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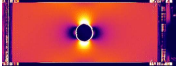
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# EFFECT OF STRESS RELAXATION ON MECHANICALLY INDUCED CRYSTALLIZATION IN NATURAL RUBBER

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## Context & Objectives

Mechanically induced crystallization (MIC) is a widely studied phenomenon. Its characteristics are often determined by wide-angle X-ray diffraction (WAXD) at synchrotrons, ESRF, SOLEIL, DESY...

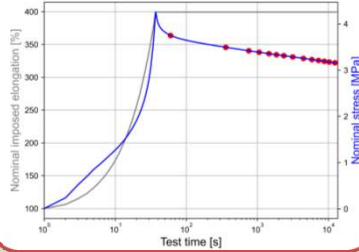


$$\Delta\theta, \underline{\sigma}, \underline{\varepsilon}, \dots? \leftrightarrow \underline{\chi}_c$$

The main objective is to be able to measure crystallization by a less consequential way : infrared thermography. It is necessary to generate data and better understand MIC in order to realize an infrared-crystallization conversion model.

## Performed Tests

WAXD measures during stress relaxation for different temperatures and elongations :

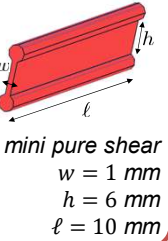


## Studied Materials

Formulation :

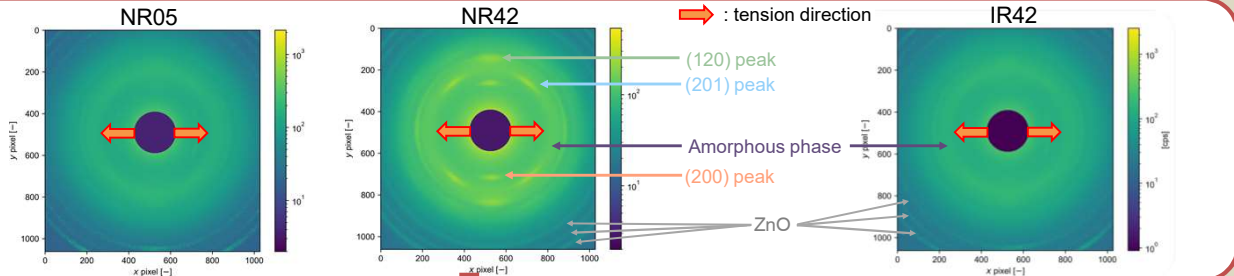
	NR05	NR42	IR42
Matrix	<chem>[*]C=CC=C[*]</chem> cis-polyisoprene	<chem>[*]C=CC=C[*]</chem> cis-polyisoprene	<chem>[*]C=CC(=C)[*]</chem> trans-polyisoprene
CB	5 phr	42 phr	42 phr
Other	processing agent, accelerator activator, vulcanization agent, antioxidant ...		

Samples :



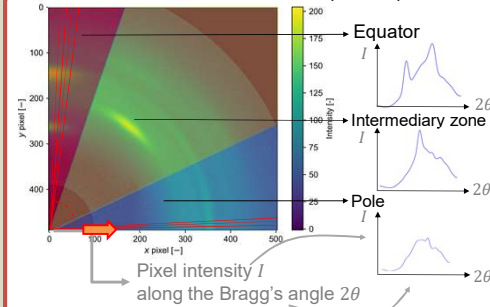
## 0 Raw images

- Wavelength :  $\lambda = 1,54 \text{ \AA}$
- Exposure time : 240s
- Tests performed at INSA Lyon
- Significant MIC for filled natural rubber
- Identification of  $(d_{hkl})$  peaks and all different components from literature [1], [2].

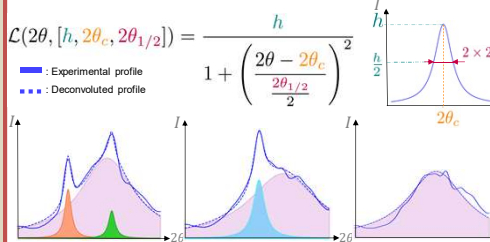


## 2 Bragg's profiles & deconvolutions

Definition of 3 sectors & addition of profiles per sector :



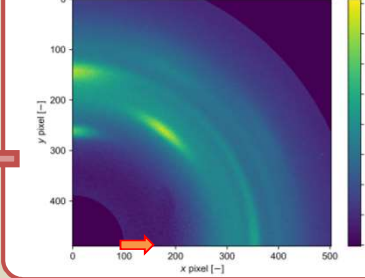
Deconvolution of profiles by Lorentzian functions [1] :



→ crystalline / amorphous split : crystallites dimensional parameters

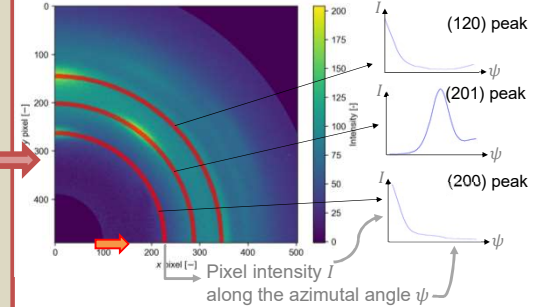
## 1 Data reduction

Use of double symmetry [3] & averaging



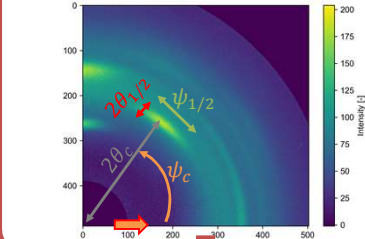
## 3 Azimutal profiles & deconvolutions

Definition of 3 azimuthal profiles :

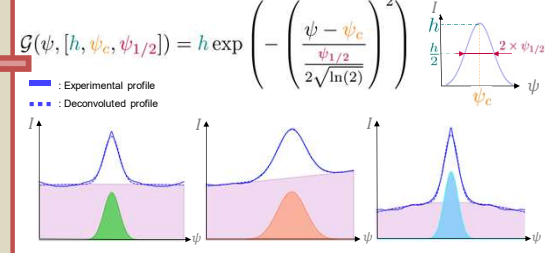


## 4 Obtainable information

- Bragg's angle  $2\theta_c \rightarrow$  Lattice parameter
- Peak's width  $2\theta_{1/2} \rightarrow$  Crystallites' size
- Peak's intensity  $I \rightarrow$  Index of crystallinity
- Azimuthal angle  $\psi_c \rightarrow$  Crystallite's mean orientation
- Peak's length  $\psi_{1/2} \rightarrow$  Crystallite's orientation dispersion

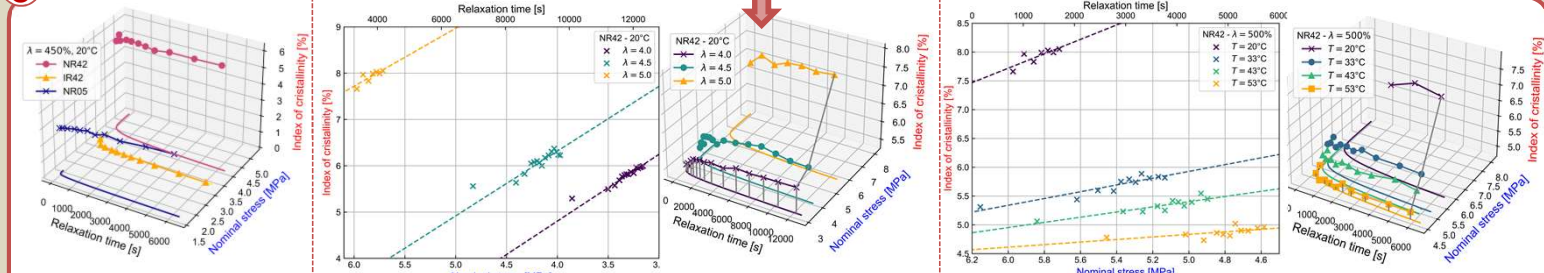


Deconvolution of profiles by Gaussian functions [1] :



→ crystalline / amorphous split : crystallites orientation data

## 5 Results



As expected, only NR-matrix materials crystallize. Fillers permit to increase the MIC phenomenon.

During stress relaxation, the index of crystallinity  $\chi$  varies linearly with stress  $\sigma$ . The  $\frac{\partial \chi}{\partial \sigma}$  coefficient remains the same whatever the value of the imposed elongation.

During stress relaxation, temperature has two effects : it leads to lower indexes of crystallinity  $\chi$  and it reduces the  $\frac{\partial \chi}{\partial \sigma}$  coefficient (i.e. MIC kinetics).

- [1] N. Candau, « Compréhension des mécanismes de cristallisation sous tension des élastomères en conditions quasi-statiques et dynamiques », Thesis, INSA Lyon, 2014
- [2] P.-A. Albouy, J. Marchal, et J. Rault, « Chain orientation in natural rubber, Part I: The inverse yielding effect », *Eur. Phys. J. E*, vol. 17, n° 3, p. 247-259, 2005.
- [3] S. Beurrot, « Cristallisation sous contrainte du caoutchouc naturel en fatigue et sous sollicitation multiaxiale », Thesis, Ecole Centrale de Nantes, 2012

## References

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