

ACOUSTIC EMISSION IN PZT CERAMIC IN POLING PROCESS

M. ALEKSIEJUK

Polish Academy of Sciences
Institute of Fundamental Technological Research
(00-049 Warszawa, Świętokrzyska 21, Poland)

Results of measurements of acoustic emission in PZT type ceramic in strong alternating electric field are presented. Measurements were performed with Sawyer–Tower experimental setup for poling of polarisation of these ceramic materials. Obtained signals of acoustic emission were submitted to spectrum analysis. To the interpretation of spectrum the statistical theory of noise with interactions between events was applied. Time interval parameters applied to the description of domain walls hops were found for the investigated ceramic materials.

1. Introduction

Acoustic emission method (AE) is widely applied in nondestructive testing of ferroelectric materials [1, 2]. AE was applied, among others, in studies of phase transitions [3, 4, 5] and of structure and domain dynamics [6–9]. Besides the cognitive value, the knowledge of behaviour of the domain structure in strong electric fields is important with regard to possibility of application of ferroelectric ceramics to construction of actuators, transformers, large power ultrasonic transducers etc. [9].

In this paper results of investigations of the poling process of industrial ferroelectric PZT ceramics are presented. In Sec. 2 an experimental setup is presented. In section 3 the results of AE measurements are described. In Sec. 4, analysis of the obtained experimental results and the use of statistical theory of noise to explanation of AE related to domain processes in ferroelectric materials is described.

2. Experimental setup

Acoustic emission measurements were performed in PZT ceramic materials (commercial symbols PP-1, PP-3, PP-6 and PP-9) produced in Cerad works in Warsaw. Basic properties of these materials are presented in Table 1. Measured samples had cylindrical shape (thickness 1–2 mm, diameter 10 mm). On the back surfaces of samples, metallic electrodes were fixed. Measurements of hysteresis loops were performed using

Table 1. Properties of measured PZT samples.

Name of parameter	Dimension	PP-1	PP-3	PP-6	PP-9
density	g/cm^3	7.4	7.4	7.4	7.0
dielectric constant (E_{33}^T/E_0)	–	1700	550	850	450
quality factor (Q)	–	200	1000	600	650
piezoelectric constant (d_{31})	10^{-12} C/N	160	30	90	60
piezoelectric constant (d_{33})	10^{-12} C/N	400	60	210	450
electr.-mech. coupling factor (d_{33})	–	0.3	0.16	0.26	0.22
electr.-mech. coupling factor (d_{33})	–	0.7	0.4	0.6	0.50

Sawyer–Tower method. To make it possible to disseminate heat emerging as a result of dielectric losses in ceramics the measured samples were placed in a container with transformer oil. Samples were alternatively polarised with a sinusoidal voltage at 50 Hz and with controlled amplitude up to 3.5 kV. Acoustic emission signals were detected with a broadband AE transducers located in the container. Location of the transducers in the container made it possible to obtain stronger AE signals and reduced the level of external noise. In the head of each transducer a large rate preamplifier was mounted. By this way signals from transducers were fed directly to a Tektronix oscilloscope without an additional amplification. The obtained AE signals were recorded in the memory of the oscilloscope and then they were transmitted trough a GPIB interface to a PC computer. Experimental setup is presented in Fig. 1.

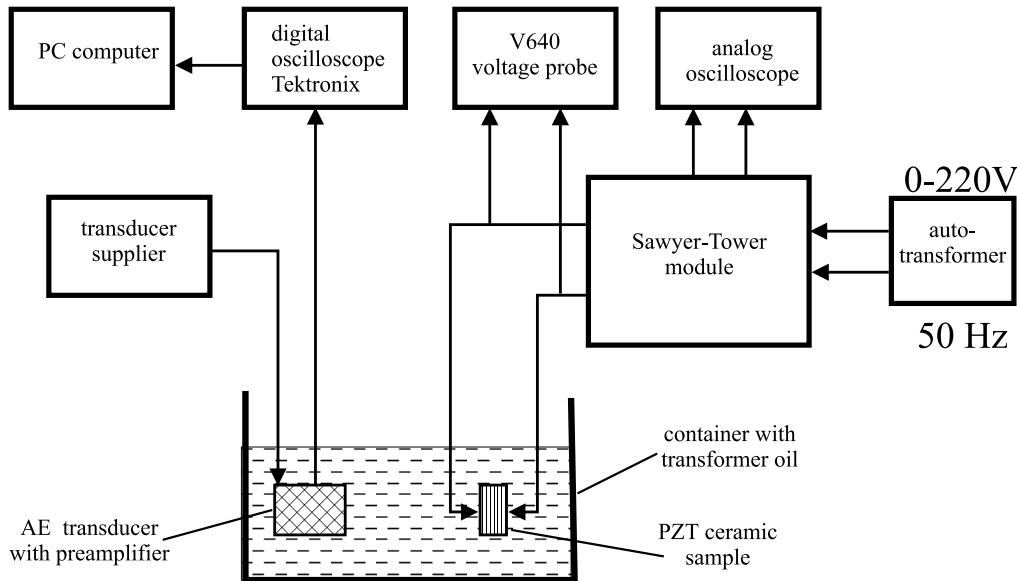


Fig. 1. Schematic diagram of experimental setup.

3. Experimental results

The AE signals repeated at 10 ms periods when the poling electric field reached its maximum. An example of a dielectric hysteresis loop for a measured sample is presented in Fig. 2. The presence of two AE signals in each cycle of change of the electric field testified that AE signals were caused by poling of material and not by creation of microcracks. The motion of domain walls caused by the electric field applied to the sample was the source of acoustic emission. In the literature there exist two different interpretations of this phenomena. In the majority of cases it is assumed that the main source of acoustic emission are the movements of the domain walls by 180° [9]. In publications of others authors it is supposed that the movements of the domain walls by 90° are the reason of the phenomenon of acoustic emission [10]. Figures 3–7 present the signals of acoustic emission for the following ceramic samples: PP-1, PP-3, PP-6, PP-9 (time scale $-50 \mu\text{s}/\text{cm}$). It can be seen that for a certain value of electric field a strong AE signal lasting for few milliseconds appears. A fast increase of amplitude of the AE signal after a certain level of poling voltage is exceeded is caused by group hops of domain walls, that is, by the

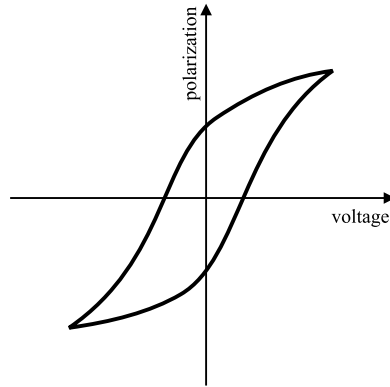


Fig. 2. Dielectric hysteresis loop of PZT ceramic (oscilloscope view).

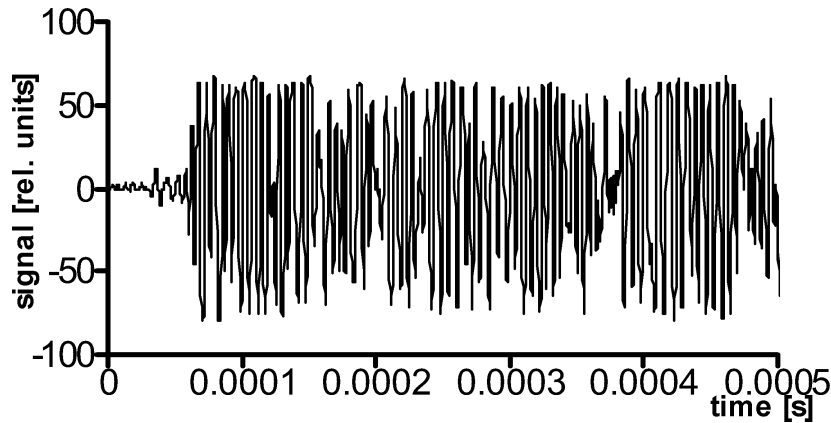


Fig. 3. AE signals of PZT ceramics (PP-1); poling voltage $U = 2.4 \text{ kV}$.

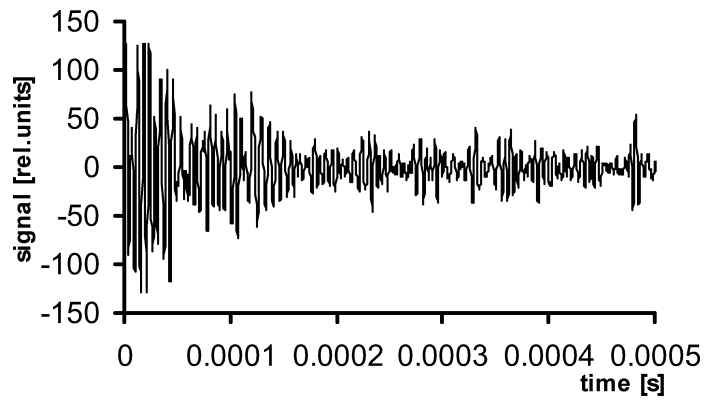


Fig. 4. AE signals of PZT ceramics (PP-3); poling voltage $U = 2.4$ kV.

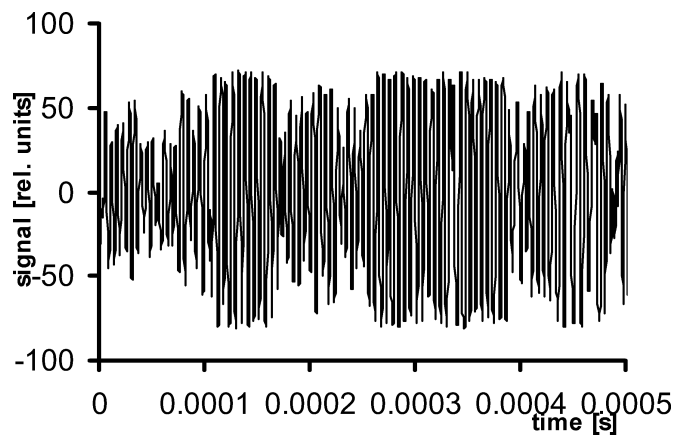


Fig. 5. AE signals of PZT ceramics (PP-6); poling voltage $U = 2.4$ kV.

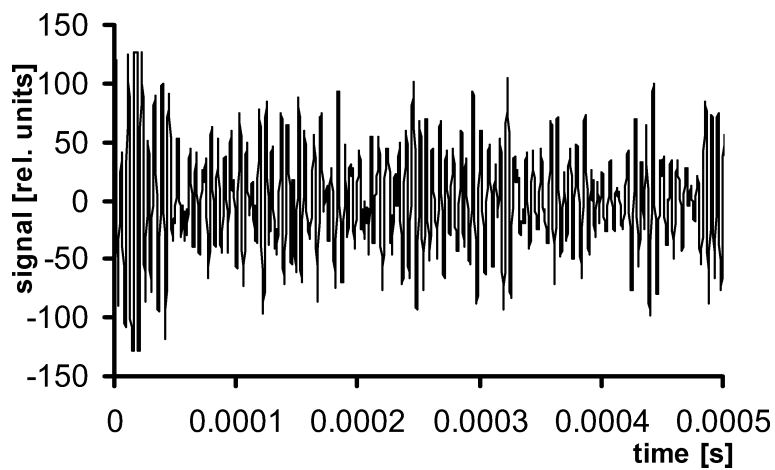


Fig. 6. AE signals of PZT ceramics (PP-6b); poling voltage $U = 2.4$ kV.

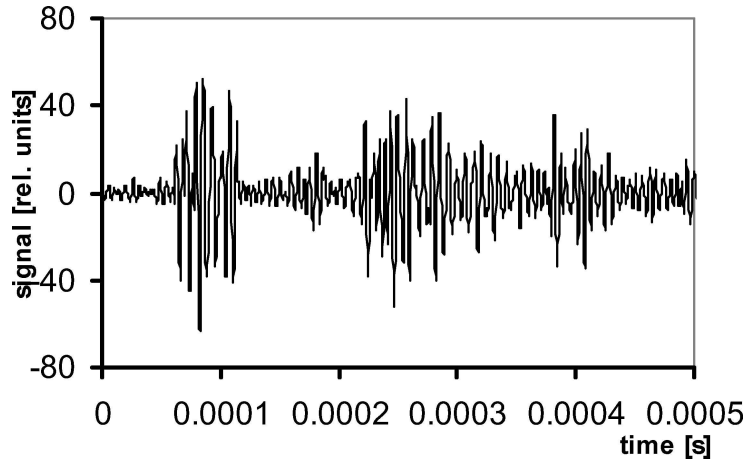


Fig. 7. AE signals of PZT ceramics (PP-9); poling voltage $U = 2.4$ kV.

emergence of clusters[11]. For magnetic materials this mechanism was observed by BITEL [12, 13]. Formation of clusters is caused by electrostatic interactions of domain walls. This interaction increases with the growth of the electric poling field in a certain range of field intensities and depends on the speed of poling of the ferroelectric material. The poling phenomenon in a material can be described in an analogy to the coherent phenomena. In the description of this phenomenon a factor is introduced which describes forced domain transitions in ferroelectric materials.

4. Spectrum analyse of acoustic emission and interpretation of results with the statistical theory

Obtained time dependencies of AE signals were subjected to spectrum analysis. In Figs. 8–9 examples of spectra of AE signals are presented. No repetitiveness of the AE spectra for the specimens made of ceramics of the same type was noticed. Spectrum is located in a range from 100 kHz to 1 MHz and have a fringe character. However, for a given ceramic specimen in a case of investigating the spectrum as a function of maximum electric field the acoustic emission appears at the same frequency ranges. This has been shown in Fig. 10 a–c. The dependence of spectrum for one sample of PP-6 ceramic is interesting (Fig. 11), as the spectrum structure is periodic with period $\Delta f = 75$ kHz. A spectrum with a similar appearance was obtained also for one sample of PP-9 ceramic. The period for this sample was $\Delta f = 30$ kHz, while the frequency range of AE in this second case was lower – only about 300 kHz. In higher frequencies only two weak fringes can be seen.

To interpret the obtained results, the statistical theory of noise created for the needs of radioelectronics, which treats random processes, was used [14]. This theory treats processes in which pulses with random parameters appear in defined time periods called time intervals (Fig. 12). These processes can be nonstationary. Spectrum function which

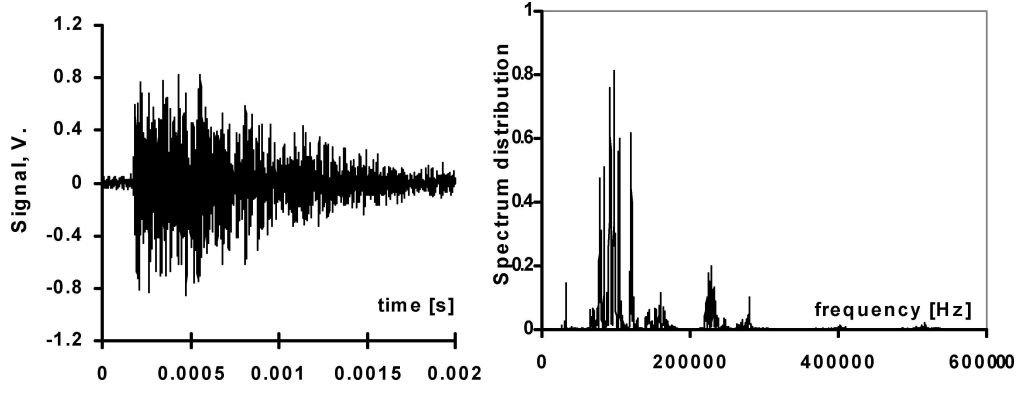


Fig. 8. Spectrum of AE signal of PZT ceramic (PP-3); poling voltage $U = 2.0$ kV.

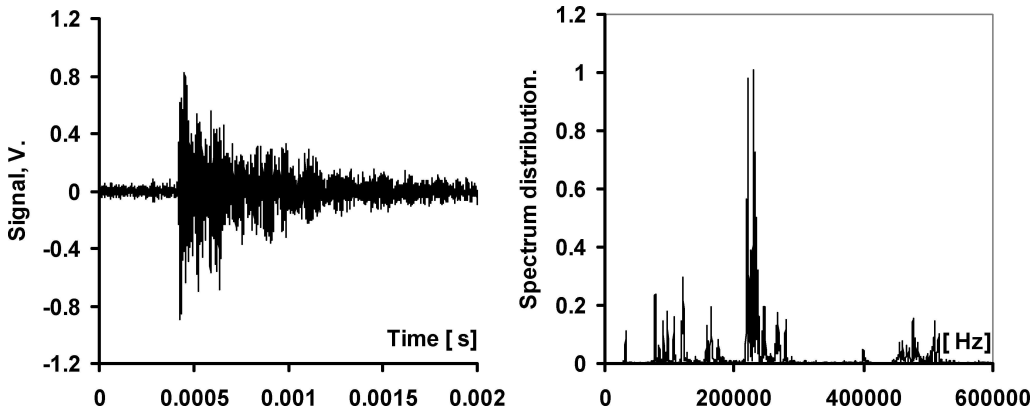


Fig. 9. Spectrum of AE signal of PZT ceramic (PP-1); poling voltage $U = 2.5$ kV.

describes such processes is shown below:

$$F(\omega) = \frac{2}{T \left(\frac{1}{\tau_p^2} + \omega^2 \right)} \sigma^2 [1 + \psi_1(\omega)] + \frac{2\pi}{T} a^2 \sum \delta \left(\omega - \frac{2\pi r}{T} \right), \quad (1)$$

where σ^2 – dispersion of amplitude of the signal, a^2 – mean value of amplitude, τ_p – constant of the transducer, r – integer value, T – time interval.

Assuming that the correlation coefficient equals:

$$R = e^{-p\beta T} \delta(p-1), \quad (2)$$

where p – difference in numbering of pulses, β – correlation constant.

The correlation function will have the form:

$$\Psi_1(\omega) = 2 \lim \sum \left(1 - \frac{p}{2N+1} \right) e^{-p\beta T} \delta(p-1) \cos p\omega T. \quad (3)$$

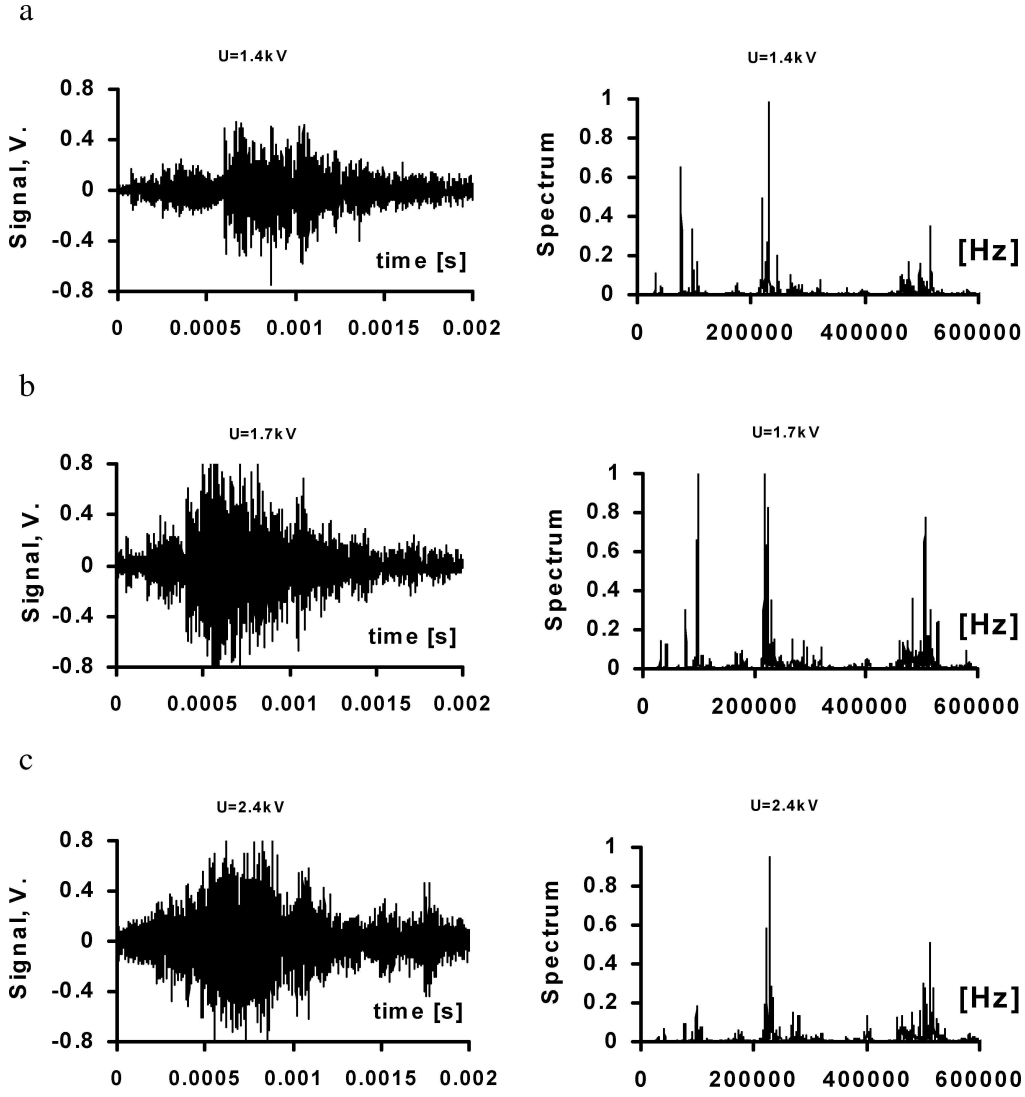


Fig. 10. AE signals and spectrum of PZT ceramic (PP-6); for different poling voltage a) $U = 1.4$ kV, b) $U = 1.7$ kV, c) $U = 2.4$ kV.

Spectrum distribution takes the form:

$$g(\omega) = \frac{2\sigma^2}{T(1/\tau_p^2 + \omega^2)} (1 + e^{-\beta T} \cos \omega T). \quad (4)$$

The introduction form of the correlation coefficient of pulses in the form (2) is related to that interaction only between neighbouring pulses is taken into account, that is, during annihilation of one domain wall a new event is generated: a hop of a second domain wall (after time period T).

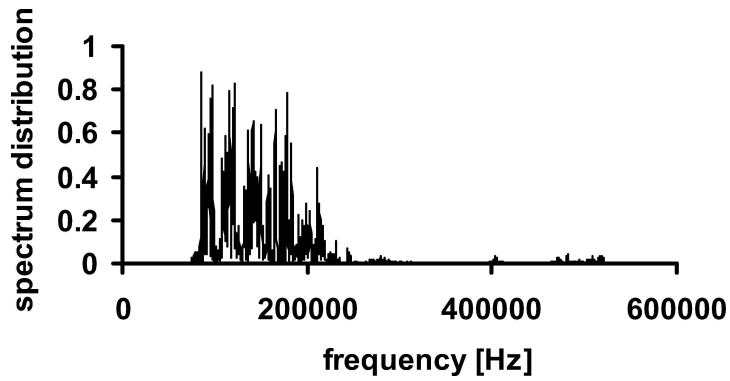


Fig. 11. Spectrum of AE signal of PZT ceramic (PP-9); poling voltage $U = 2.5$ kV.

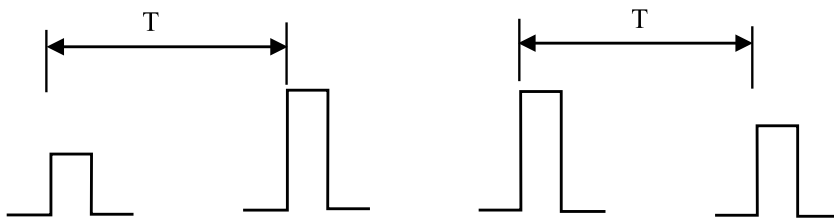


Fig. 12. Pulses with random amplitude delayed on time period T .

In Fig. 13 a theoretical spectrum obtained from formula (4) is presented. This theoretical dependence is in agreement with the dependence obtained for the sample PP-9 (Fig. 11). Time intervals obtained for these cases equal respectively: $T_1 = 300 \mu\text{s}$ and $T_2 = 100 \mu\text{s}$. These are times between following hops of domain walls. This results from that there is an interaction between the domain walls and the subsequent events of AE are dependent events.

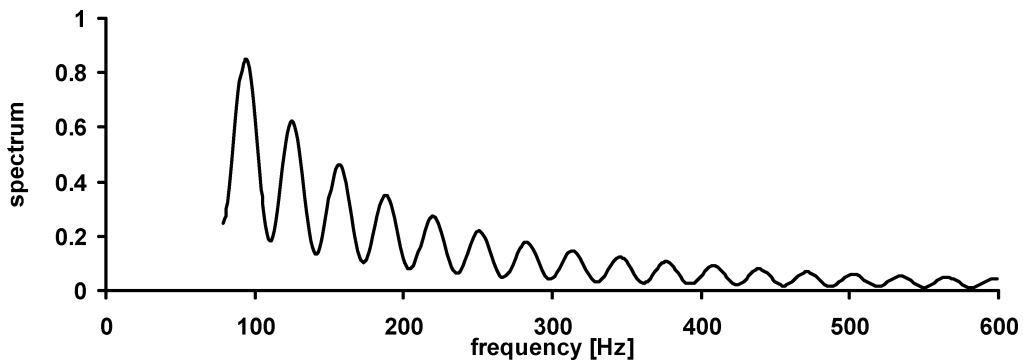


Fig. 13. Theoretical spectrum of EA signal obtained from formula (4).

5. Conclusion

From our research it results that a basis of the theory of AE in ferroelectric materials in poling processes can be the statistical theory taking into account interactions between events. Parameters of this theory are: mean value of amplitude, dispersion of amplitude and time interval T . Further studies should be carried out with this same materials. The following factors should be taken into account: changes of frequency of the poling electric field and of the internal strains caused by external factors, and the relations of the obtained results with other properties ferroelectric materials.

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