I5 14:30 – 15:10

TEM Investigations of $CaTiO_3$ - $NdAlO_3$ Ceramics

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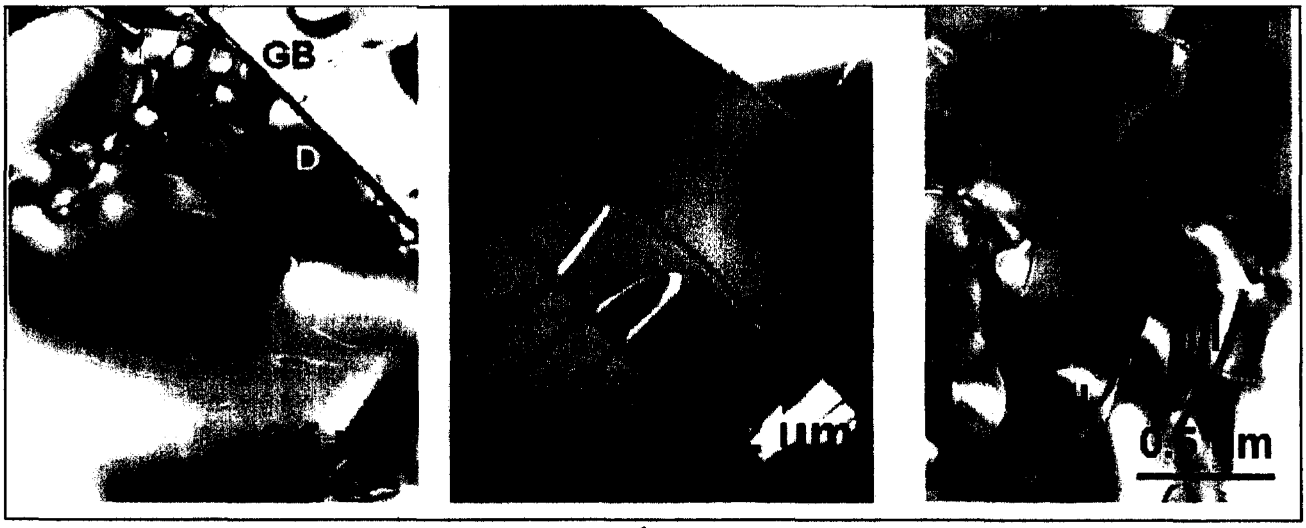
Microwave dielectric ceramics for wireless and global communications must exhibit low dielectric losses (high Q-values), high relative permittivities (ε'_r) and temperature stable dielectrical properties. There are permanent demands for electronic devices with improved properties. Materials with ε'_r < 35 and Q ~ 50.000 should be replaced with those possessing ε'_r > 45 and Q ~ 45.000. Calcium titanate based solid solutions are very promising candidates for such materials. In the present work preliminary results of a TEM-EDXS investigations of 0.7 $CaTiO_3$ - 0.3 $NdAlO_3$ based ceramics are reported. Samples investigated in this study were prepared from oxides, using common ceramic procedures and fired at 1450 °C for 10h.

Using energy dispersive X-ray spectroscopy (EDXS) it was found that just one phase was present in the samples. In Fig. 1a a TEM mircograph of an area near the grain boundary (GB) in $CaTiO_3$ - $NdAlO_3$ sample is displayed. Grains, few µm in size, are composed of many smaller, irregularly shaped domains (D). Beside domains planar, loops forming features were observed inside the grains. Due to characteristic shape and based on bright field - dark field experiments and the contrast and symmetry of the fringes we concluded that this features are antiphase boundaries (APB). In Fig. 1b (dark field micrograph) a part of a grain with two types of planar defects, namely domain boundaries and APBs, is presented. It is obvious that antiphase boundaries are passing unimpeded through the domain boundaries. The change in contrast of APB when passing through adjacent domains is clearly visible. In Fig. 1c a region with domains inside a grain is shown. From this region an electron diffraction pattern was recorded (Fig. 2a). Using smaller aperture size SAED patterns from three different domains, labeled as I, II and III (Fig. 1c) were obtained. Those three diffraction patterns are schematically drawn on Fig. 2b.

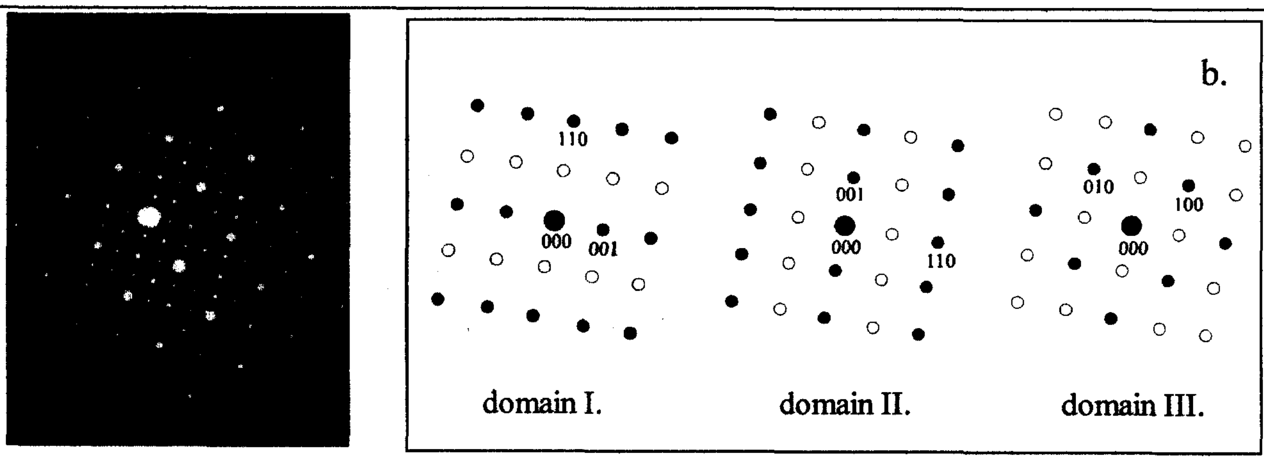
According to analogy from Pb doped $CaTiO_3$ [1,2] where different types of ordering are present (chemical ordering and ordering due to tilting of the octahedra [3]) SAED pattern shown in Fig. 2a. can be indexed as a cubic perovskite (a = 0.38 nm) in [001] zone with 1/2{100}, 1/2{110} and 1/2{111} superstructure reflections. On the other hand diffraction pattern in Fig. 2a. is a sum of three patterns from three different adjacent domains (Fig. 2b). Each of these patterns can be index as a tetragonal phase with a = 0.54 nm and c = 0.76 nm. Such tetragonal phase was reported in $(Pb,Ca)TiO_3$ [4] and its appearance was explained by the chemical ordering of A site atoms.

In the on-going work, using in-situ heating experiments and correlating the chemical composition, firing temperature and cooling regime with the appearance of antiphase boundaries, domains and superstructure reflections, we expect to identify the origins of the features introduced in this work.



a. b. c.

Fig 1. TEM mircographs of $CaTiO_3$ - $NdAlO_3$ sample. a.- an area near the grain boundary (GB) with antiphase boundaries (APB) and domains (D) present, b. - part of a grain with two types of planar defects (dark field image), c. - domains inside a grain with labeled areas (I, II, III) where SAED patterns shown in Fig. 2. were recorded.



Cubic, z = [001] Tetr. z = $[\bar{1}10]$ Tetr. z = $[\bar{1}10]$ Tetr. z = [001]

a.

Fig 2. Selected area electron diffraction (SAED) patterns from a region shown in Fig. 1c. a. - pattern from a whole region where all three domains (I., II. and III.) were included, indexed as a cubic perovskite with superstructure reflections. b. - schematic drawing of the diffraction patterns from three different (I, II and III) domains indexed as a tetragonal phase. (Open circles in Fig.2b represent missing reflections.)

References

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