samples with zirconium the texture components are sharper.
The above findings were more or less confirmed by the EBSD analysis. The analysis of the deformed and recrystallized parts indicate the changes from deformed part after one pass to about 70% recrystallized fraction after four and eight passes.

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MS46 P06

Structural and ferroelectric characteristics in the $Ba(Ti_{1-x}Zr_x)Li_{xy}O_{3(1-xy)}F_{3xy}$ solid solution K. Taïbi[a], A. Kerfah[a], A. Guehria-Laidoudi[a], A. Simon[b] and J. Ravez[b] [a]*Faculté de Chimie, U.S.T.H.B., BP 32 El-Alia, Bab-Ezzouar, Alger, Algerie.* [b]*I.C.M.C.B.-C.N.R.S., 87 avenue D' A. Schweitzer, 33608 Pessac, France.*
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Keywords: lead-free, ferroelectric, transition

Relaxor are a special class of ferroelectrics with very interesting properties. Relaxor materials actually used are lead-based ceramics which present a disadvantage due to the toxicity of PbO. The actual evolution of research is oriented to environment-friendly application. In this way, we have previously investigated new lead free compositions. Dielectric studies performed on $Ba(Ti_{1-x}Zr_x)Li_{xy}O_{3(1-xy)}F_{3xy}$ solid solutions showed three kinds of behaviour. For compositions very close to $BaTiO_3$ the three phase transition were retained as was the case in the classical ferroelectric $BaTiO_3$. For compositions $Ba(Ti_{1-x}Li_x)O_{3(1-x)}F_{3x}$ and $Ba(Ti_{1-x}Zr_x)O_3$, it appears one broad peak with frequency dispersion characteristic of relaxor behaviour. For compositions close to $Ba(Ti_{1-x}Zr_x)O_3$ only one diffuse phase transition, without frequency dispersion was evidenced. In this latest domain, it is possible to observe the spontaneous phase transition from ferroelectric to relaxor state just by thermal change.
To understand the origin of this behaviour much works based on structural and physical models are performed. It has been found that the frequency and the temperature T_m can be described by using the Vogel-Fulcher relationship [3, 4]. In the present work we fitted all the dielectric data to this Vogel-Fulcher equation and correlated the results with structural data obtained by powder diffraction study. The fitting parameters and the upper limit of the composition for ceramics with relaxor state were defined. The plot of Tm(f)/Tm (1 kHz) versus the logarithm of the frequency allows to determine the limit of composition between the ferroelectric and the relaxor state which is close to 0.155.
Powder XRD patterns indicates a particular behaviour for $y = 0.25$ where two kinds of single domain phases are observed: tetragonal, for $0 \leq x < 0.15$ and cubic for $0.15 \leq x \leq 0.25$. The latest domain corresponds to the relaxor phase. The change from tetragonal to cubic symmetry related to the disappearance of the (200) and the appearance of the (111) reflexions, characteristic of the cubic perovskite structure. The relaxor to normal ferroelectric transformation and the structural phase transition are strongly related to the domain morphology evolution. The presence of a local disorder related to nanoscale heterogeneities is responsible of the relaxor behaviour when the composition deviates from the well known $BaTiO_3$ [3, 4].

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MS46 P07

Structural studies of crystallization and growth of magnetron deposited TiO_2 thin films by X-ray diffraction and reflectivity Kužel Radomir, Zdeněk Matěj[a]., Jan Šícha[b], Jindřich Musil[b], [a]*Department of Condensed Matter Physics, Faculty of Mathematics and Physics, Charles University in Prague, Ke Karlovu 5, 121 16 Praha 2*, Czech Republic, [b]*Department of Physics, Faculty of Applied Sciences, University of West Bohemia in Pilsen, Czech Republic*. E-mail : nichtova@gmail.com

Keywords: thin films; TiO_2, anatase; dc magnetron sputtering; microstructure

TiO_2 films are nowadays widely used because of their interesting photocatalytic and self cleaning properties. Complex X-ray scattering studies were performed on sets of titanium dioxide thin films sputtered by dual dc magnetron [1]. Three sets of nanocrystalline and amorphous TiO_2 thin films magnetron deposited on glass and silicon substrates have been studied. Phase analysis and X-ray line broadening were studied by X-ray powder diffraction in parallel beam optics; the residual stresses were measured with the aid of the Eulerian cradle and surface roughness determined by X-ray reflectivity measurement. Microstructure parameters were extracted from XRD measurements by individual peak profile fitting and also by whole powder pattern modelling [2] approach (MAUD [3], modified FOX[4]).
By both thickness dependence of XRD patterns of nanocrystalline films and depth profiling measurements it was found that rutile phase growths on the substrate and it is transformed to anatase with increasing distance from the substrate. This may be caused by temperature gradient during deposition. Another set of amorphous films with different thickness was studied after annealing and also by in-situ measurements during the heating.
It was found that the crystallization temperature started at about 250 °C for thicker films but it was higher for thinner films (< 200 nm) and reached about 350 °C. Thinner films were single phase (anatase) while thicker films above 1200 nm contained also a smalll amount of nanocrystalline rutile. The crystallite size of these samples immediately after crystallization was larger than 100 nm by contrast to the nanocrystalline films which did not show any significant changes after annealing in this temperature range and their crystallites remain small under about 10 nm. Annealing at temperatures above 500 °C leads to increase of crystallite size and transformation of anatase into rutile.
Simple uniaxial tensile stress and only a weak texture were found for the amorphous films after crystallization. Only for the thinnest films (~ 100 nm), the 101 texture (anatase) was found. In case of nanocrystalline films the stress was low but complicated. This can be related to significantly stronger and more complicated texture due to dual magnetron geometry.