continuous evolution of the clay nanostructure within the sample depth.

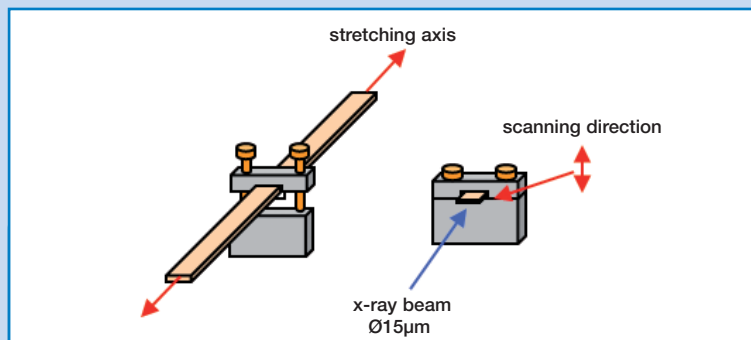


Fig. 3 : Rubber sample in the stretching cell

Fig. 4 : Diffraction patterns of elastomer filled with exfoliated clay (5 minutes exposures). The position ( $z$  in mm) of the sample is indicated.

