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# Mechanical loss associated with the grain size in lead zirconate titanate ceramics

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**Abstract.** The mechanical loss ( $Q^{-1}$ ) and Young's modulus ( $E$ ) were measured as a function of temperature in the range  $293\text{K} < T < 750\text{K}$  on rectangular bar samples of ferroelectric  $\text{PbZr}_{1-x}\text{Ti}_x\text{O}_3$  ceramics. There are two mechanical loss peaks, which are located below the phase transition point in coarse grained samples and they seem to be associated with the motion of domain walls. One of these peaks are not observed in fine grained undoped ceramics. The use of dopants such as Ge, Bi, Nb produces an attenuation of mechanical loss, a smoothing of elastic modulus anomalies, and a shifting of the phase transition temperatures.

## 1. INTRODUCTION

The lead zirconate titanate ceramics is one of the most attractive materials for various sensors and actuators, because of their excellent dielectric, piezoelectric, mechanical and optical parameters. The properties of PZT and another ferroelectric materials largely depend on the domain structure, oxygen vacancy and the interaction among various lattice defects. However, the mechanism that the oxygen vacancies and domain walls play a decisive role in the applications of ferroelectrics, is still not very clear up to now.

It is well known that the internal friction method is very sensitive to the microstructure of materials and thanks to it's analytical possibilities [1-5] can give true information about acoustic loss mechanisms and reconstruction of domain structure, domain boundary kinetics as well as permits to estimate different microstructure parameters.

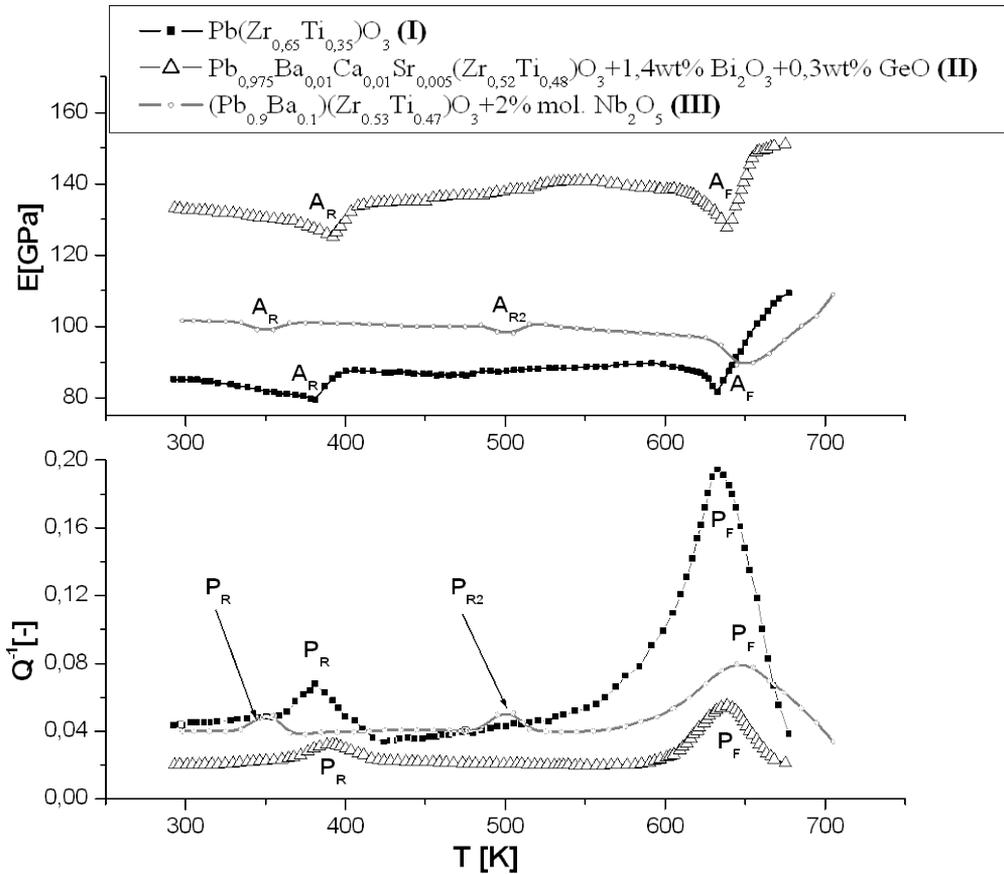
## 2. EXPERIMENTAL PROCEDURES

In our experiments three kinds of samples were prepared:  $\text{Pb}(\text{Zr}_{0.65}\text{Ti}_{0.35})\text{O}_3$  (I) by the sol-gel method,  $\text{Pb}_{0.975}\text{Ba}_{0.01}\text{Ca}_{0.01}\text{Sr}_{0.005}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3 + 1,4 \text{ wt\% Bi}_2\text{O}_3 + 0,3 \text{ wt\% GeO}$  (II) by using the hot pressing method,  $(\text{Pb}_{0.9}\text{Ba}_{0.1})(\text{Zr}_{0.53}\text{Ti}_{0.47})\text{O}_3 + 2\% \text{ mol. Nb}_2\text{O}_5$  (III) by conventional ceramic sintering method (CCS). The average grain size for obtained samples is the following: about  $3.0 \mu\text{m}$  for composition (I),  $2.0 \mu\text{m}$  for (II) and about  $8.0 \mu\text{m}$  for composition (III). The samples were sliced into bars of  $30 \times 10 \times 0.9 \text{ mm}^3$  for mechanical loss and Young's modulus measurements.

The grain size of the ceramics was observed using a scanning electron microscopy (SEM). The mechanical loss  $Q^{-1}$  and Young's modulus  $E$  measurements were performed on a computer-controlled resonance mechanical spectrometer of the RAK- 3 type from about 293 K to 750 K during the heating process. The heating rate was about 3 K/min.

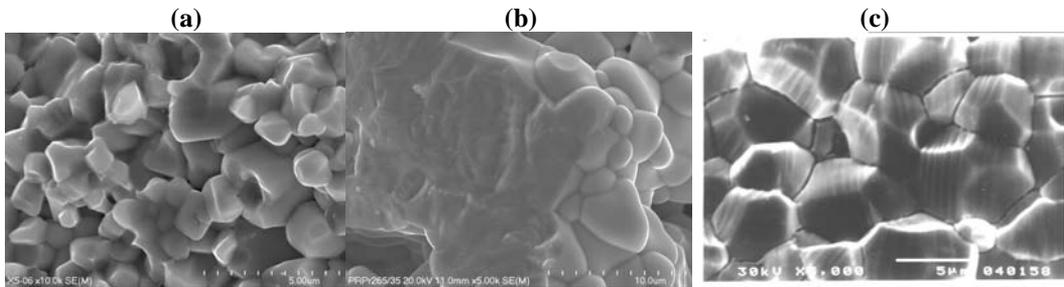
### 3. RESULTS AND DISCUSSIONS

**Fig. 1** shows the variations in the Young's modulus  $E$  and the mechanical loss  $Q^{-1}$  as a function of temperature  $T$  of investigated ceramic samples (I), (II), (III). For the ceramic samples with the chemical composition (I) and (II) we can observe on the  $E(T)$  curves two very sharp anomalies  $A_R$  (385 K for (I), 390 K for (II)),  $A_F$  (635 K for (I), 638 K for (II)), which correspond to the anomalies associated with two peaks  $P_R$  and  $P_F$  on the  $Q^{-1}(T)$  curve. For the chemical composition (III) on the fig. 1 we can observe additional anomaly  $A_{R2}$  (502 K) on the  $E(T)$  curve and  $P_{R2}$  peak on the  $Q^{-1}(T)$  dependence. This peak is observed only in the coarse grained ceramics obtained by CCS method. The structure observed by SEM shows (**Fig. 2c**) large grains (about 8.0  $\mu\text{m}$  in size).



**Figure 1.** Temperature dependencies of Young's modulus  $E(T)$  and mechanical loss  $Q^{-1}(T)$  for investigated ceramics with the chemical composition (I), (II), (III).

**Fig. 2a** and **2b** show a more homogeneous structure of fine grain size (about 3.0  $\mu\text{m}$  for composition (I) and 2.0  $\mu\text{m}$  for composition (II)). Therefore, the  $P_{R2}$  peak may be attributed to the motion of domain walls in the coarse grained ceramics and such a motion could be limited by grain boundaries in fine grained ceramics. This is consistent with the results reported by Cheng et al [6], where the internal friction peaks related to the motion of domain walls are only observed in ceramics with large grains.



**Figure 2.** An SEM images of (I), (II), (III) investigated ceramic samples.

According to the PZT phase diagram [7], the  $P_F$  peaks, observed on the **fig. 1** are due to the Curie transition between ferroelectric and paraelectric phases. The  $P_R$  peak's activation energy close to 1,1 eV and the limit relaxation time  $\tau_0 = 10^{-14\pm 1}$  s, could be linked to a point defect relaxation [8, 9].

#### 4. CONCLUSIONS

In coarse grained doped PZT ceramics there are two mechanical loss ( $P_R$  and  $P_{R2}$ ) peaks and  $P_F$  phase transition peak. The  $P_{R2}$  peak can be explained by the interaction of domain walls and the diffusion of oxygen vacancies. In the fine grained size ceramic samples the  $P_{R2}$  peak is not observed. The influence of dopants such as Nb, Bi or Ge results in the attenuation of mechanical loss and smoothing of the Young's modulus anomaly.

#### Acknowledgments

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