

**MS10.05.09 ORIENTED CRYSTALLINE LAYERS ON THE SURFACE OF BISMUTH SUPERCONDUCTING GLASS CERAMICS.** S. Simon<sup>1</sup>, Gh. Borodi<sup>2</sup>, T. Farcas<sup>1</sup>, <sup>1</sup>Babes-Bolyai University, Faculty of Physics, 3400 ClujNapoca, Romania, <sup>2</sup>Institute of Isotope and Molecular Technologies, 3400 Cluj-Napoca, Romania

X-ray diffraction, scanning electron microscopy (SEM) and electron paramagnetic resonance (EPR) results on undoped and Gd-doped  $\text{Bi}_{1.8}\text{Pb}_{0.2}\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_z$  and  $\text{Bi}_{1.8}\text{Pb}_{0.2}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_z$  systems are presented. The samples were prepared by quenching of melted oxides mixture corresponding to the desired compositions on different metallic supports like steel, copper and silver.

The glass-ceramic route is a very attractive preparation technique for the fabrication of dense superconductors with desired shapes like unsupported and metal-supported fibres. For a large superconducting current flow it is very important to have the crystalline grains on the surface parallel oriented to each other and with Cu-O planes parallel to the surface. The type and concentration of initial crystalline grains that appear at the metal-glass interfaces and their orientation are function on metallic support temperature, its nature and temperature gradient between support and molten. In order to improve the crystallinity and to have a preferentially oriented superconducting microcrystals with *c* axis perpendicular to the surface, we followed different heat treatment procedures in which we changed the heating rate, the final heat treatment temperature and its duration and the atmosphere nature: air or oxygen. After heat treatments spectacular changes of preferential orientation and grains shape took place as were evidenced by X-ray diffraction and SEM. We found that the presence of gadolinium in the samples in which calcium was partially substituted by this affect not only the type of final crystalline phases [1] but also the stability of their precursor glasses. The crystalline data obtained by X-ray diffraction and SEM on these samples were correlated with EPR data.

1. S. Simon, E. Burzo, O. Cozar, I. Barbur, V. Simon, I. Ardelean, V. Pop, G. Borodi, *Physica C*, 185 - 189 (1991) 899

**MS10.05.10 THE PREPARATION OF CRYSTALLINE  $\text{NbSe}_2/\text{TiSe}_2$  SUPERLATTICES FROM MODULATED ELEMENTAL REACTANTS.** David C. Johnson and Myungkeun Noh, Materials Science Institute and Department of Chemistry, University of Oregon, Eugene, Oregon 97403.

A series of crystalline superlattice compounds containing an integral number of inter grown transition metal dichalcogenide layers were prepared through controlled crystallization of Ti/Se/Nb/Se superlattice reactants with designed compositional modulation. The component elemental layer thicknesses and annealing sequence were chosen to favor interfacial nucleation of the component binary compounds. Theta-theta and rocking curve data were collected to study of the evolution of the initially layered reactants into the crystalline superlattices as a function of temperature. The initial layered reactant was found to contract in the *c*-axis direction upon initial annealing and the interfaces appear to become smoother during this initial interdiffusion. The growth of *c*-axis oriented  $\text{NbSe}_2/\text{TiSe}_2$  crystal structure perpendicular to the substrate surface occurs upon annealing at temperature above 200°C. The gradual decrease of the (00*l*) diffraction linewidths of the growing compound as a function of annealing time and temperature indicating increases in the *c*-axis domain size. High quality *c*-axis oriented  $\text{TiSe}_2/\text{NbSe}_2$  crystalline superlattices result from extended annealing at the relatively low annealing temperature of 500°C. The large number of observed 00*l* diffraction orders permits the crystal structure of the superlattice in the *c*-axis direction to be determined through a Reitveld analysis. The rational synthesis of intergrowth compounds from superlattice reactants as described herein will permit the tailoring of physical properties as a function of compositional layer thicknesses and native properties of the parent compounds. This will permit the exploration of the transition from composite behavior to that of a new compound as the lengthscale of the compositional modulation decreases.

**PS10.05.11 STRUCTURAL ANALYSIS OF THE THIN FILMS OF MERCURY CUPRATES USING IP-WEISSENBERG METHOD.** K. Nakanishi, Y. Moriwaki, T. Sugano, S. Adachi, A. Tsukamoto, K. Tanabe, Superconductivity Research Laboratory, ISTE, 10-13 Shinonome 1-chome, Koto-ku, Tokyo 135, Japan

The thin-film synthesis as well as the high-pressure synthesis technique is promising for creating new oxide superconductor materials. Recently, the "layer-by-layer" growth has been realized using MBE (Molecular Beam Epitaxy), PLE (Pulsed Laser Epitaxy) and so on. We have grown by PLE using ArF excimer laser (193nm) and controlled by in-situ monitoring using image-processing Reflection High Energy Electron Diffraction (RHEED) and coaxial impact collision ion scattering spectroscopy (CAICISS).

However, the exact crystal structures of the obtained thin films are not clear, because of difficulties in separating the peaks of the thin film from those of the substrate in the lattice-matched systems.

We have applied an X-ray Weissenberg camera equipped with an imaging plate (IP-Weissenberg Camera) to the thin films and succeeded in the separation and the observation of all the reciprocal space including weak spots such as Laue oscillation, diffuse streak and satellite with good accuracy. Furthermore, this method has an advantage that the corrections of volume, absorption and extinction are relatively simple, because a sample is rotated about an epitaxial axis keeping same incident angle (i.e. same irradiated volume). This also makes the blind region smaller than that of the bi-secting configuration. This has enabled to analyse a new superstructure of a  $\text{BaCuO}_2$  and a new pseudo-cubic phase of a  $\text{LnBa}_2\text{Cu}_3\text{O}_7$  (Ln=Nd,Pr).

This study is the first structural analysis of the thin films of Mercury cuprates. We observed  $\text{HgBa}_2\text{CaCu}_2\text{O}_y$  and  $\text{HgBa}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  thin films grown on  $\text{SrTiO}_3$  and  $\text{LaAlO}_3$  substrates. As a result, not only *c*-axis (epitaxial axis) but also *a*-axis (in-plane axis) of the thin films was consistent with that of these substrates, although the lattice constants ( $a = 3.85 \sim 3.86 \text{ \AA}$ ) was different, indicating that this epitaxy was "free standing mode". We will discuss the relationship between superconducting properties and crystal structure of thin films.

**PS10.05.12 NEUTRON HIGH RESOLUTION SINGLE CRYSTAL DIFFRACTION STUDY OF PHASE SEPARATION PHENOMENON IN  $\text{La}_2\text{CuO}_{4+\delta}$**  A.M. Balagurov<sup>1</sup>, V.G. Simkin<sup>1</sup>, V. Yu. Pomyakushin<sup>1</sup>, A.A. Zacharov<sup>2</sup> 1) Joint Institute for Nuclear Research, Dubna, Russia, 2) Russian Scientific Center "Kurchatov Institute" Moscow, Russia

Two  $\text{La}_2\text{CuO}_{4+\delta}$  single crystals with  $\delta=0.03$  and 0.04 were studied in the temperature range of  $10 \text{ K} \leq T \leq 293$  using the neutron high resolution Fourier diffractometer in Dubna. Diffraction patterns were measured for (00*l*) and (*h*00) / (0*k*0) directions in reciprocal space. At room temperature, the Bmab phase (*h*00) / (0*k*0) reflections were split due to twinning. In the crystal with  $\delta=0.04$ , the phase separation in the phases with Fmmm and Bmab symmetry was found, after cooling, as an additional splitting of the (0*k*0) peaks. In the crystal with  $\delta=0.03$ , the uniform state was preserved down to 10 K. The analysis of diffraction peak widths as a function of *d*-spacing offers the possibility of finding the dimensions of the coherent domains of coexisting phases in the  $\delta=0.04$  crystal.

In the figure, the relative peak width vs.  $d_{hkl}$  is shown for the (00*l*) and (0*k*0) directions. The slope of the lines is connected with domain sizes which are close to 1000 Å in the (00*l*) and 1500 Å in the (0*k*0) directions, respectively.

