Elaboration of a piezoelectric force sensor for medical application

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Abstract. In this work, a piezoelectric force sensor was fabricated based on Lead-Free ferroelectric particles (Na\textsubscript{0.535}K\textsubscript{0.48})\textsubscript{0.966}Li\textsubscript{0.058}Nb\textsubscript{0.9}Ta\textsubscript{0.1}O\textsubscript{3} (NKLNT) synthesized by solid-state route. A piezoelectric characterization by FTIR spectroscopy and dielectric characterization was performed. The manufactured NKLNT sensor shows excellent sensitivity and response to external mechanical forces, which implies that this sensor presents a promising technology for different applications.

Keywords. Piezoelectric force sensor; Ferroelectric paritcles; solid-state route; NKLNT

1. Introduction

Piezoelectric materials capable of converting mechanical energy into electrical energy (generator or sensor mode) or vice versa (actuator mode) [1]. These materials are widely used in many technological applications and are incorporated in various electrical and electronic equipment such as actuators, sensors, transducers, etc. Piezoelectric ceramics such as lead Zirconate Titanate (PZT) [2] have very good dielectric and piezoelectric properties (high dielectric constant), but they have the disadvantage of containing lead, which has a polluting and therefore harmful impact on the planet. Therefore they must be replaced by other lead-free compositions such as (Na\textsubscript{0.535}K\textsubscript{0.48})\textsubscript{0.966}Li\textsubscript{0.058}Nb\textsubscript{0.9}Ta\textsubscript{0.1}O\textsubscript{3} particles (NKLNT) [3].

These materials are widely used in many technological applications and are incorporated in various electrical and electronic equipment such as actuators, sensors, transducers, etc. Piezoelectric sensors have several advantages, such as high sensitivity, wide frequency range, high durability, and fast response. Thus, these sensors can be widely used in several fields: Aeronautics [4], industry [5], energy recovery devices [6-8], and also in the medical field [9]. In the literature, there are different synthesis methods for the elaboration of piezoelectric sensors, such as hydrothermal synthesis [10], sol-gel synthesis [11, 12], combustion synthesis [13], and solid-state synthesis [14-17]. In this work, we have
developed a method for the synthesis of a solid-state piezoelectric force sensor using sodium carbonate (Na$_2$CO$_3$), potassium carbonate (K$_2$CO$_3$), lithium carbonate (Li$_2$CO$_3$), tantalum oxide (Ta$_2$O$_5$) and niobium oxide (Nb$_2$O$_5$). FTIR, dielectric and piezoelectric characterizations were performed. An interpretation of the results of the different characterizations as well as the application of the NKLNT ceramic as a force sensor was presented.

2. Experimental procedure

The method used for the synthesis of NKLNT ceramic is the solid route. The mixing of the raw materials was carried out in an alcoholic medium in a Teflon bowl with the help of an electric motor (attritor), in the presence of Zirconia beads to get a more homogeneous slip. After stirring for two hours, the suspension was then sieved and oven-dried for 12 hours. After that, the powder is subjected to a thermal cycle (Fig. 1) using a programmable oven called calcination. This operation aims to transform the mixture into a powder of well-defined compositions and structures.

The fireclay obtained after calcination underwent a second grinding to reduce the size of the grains and increase their reactivity, this step also allows a better homogenization of the powder. Disc-shaped compacts of 11.01 mm diameter and 1.16 mm thickness were then formed using a uniaxial press at a pressure of 30 kN (Fig. 2).

The ceramic is finally sintered at a temperature of 1110 °C for two hours with a mounting rate of 5 °C/min and natural cooling to room temperature inside the furnace. The sintering cycle is shown in Fig. 2.

1) Polishing of NKLNT ceramic: The ceramics after sintering did not have the same thickness. They also have slight deformations. For this, we used the surfaces of the ceramics as well as possible and remove all the apparent porosity on the surface which allows us to decrease the dielectric losses.

2) Metallization of NKLNT ceramic: Both surfaces of the ceramic are covered with a thin layer of silver lacquer (metallization) and then dried at 100°C for 30 minutes to obtain plane capacitors. These pellets are then heat-treated at 600°C for 15 minutes to form the electrodes.

3) Polarization of NKLNT ceramic

For piezoelectric measurements, polarization of the sample took place at room temperature under a 3 kV electric field for 30 minutes.

3. Dielectric and piezoelectric characterization

Measurement of dielectric properties such as capacitance C and dielectric losses tan(δ) was performed using an impedance meter under voltage (about 1V) at different frequencies (1KHz, 10KHz, 100KHz and 1MHz) and at different temperatures (cooling from 500°C to 28°C). The permittivity is then given by the following relation:

\[ \varepsilon_r = \frac{e \times C}{\sigma S} \]  

(1)
- **C**: measured capacitance (F).
- **e**: distance between electrodes (m).
- **S**: area of electrodes (m$^2$).
- **$\varepsilon_0$**: vacuum permittivity, $\varepsilon_0 = 8.85 \times 10^{-12}$ (F/m).

The evolution of the capacitance, relative permittivity, and dielectric losses as a function of the temperature at different frequencies are illustrated in Fig.3, 4 and 5 respectively.

**Figure 3.** Evolution of capacity as a function of temperature at different frequencies.

**Figure 4.** Evolution of relative permittivity as a function of temperature at different frequencies.

**Figure 5.** Evolution of dielectric losses as a function of temperature at different frequencies.
According to figure 4, the relative permittivity depends on the temperature. We can see that there is a transition from the orthorhombic phase to the tetragonal phase at low temperature, and the transition from the tetragonal phase to the cubic phase at a higher temperature. The dielectric losses obtained in Figure 5 are lower; they increase rapidly at higher temperature due to the rapid increase in material conductivity. The piezoelectric constant \( d_{33} \) coefficient \( = 147 \, \text{pC/N} \) were measured using a piezoelectric meter by applying a 1N force at 100 Hz, and measuring the variation of charges \( Q \). This coefficient is determined by the following relation:

\[
\delta_{33} = \frac{Q}{F}. \tag{2}
\]

- \( Q \): electrical charges (pC).
- \( F \): force (N)

4. Application of NKLNT ceramic as force sensor

a) The electrical circuit

After the polarization step, the sensor has acquired piezoelectric properties. By applying mechanical stress (vibrations), electrical charges were generated on both sides of the ceramic sensor. For this, a conditioning circuit is used to convert the generated charges into an equivalent electrical signal. This circuit is mainly composed of two parts (charge-voltage converter and amplifier stage).

![Figure 6. Electrical circuit of the conditioning system used.](image)

b) Charge-voltage converter

The force sensor is connected to an operational amplifier which functions as an integrator to compensate for the electrical charges produced. The voltage measured at the capacitor terminals is proportional to the charges produced by the force sensor. These charges are converted into a voltage using the following equation:

\[
V = - \frac{Q}{C} \tag{3}
\]

- \( Q \): electrical charges (pC).
- \( C \): capacitance (F).

c) The amplification part

The output signal of the sensor is weak and noisy, so an instrumentation amplifier with high rejection and a gain of 20 was used to reduce the noise and amplify the signal.

d) Results of tests

The experimental setup for our application is shown in Fig. 7.
Figure 7. The electrical circuit of the proposed application.

Figure 8. Output voltage of the sensor as a function of the applied force.

5. Conclusion

A force sensor based on lead-free ferroelectric particles NKLNT has been realized. The dielectric loss (\(\tan(\delta)\)) obtained was lower, and the dielectric constant (\("\varepsilon_r"\)) varied from 800 to 6000 at room temperature and \(T_c\), respectively. A good piezoelectric coefficient \(d_{33} = 147\ \text{pC/N}\) was measured. A relatively homogeneous microstructure and low porosity were also obtained.

These results show that the NKLNT ceramic is a technology that can fulfill the role of a force transducer for a medical application (functional hand rehabilitation).

References

[1] Li, J. F., Wang, K., Zhu, F. Y., Cheng, L. Q., & Yao, F. Z. (2013). (K, Na) NbO 3-Based Lead-Free Piezoceramics: Fundamental Aspects, Processing Technologies, and Remaining Challenges. Journal of the American Ceramic Society, 96(12), 3677-3696.

[2] Kreft, H., & Jetz, W. (2007). Global patterns and determinants of vascular plant diversity. Proceedings of the National Academy of Sciences, 104(14), 5925-5930.

[3] Chelli, Z., Achour, H., Saidi, M., Laghrrouch, M., Chaouchi, A., Rgui, M., ... & Courtois, C. (2021). Fabrication and characterization of PU/NKLNT/CFs based lead-free piezoelectric composite for energy harvesting application. Polymer-Plastics Technology and Materials, 1-13.
[4] Kras, A., Brahim, M., Porchez, T., Bouchet, C., & Claeyssen, F. (2014, June). Compact, lightweight, and efficient piezoelectric actuation chain for aeronautical applications. In 14th International Conference on New Actuators.

[5] Nuffer, J., & Bein, T. (2006, October). Application of piezoelectric materials in transportation industry. In Global symposium on innovative solutions for the advancement of the transport industry (Vol. 4, No. 6).

[6] Choi, W. J., Jeon, Y., Jeong, J. H., Sood, R., & Kim, S. G. (2006). Energy harvesting MEMS device based on thin film piezoelectric cantilevers. Journal of Electroceramics, 17(2-4), 543-548.

[7] Jeon, Y. B., Sood, R., Jeong, J. H., & Kim, S. G. (2005). MEMS power generator with transverse mode thin film PZT. Sensors and Actuators A: Physical, 122(1), 16-22.

[8] Lee, B. S., Lin, S. C., Wu, W. J., Wang, X. Y., Chang, P. Z., & Lee, C. K. (2009). Piezoelectric MEMS generators fabricated with an aerosol deposition PZT thin film. Journal of Micromechanics and Microengineering, 19(6), 065014.

[9] Boukhenous, S., & Attari, M. (2011). A low cost instrumentation based sensor array for ankle rehabilitation. Chapter Book: Biomedical Engineering, trends in Electronics, Communications and Software, 69-78.

[10] Xin, R., Ren, F., & Leng, Y. (2010). Synthesis and characterization of nano-crystalline calcium phosphates with EDTA-assisted hydrothermal method. Materials & design, 31(4), 1691-1694.

[11] Barrow, D. A., Petroff, T. E., Tandon, R. P., & Sayer, M. (1997). Characterization of thick lead zirconate titanate films fabricated using a new sol gel based process. Journal of Applied Physics, 81(2), 876-881.

[12] Kobayashi, M., Jen, C. K., & Levesque, D. (2006). Flexible ultrasonic transducers. IEEE transactions on ultrasonics, ferroelectrics, and frequency control, 53(8), 1478-1486.

[13] Aruna, S. T., & Mukasyan, A. S. (2008). Combustion synthesis and nanomaterials. Current opinion in solid state and materials science, 12(3-4), 44-50.

[14] Gabbott, P. (Ed.). (2008). Principles and applications of thermal analysis. John Wiley & Sons.

[15] Newman, A. W., & Byrn, S. R. (2003). Solid-state analysis of the active pharmaceutical ingredient in drug products. Drug discovery today, 8(19), 898-905.

[16] Savolainen, M., Kogermann, K., Heinz, A., Aaltonen, J., Peltonen, L., Strachan, C., & Yliruusi, J. (2009). Better understanding of dissolution behaviour of amorphous drugs by in situ solid-state analysis using Raman spectroscopy. European journal of pharmaceutics and biopharmaceutics, 71(1), 71-79.

[17] Saidi, M., Chaouchi, A., d’Astorg, S., Rguiti, M., & Courtois, C. (2015). Dielectric, ferroelectric properties and impedance spectroscopy analysis of the [(Na 0.535 K 0.480) 0.966 Li 0.058](Nb 0.90 Ta 0.10) O 3-based lead-free ceramics. Journal of Advanced Dielectrics, 5(01), 1550007.