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Short Communication

Effect of Temperature Path on the Poling of Commercial Hard PZT Ceramics and its Implication for Mass Production

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Abstract

In order to identify process parameters for the efficient poling of hard piezoelectrics in mass production, the effect of temperature modification during poling is investigated for a commercial hard PZT in the temperature range from 25 °C to 150 °C. While an electric field is applied, the temperature of the specimens was kept constant or the specimens were heated or cooled. The resulting piezoelectric performance was evaluated on the basis of the planar coupling factor at room temperature. An optimal poling temperature associated with maximum piezoelectric performance was found for poling with constant temperature. However, for poling while cooling, even higher performance is attained if the starting temperature is high enough. If poling becomes necessary after fabrication at high temperature, the cooling period could be exploited. The observed correlation between permittivity and the coupling factor could be exploited in quality control.

Keywords: Piezoceramic, hard PZT, polarization, serial production

I. Introduction

Piezoceramics are used as sensors and actuators in smart structures. At the moment, piezoceramic elements are applicated after fabrication of the base structure, which involves considerable effort and expense. Current technological investigations aim at the direct integration of the functional elements during the production, reducing production time and costs, enabling low-cost mass production. Promising approaches are the serial production of composites with spherical particles and fibers made of PZT ceramic [1], consolidated directly into polymer packages [2] and thermoplastic composite structures [3], metal die casting [4] as well as ceramic packages [5, 6] (see Schönecker [7] for a detailed overview). These approaches are linked by the fact that elevated temperatures are used for integrating the functional elements.

To activate the piezoelectric effect, the ceramic has to be poled to achieve a remanent polarization in the material. Soft PZT ceramics are easily poled since they require low temperatures, low electric fields and poling times. Some soft piezoceramics can be poled with poling times lower than 0.05 s [8]. Poling of soft PZT can thus be conducted with poling times from 1 to 30 s under isothermal conditions. On the contrary, hard PZT ceramics, which are essential for applications requiring high piezoelectric performance at high temperatures (> 150 °C), high power operation modes (minimized losses) and highly linear piezoelectric behavior, are much more complex with regard to achieving remanent polarization. Poling of hard piezoceramics demands raised temperatures, long poling times and high electric fields – these conditions necessitate considerable extension of the manufacturing process, demanding additional process steps (heating, cooling) associated with additional equipment as well as longer processing times. In this context, hard PZT seems unsuitable for mass production with respect to cycle times and production costs. This situation could be avoided by direct implementation of the poling step into the manufacturing chain of the structural device. In particular, exploiting the process temperature during integration appears beneficial and effective. Often, the temperature is decreasing or increasing during these processes. The influence of a constant temperature on the poling behavior has been reported [9, 10]. In the present work, poling of the commercial hard PZT ceramic PIC181 (PI Ceramic GmbH, Lederhose, Germany) under different temperature-electric field regimes is investigated with the goal of identifying optimized process parameters. Thus, thermal conditions could be systematically used for the poling process. The planar coupling factor k_p^{RT}, the loss factor tan δ, and the relative permittivity ε^{RT} are determined at room temperature (RT). k_p^{RT} is used as a direct measure for the obtained piezoelectric performance after poling. Additionally, the correlation between ε^{RT} and k_p^{RT} is analyzed with the aim of deriving a simple and robust indirect measure for the poling state. The loss factor tan δ is determined in order to recheck the effect of the temperature paths on the dissipative characteristics. After the experimental procedure is explained in Section 2, the results are presented and discussed in Section 3. In the last section, conclusions con-

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cerning the application of the presented findings to mass production as well as for future work are given.

II. Experimental

Experiments were conducted using samples of the commercial hard PZT ceramic PIC181 (PI Ceramic GmbH, Lederhose, Germany). The disc-shaped specimens have the dimensions of 10 mm in diameter and 1 mm in thickness and are coated with a burned-in silver electrode. Ten specimens were poled and measured for each temperature path point. In order to achieve a random distribution of domains and thus an initially unpoled state, the specimens were depolarized by heating above their Curie temperature (350 °C, see Ref. 11) with closed contacts for 1 hour. For poling, the specimens were placed in an insulating oil bath (Midel 7131) to avoid flashovers.

The oil was heated using a heating plate (IKAMAG REC-G) with temperature control (IKATRON ETS03) in the range between 25 °C – 150 °C. The temperature was measured at a distance of 20 mm to the sample. LabView was used to control the high-voltage amplifier (GBR electronics 3 – 2a) and the voltage measurement (Oscilloscope DSB7000B).

In the first experiment (poling path **"heating"**), the samples were heated from room temperature to the upper temperature limit T_{max} with an applied electric field strength of 2.5 kV/mm for 20 min. After the target temperature had been reached, the electric field was removed and the samples were cooled to room temperature with open contacts.

In the second experiment (poling path **"const. T"**), the samples were heated and then poled at a constant temperature for 20 min with an electric field of 2.5 kV/mm. After poling, the electric field was removed and the samples were cooled to room temperature with open contacts.

In the third experiment (poling path **"cooling"**), the samples were heated to T_{max} and then poled during the cooling process back to approximately room temperature with an electric field strength of 2.5 kV/mm applied. After room temperature had been reached within 30 to 110 min, depending on the start temperature, the electric field was removed.

The impedance spectrum was measured at room temperature 80 hours after poling using the HP 4194A Impedance/Gain-Phase analyzer. The planar coupling factor k_p^{RT} was calculated from the values of resonance and anti-resonance frequency of the planar mode, according to the metrological standard DIN EN 50324 – 2.

III. Results and Discussion

The graph in Fig. 1 shows the dependence of the planar coupling factor k_p^{RT} on the upper temperature T_{max} for the different temperature paths.

The reproducibility is very high, as indicated by the error bars. Exceptions are observable for the poling path **cooling** at two measurement stations, which could be caused by discontinuous experimental conditions.

The poling path **const. T** shows an increase of k_p^{RT} with rising poling temperature T_{max} up to 120 °C. The local maximum corresponds to k_p^{RT} = 0.552 (cf. Table 1). Further increase of the temperature leads to a decrease in k_p^{RT} which amounts 6.8 % at 150 °C. The coupling factor shows dependence on temperature, but also on electric field strength and poling time. The optimum temperature of 120 °C thus only corresponds for the poling conditions used here (electric poling field 2.5 kV/mm, 20 min holding time).

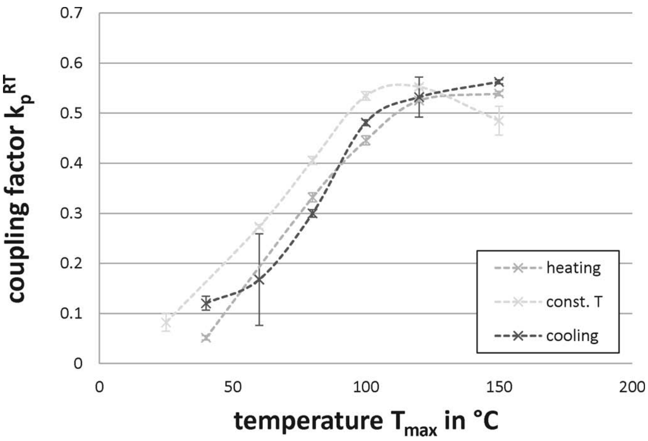

Fig. 1: Planar coupling factor of PIC181 as a function of temperature T_{max} for different poling paths.

Poling during **cooling** does not result in better coupling factors for the temperature range from 25 °C to 120 °C. The coupling factors are up to 10 % lower than for poling at constant temperatures. Here, the poling time spent at high temperatures, e.g. target temperature, is shorter, which seems to lead to lower k_p^{RT}. For the temperature path point of T_{max} = 150 °C, **cooling** attains 8.3 % better results than the poling path **const. T**. The ongoing increase of k_p^{RT} with increasing temperature, which stands in contradiction to the result found for load path **const. T**, may be due to the shorter time spent above 120 °C. At 150 °C, a 2 % higher value in the coupling factor related to the maximum value obtained for **const. T** is attained.

Table 1: Maximal achieved coupling factors and corresponding relative permittivity for the temperature paths investigated.

Temperature path	$k_p^{RT}{}_{max}$	ε^{RT}	T in °C
Heating	0.539 ± 0.00437	1211 ± 11.25	150
T const.	0.552 ± 0.00530	1184 ± 7.12	120
Cooling	0.563 ± 0.00398	1120 ± 9.93	150

The temperature path **heating** shows a similar progression. In the temperature range up to 120 °C, the results show no improvement in comparison to **const. T**, but further increased temperatures result in similar effects as experienced with **cooling**: at 150 °C, the coupling factor almost reaches the value for **const. T**.

Potentially, higher k_p^{RT} may be attained by a further increase of T_{max} for the temperature paths **heating** and **cooling**. Within the scope of this investigation, the test stand was limited to 150 °C maximum.

Enhanced temperatures in manufacturing processes can be utilized for poling. If the poling is started at sufficiently high temperatures (e.g. 150 °C or above) and conducted during **cooling**, it is not necessary to implement additional holding times at a constant poling temperature. It is even possible to attain better poling properties than with conventional poling procedures.

The dissipation factor tan δ was also measured within this investigation. It does not change significantly for the different temperature paths and amounts to 0.001 – 0.003, reproducing the values given by the material supplier [11]. Thus, the here considered temperature paths do not affect the outstanding low loss characteristics of hard PZT.

The relative permittivity ε^{RT} is plotted over k_p^{RT} in Fig. 2. All curves show a distinguishable exponential increase in permittivity with increasing k_p^{RT}. The correlation between ε^{RT} and k_p^{RT} allows for an indirect determination of the poling state and thus the piezoelectric performance of the PZT. This important finding provides a method for quality assurance in mass production based on capacitance measurements that are simple, fast and robust. In addition, this method is not limited by the geometry of the active structure or the functional element, which becomes essential in the context of mass production of directly integrated structures. Complex measurement techniques for non destructive evaluation of the poling state are avoided.

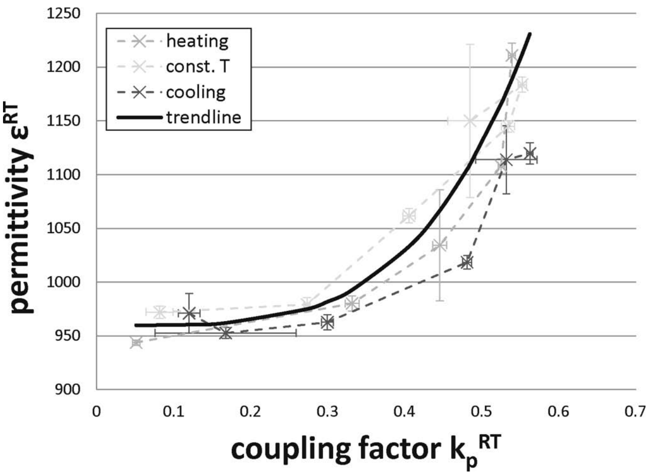

Fig. 2: Relative permittivity of PIC181 over coupling factor k_p^{RT} for different poling paths.

IV. Conclusions

The results show that integration processes that involve high temperatures can be combined efficiently with the poling process. When additional heating of the ceramic is necessary for poling, the optimum temperature is 120 °C combined with a holding time of 20 min and an electric poling field of 2.5 kV/mm. If higher temperatures are present during the integration process, utilizing of cooling or heating achieves better room temperature results than poling at a constant poling temperature. Thus, holding times at constant temperatures can be omitted if the poling process is implemented directly into the integration process, saving additional process steps and time.

Also, it could be verified that a correlation between the coupling factor and permittivity exists. This circumstance may be used for quality control in mass production, demanding only a minimum of measurement effort and time while providing a highly robust method.

Future work should be aimed at the application of these findings to a specific component or structure. Furthermore, the influence of poling time and electric field on the maximum achievable poling state should be investigated. The investigated temperature range should be expanded to higher temperatures in order to investigate whether even better piezoelectric performance is attainable. In addition, it should be interesting to study at which temperature during cooling the electric field can be removed without diminishing the attained performance.

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