APPLICATION OF THE NON DESTRUCTIVE METHODS IN THE INVESTIGATION OF THE FERROELECTRIC CERAMICS

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This work shows the results of researches of the mechanical properties of the piezoelectric ceramics obtained by the hot pressing method. The measurements of the velocity of propagations longitudinal and transversal supersonic waves were carried out by ultrasonic defectoscope UMT-11. All measurement of the piezoelectric parameters such as: resonance and antiresonance frequency, the piezoelectric modulus, the electromechanical coupling coefficient, the tensor component of permittivity, elastic susceptibility were calculated by resonance-antiresonance method.

1. INTRODUCTION

Maintenance of quality has a decisive influence on the possibility of the uses of the ceramic materials. It concern as well as initial material and part of engines and many another devices made from this material. During process of the production become stresses, which accumulation on the edge of the gradient of the density, interpolation, cracks, holes, concentration of the molecules. There is no possibility to reduction this stresses. Especially on the cracks creates large concentration of the stresses. It could take to the critical growth of the crack. Mentioned defects are the reason of the relative large changeableness of the properties and especially big dispersion of the strength. Small under relation of the shape and sort of the defects could brings to the destruction of the elements. The result of that is a fragile ceramic structure. The gradient of the density makes deformation of the elements during condensation and forming heterogeneity of the microstructure. During process of the sintering are making shrinkage cracks in the effect of concentration of the molecules. The internal stress made by final processing helps making cracks. The quality of the manufacture process could be defined by Non Destructive Evaluation method – NDE [1].

In the work, possibilities of the supersonic and resonance and anti-resonance methods of the investigation are shown.

2. SUPERSONIC RESEARCHES

Reflection method sometimes called impulse method of response was used in investigation. This method brings a lot of information about investigated material. The effect of the supersonic impulse reflection was used to opinion quality of the material. The oscillation head make as piezoelectric transducer works as receiver and transmitter too. The received energy is weaker than transmitted. There is no possibility to works with continuous waves and impulse waves are only used. The acoustic waves came into the material till meets the border surface. On the border surface occurs full or partial reflection of this wave. If the reflection surface is perpendicular to the direction of the propagation than the acoustic waves is reflected in the original direction and after transit time meets the oscillator again and has been changed to the electric impulse. Not the whole of the recurring energy has been changed to the electric energy. On the border surface oscillator-surface of the material occur partial reflections. The part of the sound goes second time through the object and etc. In this way sequence of the echoes were made. To obtain the velocity of the propagation of the longitudinal c_L and transversal c_T waves during the measurement by supersonic defectoscope were used following relationships [2-4]:

$$c_L = \sqrt{\frac{E(1-\nu)}{\rho\,(1+\nu)(1-2\nu)}}\,, \qquad c_T = \sqrt{\frac{G}{\rho}}\,, \tag{1}$$

where: E – Young’s modulus, G – shear modulus, ρ – density of the material, ν –Poisson’s ratio, also Young modulus E and shear modulus were obtained from relationship:

$$E = \frac{c_L^2\rho\,(1+\nu)(1-2\nu)}{1-\nu}\,, \qquad G = c_T^2\rho\ . \tag{2}$$

proportions of the velocity $c_T \backslash c_L$ depends only from Poisson’s ratio:

$$\frac{c_T}{c_L}=\sqrt{\frac{1-2\nu}{2(1-\nu)}}\,, \quad \text{result} \quad \nu=\frac{2c_T^2-c_L^2}{2\left(c_T^2-c_L^2\right)}\,. \tag{3}$$

The heterogeneity of the sample for longitudinal waves and anisotropy of the velocity was obtained from dependence [2]:

$$N=\frac{c_{L\max}-c_{L\min}}{c_{L\max}}\cdot 100\%\,, \qquad K=\frac{c_{Lsur}-c_{Lthick}}{c_{Lsur}}\cdot 100\% \tag{4}$$

where: $c_{L\min}$ – maximum of the velocity for longitudinal supersonic wave,

$c_{L\max}$ – minimum of the velocity for longitudinal supersonic wave,

c_{Lsuf} – average value of the velocity for longitudinal supersonic wave measured by the surface of the sample,

c_{Lthick} – average value of the velocity for longitudinal supersonic wave measured by the thickness of the sample.

The ferroelectric samples of the PZT ceramics were investigated. The samples were obtained by the hot pressing method and had a shape of the half of cylinder with base diameter 62,46 mm and height 16,48 mm. The chemical composition of the tested material: $Pb_{0,975}Ba_{0,01}Ca_{0,01}Sr_{0,005}(Zr_{0,52}Ti_{0,48})O_3$ + 1,4 wt. Bi_2O_3 + 0,3 wt. GeO. The density of the material was 7332,5 kg/m^3.

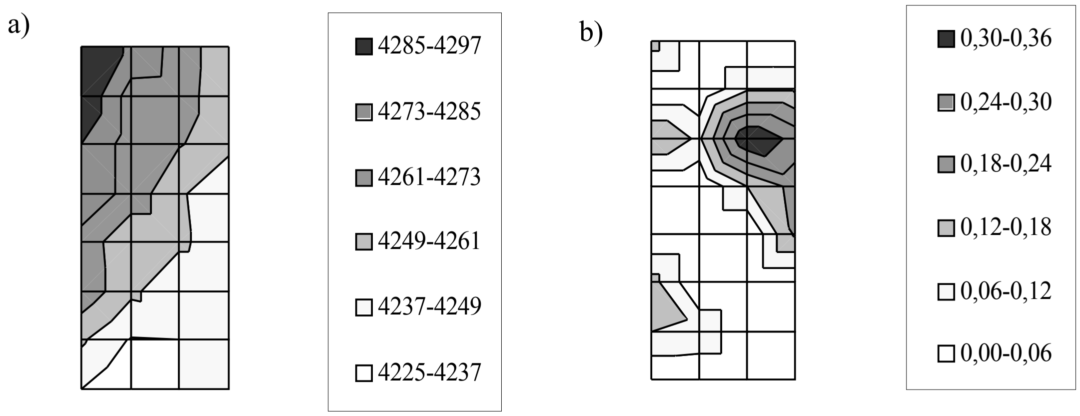

Fig. 1. The distribution on the velocity of the longitudinal waves measured by the surface (a) and the resolution of the heterogeneity (b) occurring in the investigated sample.

The surfaces of the investigated samples were divided to measurement areas. The velocities of the propagation of the longitudinal and transversal waves were measured by supersonic defectoscope UMT-11, in each of the areas of the surface.

Figure 1 shows resolution of the velocity of the propagation of the longitudinal waves (by surface) and resolution of the heterogeneity. The properties of the ceramics, obtained on the base of relationships (2) and (3) shows the table 1:

Table I. The properties of the piezoceramics investigated by the supersonic method

$\mathbf{c_L}$ [m/s] - the velocity of the propagation for longitudinal supersonic wave measured by the surface	4222 – 4299
$\mathbf{c_L}$ [m/s] - the velocity of the propagation for longitudinal supersonic wave measured by the thickness	3992 – 4304
$\mathbf{c_T}$ [m/s] - the velocity of the propagation for transversal supersonic wave	2305 – 2393
Poisson's ratio $\boldsymbol{\nu}$	0,27 – 0,30
Young's modulus **E** [GPa]	131 – 135
Shear modulus **G** [GPa]	39 – 42
Heterogeneity **N** [%]	1,7
Anisotropy of the velocity **K** [%]	3,5

Table 1 shows, that uses of supersonic methods can prove anisotropy of the physical properties and heterogeneity of the material, which is formed during the technological process. The investigated ceramics is characterized by large homogeneity (low values of N and K), high values of the Young's and shear modulus and stability of the propagation of acoustic waves.

3. RESONANCE AND ANTIRESONANCE METHODS RESEARCHES.

In the researches were used samples of the ferroelectric ceramics of the PZT type with chemical composition: $0{,}65PbTiO_3$-$0{,}33PbZrO_3$-$0{,}02[Pb(Cd_{0,5}W_{0,5})O_3]$. The samples were obtained by the hot pressing method in the shape of discs with dimensions (9x1) mm^2.

The piezoelectric properties (k_p – electromechanical coupling coefficient, V_R – acoustic velocity, d_{31} – piezoelectric modulus, S_{11}^E, C_{11}^E, S_{12}^E – elastic susceptibility) can be obtained by resonance and antiresonance method. This method depends on the measurement of the resonance vibration of the elastic elements. In the relationships below, measured frequencies of the resonance f_r and f_a and frequency of the first overtone f_h were used to obtained piezoelectric properties [5, 6]:

$$k_p = \sqrt{\frac{n^2 - 1 + \nu^2}{2(1+\nu)}\left(1 - \frac{f_r^2}{f_a^2}\right)},\quad d_{31} = \frac{0{,}188 \cdot k_p}{\frac{d}{2} f_r} \cdot \sqrt{\frac{\varepsilon}{\rho}},\quad V_R = \frac{f_R \cdot 2\pi\ r}{n},\quad S_{11}^E = \frac{2 \cdot (d_{31})^2}{(k_p)^2 \cdot (1-\nu) \cdot \varepsilon \cdot \varepsilon_0}, \tag{5}$$

where: f_a - antiresonance frequency [kHz], f_r - resonance frequency [kHz], ν - Poisson's ratio, n - the lowest positive root of molecular equation, ε - the tensor component of permittivity, ρ - density of the specimen, d - specimen diameter, r - the radius of the specimen, ε_0 - permittivity in the vacuum.

Table 2. The values of the piezoelectric parameters for investigated PZT type ceramic.

The kind of the parameters	**Values of parameters**
electromechanical coupling coefficient k_p	0,42
transversal electromechanical coupling coefficient k_{31}	0,23
piezoelectric modulus d_{31}	$77{,}3 \cdot 10^{-12}$ [C/N]
acoustic velocity V_R	2223 [m/s]
elastic susceptibility S_{11}^E	$1{,}16 \cdot 10^{-11}$ [m^2/N]
elastic modulus C_{11}^E	$8{,}61 \cdot 10^{10}$ [N/m^2]
elastic susceptibility S_{12}^E	$-4{,}59 \cdot 10^{-12}$ [N/m^2]
modulus g_{31}	$8{,}12 \cdot 10^{-3}$ [Vm/N]
electric permittivity ε_{33}^T	$9{,}52 \cdot 10^{-9}$ [F/m]
Poisson's ratio ν	0,39
density ρ	7813 [kg/m^3]

Table 2 shows values of the piezoelectric parameters in 293 K temperature obtained. The stability of the piezoelectric parameters were defined with changes of temperature and measurement of the f_r resonance and f_a antiresonance frequencies. The measurements were made in the range of temperature 293 K to 383 K. The temperature dependencies of the piezoelectric properties are shown on the figure 2. The highest changes of the piezoelectric parameters are in the range of temperature between 290K to 330K, what is connected with mobility of the domain walls.

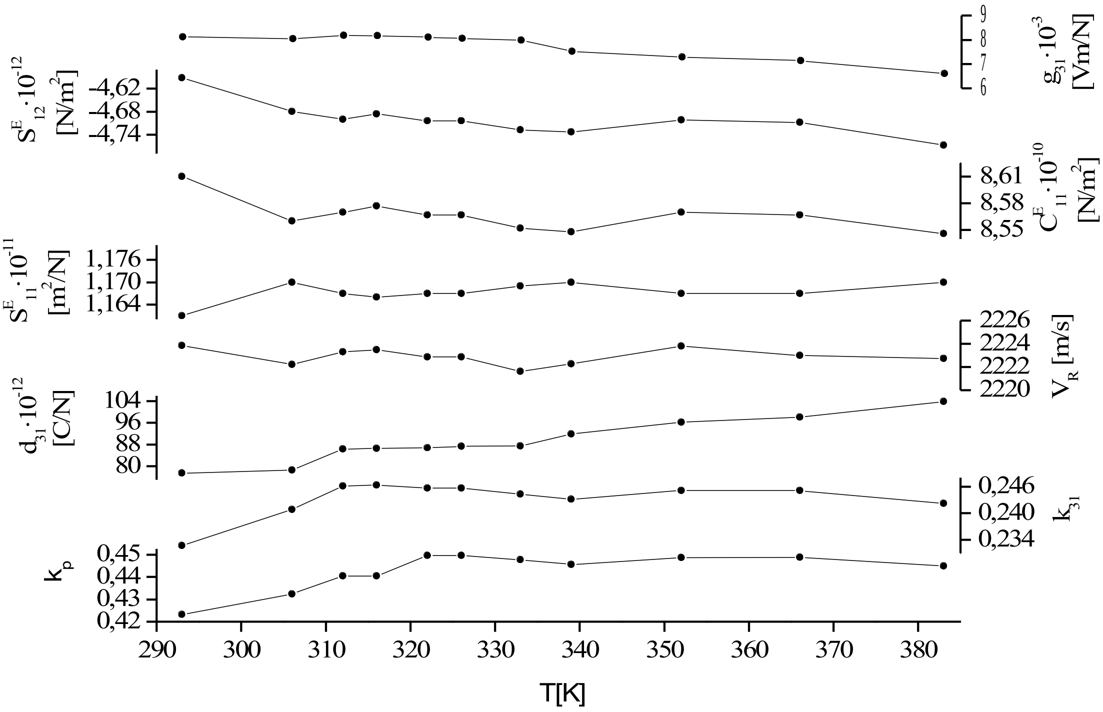

Fig. 2. Temperature dependences of the piezoelectric parameters.

Investigated ceramic belongs to the tetragonal area. In tetragonal phase exist complex of the 90° domain structure with big quantity of the 90° domain with small mobility. This stabilized domain structure brings to decreasing of the values of the piezoelectric parameters in comparison to the ceramic from morphotropy area and it causes concurrently temperature stability of this parameters. The high stability of piezoelectric parameters is condition with large value of the parameter of spontaneous deformation of the unit cell too [6].

4. CONCLUSIONS

- Application of the supersonic method allow to obtained anisotropy of the physical properties and resolution of heterogeneity (both by surface and by volume) of the investigated material.
- It has been shown that the method of the propagation of supersonic waves made possible to define Young’s and shear modulus for any area of the specimen.

- Application of the supersonic and the resonance and antiresonance methods are non destructive methods of the investigation of the real structure of ceramic materials. It brings to optimalization of the technological processes and evolutions of the range of the practical possibility of uses.
- Uses of the resonance and antiresonance made possible to obtained basic piezoelectric parameters of the ceramic specimen and description temperature stability of these parameters.

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