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X-Ray Diffraction Use for PZN-PT Single Crystal's Synthesis and Microscopic Thermal Dilatation Coefficient Determination

Diouma KOBOR[1], Laurent LEBRUN[2] and Daniel GUYOMAR[2]

1: ICPM, Université Assane Seck de Ziguinchor, BP 523, Ziguinchor, Sénégal, dkobor@univ-zig.sn

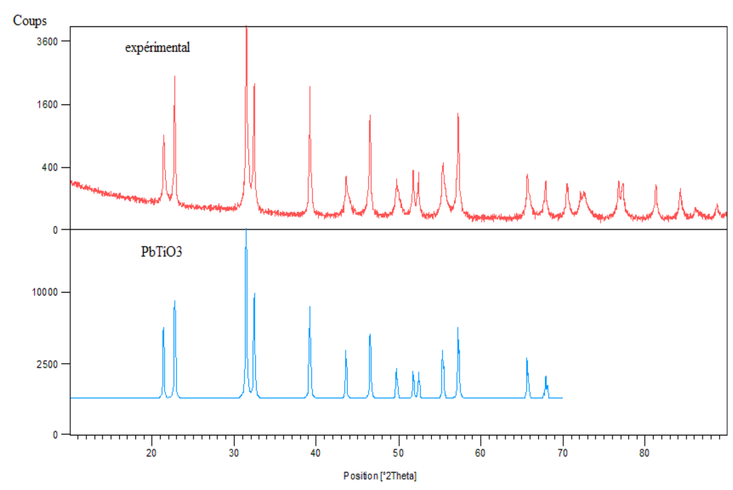
2: LGEF, INSA de Lyon, Batiment Gustave Ferrié, 8 rue de la Physique, 69621 Villeurbanne Cedex, France, laurent.lebrun@insa-lyon.fr

Introduction:

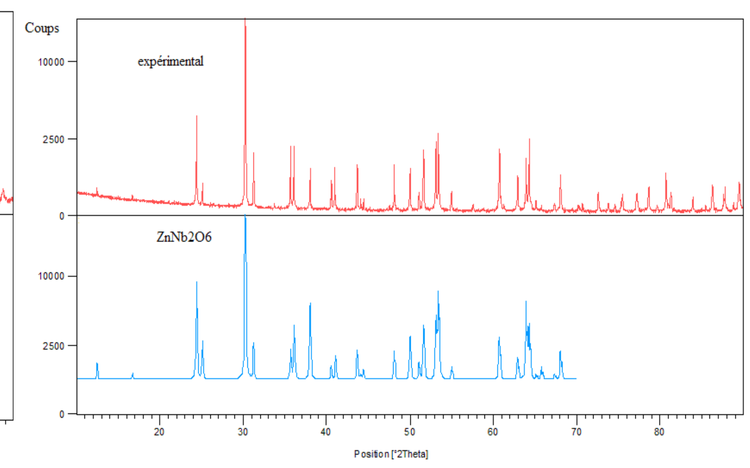
The performance of transducers can be significantly increased by the use of active materials with large piezoelectric coefficients (d_{ij}) and large coupling factors (k_{ij}) [1]. Recently, high levels of piezoelectricity have been published in $Pb(Mg_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ (PMN-PT) and $Pb(Zn_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ (PZN-PT) single crystals: d_{33} > 2500 pC/N, d_{31}> -1500pC/N, k_{33}> 90%, k_{31}> 80% and k_t> 60% [1-5].
X-Ray Diffraction is an excellent technique to follow and analyze structures and crystallographic phases of solid materials.
In this paper we use the X-Ray powder Diffraction technique for controlling the different steps PZN-4.5PT single crystals synthesis and its thermal characterization.

Furnace used for growth of PZN-4.5PT single crystals by Flux method

X-Ray Powder Diffraction of $PbTiO_3$ precursor



X-Ray Powder Diffraction of $ZnNb_2O_6$ precursor



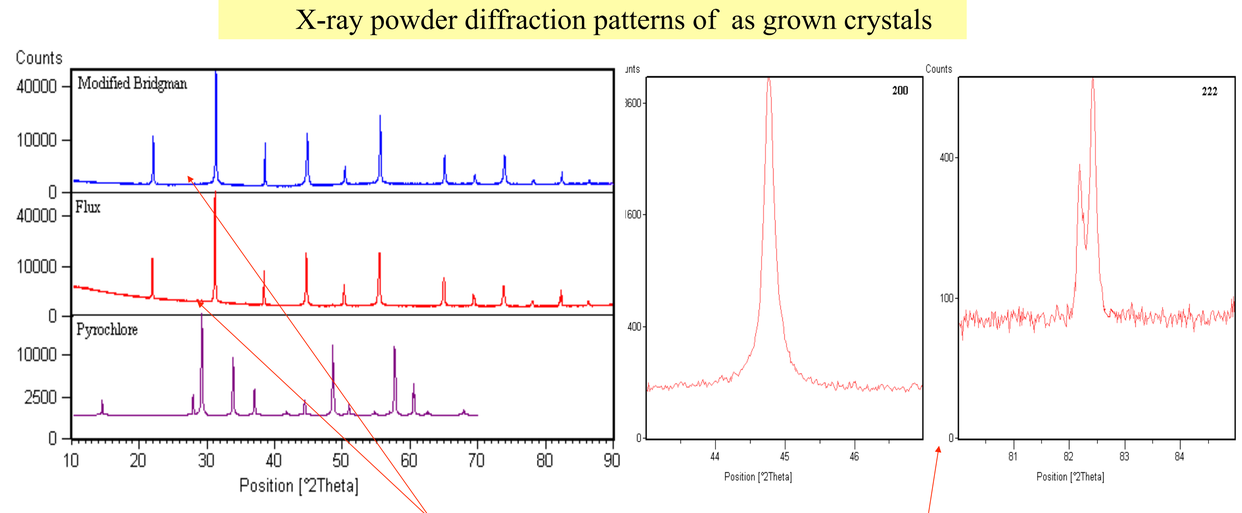
The experimental and the bibliographical spectra present the same peaks at the same angles

Typical PZN-4.5PT single crystals

By Flux method

By Modified Bridgman method

X-ray powder diffraction patterns of as grown crystals



no pyrochlore phase is detected

Rhombohedral symmetry in ambient temperature

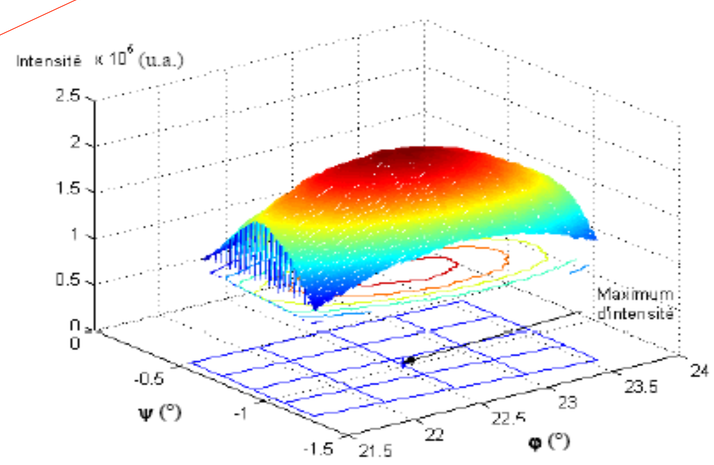
Table 1: Lattice parameters at T ambient and T = 300°C

Parameters	Rhombohedral (ambient temperature)		Cubic (300 °C)	
	modified Bridgman	Flux method	modified Bridgman	Flux method
a = b = c (Å)	4.054(5)	4.054(2)	4.0595(6)	4.0592(9)
α = β = γ (°)	89.9318	89.8694	90	90
V (Å³)	66.6756	66.6376	66.902	66.884

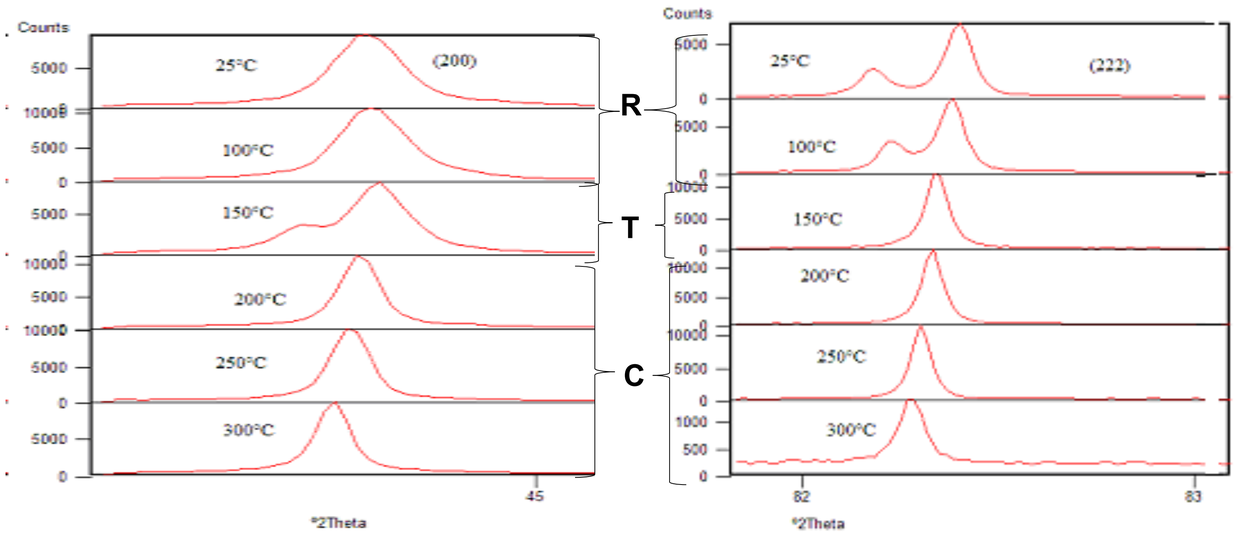
Table 2: XRD theoretical peaks for different crystal system types

Crystal System	Cell parameters	Types of spectra		
		h00	hhh	hh0
Cubic	a=b=c, α=β=γ=90°	h00	hhh	hh0
Tétragonal	a=b≠c, α=β=γ=90°	00h, 0h0	hhh	hh0, h0h
Rhombohedral	a=b=c, α=β=γ≠90°	h00	hhh, -hhh	hh0, -hh0
Orthorhomb. P	a≠b≠c, α=β=γ=90°	00h, h00, 0h0	Hhh	h0h, 0hh, hh0
Orthorhomb. B	a=c≠b, α=β=γ=90°	h0h, 0h0	0h2h, 2hh0	002h, 2h00, hhh
Monoclinic	a≠b≠c, α=γ=90°, β>90°	00h, h00, 0h0	hh-h, hhh	h0-h, h0h, 0hh, hh0

The single crystals orientation using the XRD source of X'Pertpro of Panalytical



An example of figures of pôles obtained on PZN-4.5PT single crystals



XRD diagrams: the bragg peaks 200 and 222 at different temperatures. permit to determine the different phases transitions (rhombohedral to tetragonal (ambient – 150 °C) and from tetragonal to cubic (150 °C- 200 °C) considering table 2) and to calculate the thermal dilatation coefficient at microscopic scale

The calcul of the thermal dilatation coefficient by XRD (microscopic) and TMA (macroscopic): the values are sligt the same for the two methods

Samples/α_a (10^{-6} °C^{-1})	XRD method	TMA method
PZN-4.5PT	5	-
PZN-4.5PT+1%Mn	8	9.4
PZN-4.5PT+2%Mn	10	9.8

Conclusion :

PZN-4.5PT single crystals have been grown by the flux method. The use of X-Ray powder diffraction permitted us to determine the different changes of structure at different temperatures and for the crystal orientation.

The main innovation is the use of this technique to calculate the thermal dilatation coefficient by the lattice parameters variation versus temperature and the comparison of the microscopic and macroscopic values.

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