SYNTHESIS, STRUCTURAL ANALYSIS AND INITIAL ELECTRIC SCRATCHING ON PbZr$_x$Ti$_{(1-X)}$O$_3$ PIEZOELECTRIC MATERIAL

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ABSTRACT

SYNTHESIS, STRUCTURAL AND INITIAL ELECTRIC SCRATCHING ON PbZr$_x$-Ti$_{(1-x)}$O$_3$ PIEZOELECTRIC MATERIAL. PbZr$_x$Ti$_{(1-x)}$O$_3$ (PZT) material is currently one of the highest-quality piezoelectric materials and has been widely used in various applications, including as sensors in the observation system in nuclear reactor cores. In this study, a solid state reaction method was used to synthesize PbZr$_x$Ti$_{(1-x)}$O$_3$ (PZT) material with variations in compaction pressure of 7000 and 5000 psi. The sample synthesized was analyzed by the crystal structure system using Rietveld method of X-ray diffraction (XRD) pattern. The fitting results from Highscore program showed compounds with perovskite crystals system of tetragonal (PbZr$_{0.52}$Ti$_{0.48}$O$_3$) and rhombohedral (PbZr$_{0.58}$Ti$_{0.42}$O$_3$) and PbTiO$_3$ (tetragonal). The electrifying process using an electrical voltage of kv DC has been successfully carried out on synthesis products. The voltage source used is from the electronic circuit Television (TV) 14-21 inches which has a flyback component and then continues with a voltage drop by making a voltage divider. The polling results can be identified by measuring the piezoelectric constant with the d33 meter device. Optimization of polling parameters to the material temperature was obtained at 100 ºC, then applied polling sample of PbZr$_x$Ti$_{(1-x)}$O$_3$ (PZT) material added by PVDF (1.5, 2.5 and 5% weight percent) to samples with the compaction of 7000 and 5000 psi. Maximum d33 measurement (61 x 10^{-12} C/N) was obtained on PZT material with a pellet pressure of 7000 psi. Increasing the addition of PVDF results in a reduction of d33.

Keywods: PbZr$_x$Ti$_{(1-x)}$O$_3$ Piezoelectric material, d33 Piezoelectric constant

ABSTRAK

SINTESA, ANALISA STRUKTUR DAN PEMOLINGAN LISTRIK AWAL PADA BAHAN PIEZOELEKTRIK PbZr$_x$Ti$_{(1-x)}$O$_3$. Bahan **PbZr$_x$Ti$_{(1-x)}$O$_3$** (PZT) merupakan salah satu deretan bahan piezoelektrik berkualitas tertinggi saat ini dan telah banyak digunakan dalam berbagai aplikasi, termasuk sebagai sensor pada sistem pengamatan di *core* reaktor nuklir. Pada penelitian ini digunakan metode solid state reaction untuk mensintesis bahan PbZr$_x$TI$_{(1-x)}$O$_3$ (PZT) dengan variasi tekanan kompaksi dari 7000 dan 5000 psi. Sampel hasil sintesa dianalisis sistem struktur kristalnya dengan menggunakan metode Rietveld dari pola difraksi sinar-X (XRD). Hasil *fitting* dari program *Highscore* menunjukkan senyawa-senyawa dengan sistem kristal perovskite tetragonal (PbZr$_{0.52}$Ti$_{0.48}$O$_3$) dan rhombohedral (PbZr$_{0.58}$Ti$_{0.42}$O$_3$) serta bahan PbTiO$_3$ (tetragonal). Pada proses pemolingan listrik dengan menggunakan tegangan listrik berorde kV DC telah berhasil dilakukan pada produk sintesa. Sumber tegangan yang digunakan adalah dari rangkaian elektronik Televisi (TV) 14-21 inchi yang memiliki komponen *flyback* dan kemudian dilanjutkan dengan penurunan tegangan dengan cara membuat pembagi tegangan. Hasil pemolingan dapat diidentifikasi dengan pengukuran konstanta piezoelektrik dengan alat d$_{33}$ meter. Optimasi parameter poling terhadap suhu bahan diperoleh pada 100 ºC, kemudian diaplikasikan pemolingan sampel bahan PbZr$_x$Ti$_{(1-x)}$O$_3$ (PZT) yang ditambahkan PVDF (1,5; 2,5 dan 5 % porsentase berat) untuk sampel-sampel dengan kompaksi 7000 dan 5000 psi. Pengukuran d$_{33}$ maksimum (61 x 10^{-12} C/N) didapatkan pada bahan PZT dengan tekanan pelet 7000 psi. Peningkatan penambahan PVDF menghasilkan pengurangan d$_{33}$.

Kata kunci: Bahan piezoelektrik PbZr$_x$Ti$_{(1-x)}$O$_3$, Konstanta piezoelektrik d$_{33}$

INTRODUCTION

Piezoelectric material is one of the ingredients included in the classification of smart materials and is currently being developed by researchers today. The uniqueness of this material is that it is subjected to pressure on the material or changes in shape will produce electricity and vice versa; the electric plane applied to the material will cause changes in shape or strain. Thus, many piezoelectric materials have been applied in various electronic devices. Among them are cantilever which can produce low power electricity [1], mini electric motors and medical devices on ultrasonography (USG). One of the intensive applications developed today is ferroelectric memory which is expected to replace the existing magnetic memory system as developed by G.H. Haertling [2][3]. Several researchers have now begun conducting studies on the development of applications for piezoelectric material sensors to observe the environment in the reactor cores [4,5].

To get high performance on piezoelectric based equipment, it is necessary to improve the quality of the material, both in the material preparation in the synthesis process and the polling process. PSTBM-BATAN has been and is currently synthesizing various methods, including molten salt, solid state reaction, sol gel and hydrothermal. Analyzing the crystal structure of the synthesis results is important in indicating the progress of the material quality, one of which is the existence of a morphotropic phase boundary (MPB) area that contains the boundary of the two structure phases [6]. However, the results of the synthesis itself cannot be applied directly or not ready to use, so one more step is needed, that is the material polling process [7]. The polling itself means that the domain of dipole moments arranged randomly can be rectified as shown in Figure 1 below.
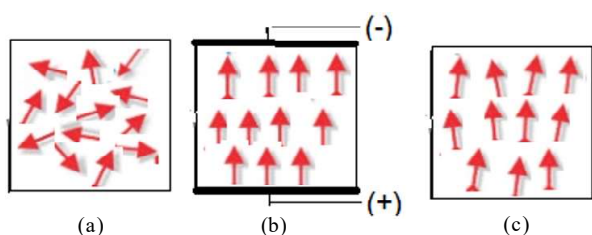

Figure 1. Direction of dipole moment, (a). random polarization domain, (b). polarization in the electric field, (c). remanent polariza-tion, after the electric plane is released.

Figure 1 shows the randomization of the electric dipole moment from the material after synthesis and then polling is conducted so that the polarization is in the direction and when the polling voltage is released, the direction of the polarization domain is still dominant (remanent) in the polling direction. This post polling piezoelectric material is generally applied to various tools. The polling voltage itself is quite high for ceramic materials, such as $Zr_{0.52}Ti_{0.48}O_3$ (PZT) that can reach kV/mm order.

Information on the polling parameter itself for piezolectric material is very limited, so it is necessary to do an initial or preliminary polling process. Besides that, the availability of high voltage equipment with special security is also necessary. Another issue is the sample making with a relatively high compaction, in order to reduce leakage current during the polling process. The polling parameters of this piezoelectric material are quite numerous, including temperature, time, high voltage and sample thickness. With the limitations of these parameters obtained, both in scientific publications and in nonscientific media, it is necessary to do preliminary/initial polling, in addition to security techniques during the polling process.

This study aims to investigate the relationship between the synthesis process with compaction changes, analysis of crystal structures in determining the content of the phases formed to represent MPB area and the polling process using high voltage Flyback TV order of kilovolts. The polling process of course is also determined by the polling parameters themselves.

PZT material synthesis is carried out by solid state reaction method [8,9]. From the synthesis results, the crystal structures were identified using the X-Ray Diffractometer (XRD) and analyzed using the Highscore program. The results of optimizing several polling parameters were applied to PZT piezoelectric composite materials with Polyvinylidene Fluoride (PVDF), as developed by Chuan Gui Wu [10]. The high voltage used in this study empowers Flyback TV.

EXPERIMENT METHOD

Solid state reaction method is widely used to produce piezoelectric materials (such as $Bi_{0.5}Na_{0.5}TO_3$ and $BaTO_3$ in PSTBM-BATAN). This method is also applied to produce $PbZr_{0.52}Ti_{0.48}O_3$ (PZT) material. The basic ingredients used in stoichiometry are PbO as 24.6823 gr, ZrO_2 as 7.0783 gr and TiO_2 as 4.2370 gr. These basic materials are crushed for 4 hours, compacted (at 7000 and 5000 psi) in the form of a 1.5 cm diameter pellet, then preheated at 200 °C for 1 hour (calcination) and continued with sintering heating at 950 °C for 4 hours. In this method, the diffusion process and reaction will occur during the sintering temperature. [8,9]

For samples of elelctric polling process of this piezoelectric material, the resulting PZT material was added Polyvinylidine Fluoride, abbreviated PVDF, (1.5, 2.5 and 5% by weight percentage) and some other samples were varied by the pellet pressures of 5000 and 7000 psi. With these samples, the polling process was carried out using a flyback TV circuit. The mixing of PZT + PVDF was carried out after obtaining PZT material from the synthesis process, so that the mixing process was composite.

The TV circuit and its Flyback are an empowered high voltage source [11], as shown in Figure 2. The Flyback voltage empowered at this study is located on Winconsan TV electronic circuit with a DC high voltage of about 18-21 kV DC. The high voltage produced is measured using a high voltage probe. However, the presence of high voltage can also be tested by displaying electron jumps between 2 separate electrodes (positive and negative) which show high voltage, as shown in Figure 2.
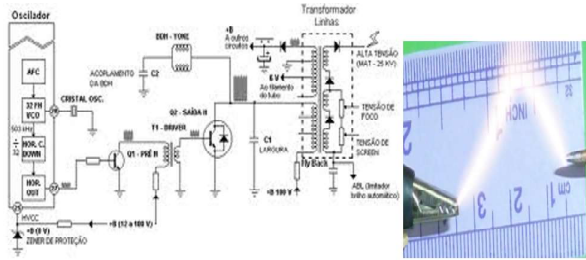

Figure 2. TV electronic circuit with Flyback and Flyback output voltage with fire-shaped electron jumps.

By giving a large enough electric plane to the material, dipoles will appear which leads to the direction of the field, giving rise to total polarization. Conversely, if the temperature returns to the Curie temperature, the electric dipoles automatically disappear again. Therefore, the polling temperature will have a major effect on the polling process itself. In this experiment, the polling temperature was far below the PZT currie temperature of 450 °C.

In the polling process, the sample is placed between 2 electrodes and then dipped in a silicone oil solution. Furthermore, polling is done with a polling temperature of around 100 °C, and the difference in polling time (15 and 25 minutes). [12]

RESULTS AND DISCUSSION

Synthesis of PZT Piezoelectric Material

Samples made by compaction at 5000 and 7000 psi were 1.5 cm in diameter and then followed by the sintering process at 950 °C for 4 hours, the results showed a color change to whitish orange. For this reason, it is necessary to identify or test this synthesis product using an X-Ray Diffraction (XRD), so that the compounds formed by the crystal system can be analyzed. This analysis is also determined by the profile of the x-ray diffraction pattern for each sample, as shown in Figure 3. The samples resulted from the synthesis have shown sharp peaks, indicating that material crystallization has occured. Whereas the PVDF material as a mixing material from PZT product (used in the polling process) was also identified using XRD, as shown in Figure 3. However, synthesis of PVDF material was not carried out. The XRD profile of PVDF material has the same diffraction pattern as the study conducted by Zhang De-Qing [13].
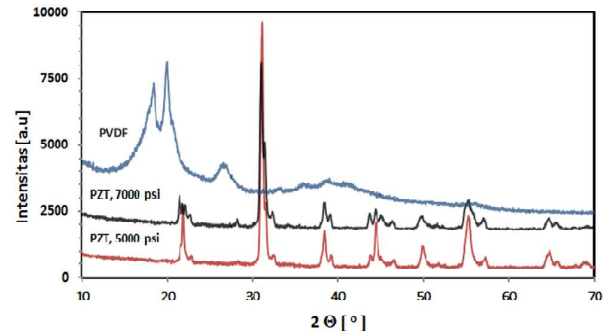

Figure 3. X-ray diffraction patterns of PZT synthesis products with compaction of 5000 and 7000 psi and PVDF materials.

The intensity peaks for samples at 7000 and 5000 psi have a pattern very identical to PZT peak, as studied by Ahda [6]. This indicates that the synthesis process has been successfully carried out. Compaction changes for 5000 and 7000 psi in PZT sample also indicate a change in the intensity and accompanied by a change in Full Width of Half Maximum (FWHM).

The XRD pattern profile of each sample clearly shows differences, especially at angles between 42.5-47.5°, where that of 7000 psi has 4 peaks, and that of 5000 psi has 3 peaks. Therefore it is necessary to analyze the crystal structure using the refinement/fitting process of Rietveld method. Generally, the peaks of PZT piezoelectric material have a perovskite crystal system with a tetragonal and rhombohedral phase with reflection planes shown in Figure 4. This also refers to the XRD data set of standard materials called The Joint Committee on Powder Diffraction Standards (JCPDS) numbers 33-0784 (tetragonal) and 73-2020 (rhombohedral), as also referred to by Rashmi Gupta [14] in identifying PZT and PZT-NiZn powders.
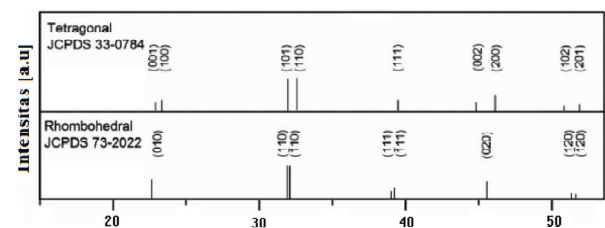

Figure 4. Reflection planes of tetragonal (JCPDS 33-0784) and rhombo-hedral (73-2022) perovskite crystal system.

The presence of peaks at an angle of around 30° represents planes (101) and (110) for the tetragonal and (110) and (ī10) phases for rhombohedral with the highest intensity, also corresponding to the synthesis results.

The initial refinement process was carried out using the HighScore PAN-Analitical software by inputting 2 phases (PZT) with space groups from the tetragonal (P4mm) and rhombohedral (R3c) perovskite crystal systems. The results of the initial refinement/fitting did not provide good performance with errors

(error rate) on The weighted R-factors (R_{wp}) dan the expected R-factor (R_{exp}) which is still high. Thus, a strong indication of another phase (as mentioned above) is indicated by the presence of 4 peaks in the angle range of 42.5-47.5° which indicate 1 other peak. Whereas for 2 phases only have 3 peaks with planes (002)T and (200)T for tetragonal and (200)R for rhombohedral.

Taking into account the presence of another phase, namely $PbTiO_3$, it is used as an additional input parameter in the fitting process with the P4mm space group. Finally, the subsequent fitting process becomes 3 phases by means of zero point, background shifts, peak profile variations (FWHM), and preferred orientations from material and unit cell parameters. The results of the fitting process for PZT samples with 5000 psi compaction obtained $R_{wp} = 5.4998$ %, $R_{exp} = 3.9756$ % with goodness of fit (gof) of 1.91, while for material samples with 7000 psi compaction, obtained $R_{wp} = 5.1157$, $R_{exp} = 4.2371$ % with gof = 1.45774. With the figures of low errors, the fitting process is reasonably good.

Fitting results for several peaks are shown in Figure 5 with angles between 30-33° and 42.5-47.5°. Figure

Figure 5. Profile of XRD peaks of PZT material with compaction of 7000 and 5000 psi at angles between 30-33° and 42.5-47.5°, and the results of the refinement process, where Iobs is the experimental intensity while Icalc is the total intensity of refinement results, Itetra is the intensity of the tetragonal phase, Irhombo is the intensity of rhombohedral phase, IPbTiO is the intensity of $PbTiO_3$ phase and diff is the difference between the experiment intensity and the total intensity of the refinement.

5 shows 3 phase types (tetragonal PZT phase in blue, rhombohedral PZT in green and $PbTiO_3$ in yellow) with each peak profile. The combined intensity of each phase from the fitting results is represented by total intensity (Icalc) colored red. This total intensity is very close to or coincident with the experiment intensity (Iobs, round red). This highly supports the success performance of this fitting process, as it is also related to the results of a fairly low error.

The experimental intensity and total (fitting results) for a sample of 7000 psi showed lower than 5000 psi. The presence of $PbTiO_3$ phase can be clearly indicated at an angle of 46.7° with very low overlapping of the tetragonal and rhombohedral phases.

Intensity of tetragonal phase (blue) for compaction samples at 7000 psi is higher than that of 5000 psi, while rhombohedral phase (green) at 7000 psi is lower than 5000 psi.

Each of the phases above shows the high difference/extent of the peak profile and also affects the content of each phase at the same time. For samples with 7000 psi compaction containing 56.3% tetragonal phase, 22.9% rhombohedral and 20.8% $PbTiO_3$, while for 5000 psi contained 32.8% tetragonal phase, 51.9% rhombohedral and 15.3% $PbTiO_3$. This shows that compaction, if raised in the synthesis process, indicates that the perovskite crystal system with the tetragonal phase increases and reduces the rhombohedral phase. With the tetragonal and rhombohedral phases which have not been 50:50% for the two samples, their presence is not in the MPB area. As expressed by B. Jaffe, the highest material performance indicated by the coupling factor and piezoelectric constant is also the highest in the MPB area [14-15].

The atomic occupancy factor in a crystal system is one of the outputs of a fitting process representing compounds from each sample. Therefore, 3 phases entered in this fitting process give the following compounds; $PbZr_{0.52}Ti_{0.48}O_3$ with P4mm spacegroup, tetragonal crystals, $PbZr_{0.58}Ti_{0.42}O_3$ with R3c space group, tetragonal crystals and $PbTiO_3$ with P4mm space group, tetragonal crystals. For more details, Table 1 shows the results of the fitting process.

Compaction increase means that the contact surface area between particles is larger, so that it can improve the diffusion process between particles of the base material during the sintering process. The diffusion process occurs as particles move randomly from areas with high to lower concentrations. Based on variations in compacting pressure this can affect the diffusion rate of each atom. Higher pressure samples have higher free energy, so that the atomic diffusion rate will occur faster with stored energy [17]. Therefore this diffusion rate greatly affects the synthesis product itself. Synthesis using solid state reaction method with sample grinding for 4 hours, sintering temperature of 950 °C for 4 hours and compaction of 7000 and 5000 psi respectively, has
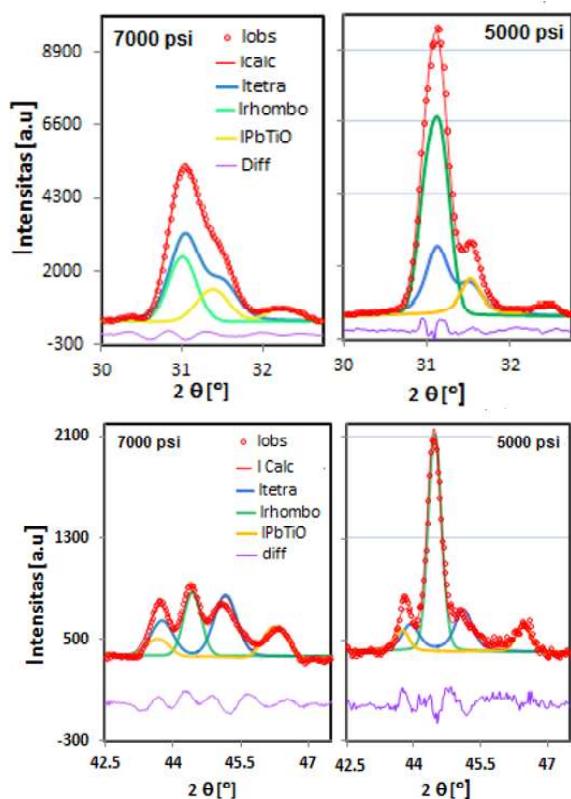
Table 1. Results of the fitting process of synthesis products using XRD data at a room temperature

- Sample with 7000 psi compaction

Phase	Lattice Parameter	Element	Atom Coordinate		
			x	y	z
Tetragonal PbZr$_{0.52}$Ti$_{0.48}$O$_3$	Space group = P4mm a = b = 4.0149 Å c = 4.1392 Å c/a = 1.0309	Pb Zr Ti O(1) O(2)	0.5 0 0 0 0	0.5 0 0 0 0	0.447 0 0 0.580 0.556
Rhombohedral PbZr$_{0.58}$Ti$_{0.42}$O$_3$	Space group = R 3 c A = b = c = 4,0791 Å α = 89.83°	Pb Zr Ti O(1)	0 0 0 0,814	0 0 0 0,186	0.514 0 0 0.148
Tetragonal PbTiO$_3$	Space group = P4mm a = b = 3.9253 Å c = 4.1469 Å c/a = 1.0564	Pb Ti O(1) O(2)	0.5 0 0.5 0	0 0 0 0	0.078 0 0,078 0.575

- Sample with 5000 psi compaction

Phase	Lattice Parameter	Element	Atom Coordinate		
			x	y	z
Tetragonal PbZr$_{0.52}$Ti$_{0.48}$O$_3$	Space group = P4mm a = b 4.0311 Å c = 4.134 Å c/a = 1.0255	Pb Zr Ti O(1) O(2)	0.5 0 0 0 0	0.5 0 0 0 0	0.447 0 0 0.124 0.541
Rhombohedral PbZr$_{0.58}$Ti$_{0.42}$O$_3$	Space group = R 3 c a = b =c= 4.0874 Å α = 89.70°	Pb Zr Ti O(1)	0 0 0 0.814	0 0 0 0.186	0.511 0 0 0.148
Tetragonal PbTiO$_3$	Space group = P4mm a = b = 3.9202 Å c = 4.1503 Å c/a = 1.0586	Pb Ti O(1) O(2)	0.5 0 0.5 0	0.5 0 0 0	0.456 0 0.031 0.622

not produced a perfect product PbZr$_{0.52}$Ti$_{0.48}$O$_3$. The reaction of this synthesis process also produces PbZr$_{0.58}$Ti$_{0.42}$O$_3$ and PbTiO$_3$. So we can say that these three compounds become a product with the parameters mentioned above.

Compaction changes can affect Full Width of Half Maximum (FWHM) for the profile of each phase peak, as shown in Figure 3. This can indicate changes in the grain size that occurs in the synthesis product. FWHM is smaller, indicating the magnitude of the particle increases, and vice versa [18]. One of FWHM magnitudes for 7000 psi in the phases of tetragonal plane (002) and the rhombohedral plane (020) is 0.5591 and 0.4351° respectively, while for 5000 psi of the same phases and planes are 0.4318 and 0.3256°. This shows that compaction of 7000 psi shows that FWHM is greater than 5000 psi, thus representing the amount of tetragonal and rhombohedral phase crystal grains for 7000 psi smaller than 5000 psi.

The influence of compaction can also be seen from changes in phase cell units that are formed. Comparison of unit cell c/a parameters from

PbZr$_{0.52}$Ti$_{0.48}$O$_3$ (tetragonal) phase between 7000 psi samples and 5000 psi also decreases i.e. 1.0309 and 1.0255, while for PbTiO$_3$ (tetragonal) phase, the magnitude is 1.0564 and 1.0586. As for PbZr$_{0.58}$Ti$_{0.42}$O$_3$ (rhombohedral) the α angle of the cell unit changes from 89.83 to 89.70°, if compaction changes from 7000 to 5000 psi.

PZT Material Polling

The function of Flyback TV in this study is to provide a high voltage supply to two polling electrodes. In principle, the circuit consists of a low voltage provider, oscillator and amplifier and flyback voltage, as shown in Figure 2. Generally a black and white, 14 inch Flyback TV has 14 kVDC with a current of 10.5 mA.

The output from Flyback is a DC voltage indicated by a diode as a rectifier [19,20]. DC high voltage measurement, as a flyback output, needs a special circuit design, so that it can be measured using an ordinary voltmeter measuring device. In Winconsan TV series used, the voltage after flyback is around 12 kV DC.

Sometimes the voltage is less stable, possibly due to the voltage of the grid from PLN to fluctuate at certain times. In order to be able to use a sample, a voltage of around 4 kV DC is required. Therefore, a divider design has been made to reduce the voltage, as shown in the Figure below.

Figure 6 above is a design of a dividing circuit by providing resistance R = 500 MΩ and the output voltage to the polling device is expected to be 4 kV DC. However, the measured voltage fluctuates around 3.5 - 4 kV DC. This fluctuation is very possible from the resitances of 2M contacted by the series to reach 500 MΩ which has a considerable error, besides the voltage of the PLN fluctuating as well.
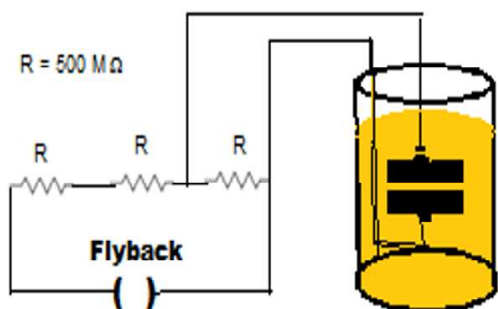

Figure 6. Voltage devider circuit with scheme of the polling process.

At the beginning of the polling process, a short connection between positive and negative electrodes has been checked, between the surface of the sample material, so as to minimize the occurrence of polling failure or potential explosion. For the first step, PZT material polling with different compaction (5000 and 7000 psi) is carried out and the temperature parameter is set to 100 °C. Each sample with a thickness of between 1.91 - 2.02 mm (average 1.97 mm) was placed between the electrodes in the silicon oil environment. The function of silicone oil is as a separator between the part with voltage or insulator and also as a cooler [19].

Determination of the polling success is tested in the measurement of the piezoelectric constant (d33) using a d33 meter measuring instrument, as shown in Figure 7.

To see the effect of the polling process, first the PZT sample material that still has random dipole domains (without polling) d33 values is measured at 5 x 10^{-12} C/N. However, post polling samples show a significant increase (about 9-12 times), as shown in figure 7. With a polling time of 15 minutes and 25 minutes with a polling temperature of 100 °C, the sample compacting of 7000 psi has a d33 higher than 5000 psi. Whereas the same compacted sample indicates a greater d33 value at the polling time of 15 minutes than at 25 minutes. This means that compaction of 7000 psi, 15 minutes and 100 °C are the best parameters in getting the highest d33, 61 x 10^{-12} C/N.
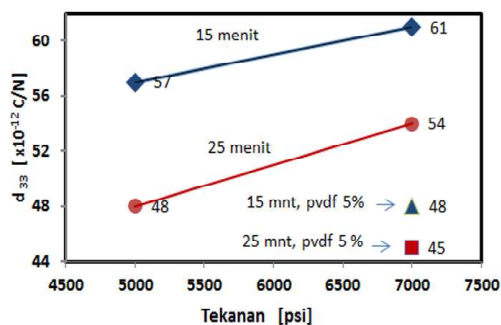

Figure 7. Measurement chart of d33 for PZT samples with a temperature of 100 °C and polling time of 15 and 25 minutes in compacted 5000 and 7000 psi samples, accompanied by addition of 5% PVDF.

As mentioned above, compaction of 7000 psi has smaller PZT particles and has an effect on increasing piezoelectric constants, d33. As happened in CaZrO$_3$-modified (K, Na) NbO$_3$ material) studied by Fang Zhou Yao [20] that the influence of crystal structure on piezoelectric properties d33 happens. It can also be indicated in this study that the different crystal grains due to compacting changes in PZT have an effect on d33. Although the increase in d33 is quite considerable from 5 to 61x 10^{-12} C/N (before and after polling), however the polling process parameters are still not optimal, considering other parameters that need to be varied, such as temperature, sample thickness, polling pressure and optimal polling time. In this process, initial polling technique has succeeded as an important activity in preparing piezoelectric materials that are ready to be applied.

The above parameter application was tested on PZT samples with the addition of 5% by weight of the compressed PVDF 7000 psi to the influence of polling time. The measurement results of d33 from the polling sample using a polling time of 15 minutes is 48 x 10^{-12} C/N higher than the time of 25 minutes at 45 x 10^{-12} C. This shows that 15 minutes is quite optimal in this polling process, both on PZT and PZT + PVDF samples.
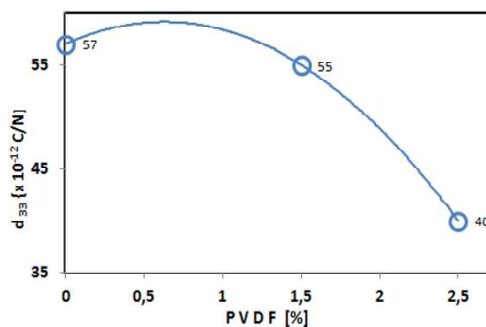

Figure 8. d33 Measurement in PZT + PVDF material for PZT sample with 5000 psi compaction.

Figure 8 shows the d33 measurement results in a sample of 5000 psi compacting variations of PVDF addition. The more addition of PVDF, the lower the value of d33.

While the highest d33 value on PZT (no PVDF added) is 57 x10^{-12} C/N. The decrease in d33 was also carried out by Zhang de Qing [13], namely the PVDF addition with a volume fraction on PZT which had an effect of decreasing d33, both by hot press and cold press.

In this initial polling process, the highest d33 (61 x 10^{-12} C/N) measured has not been at maximum value, as found in the measurements made by Q.Guo of 374 x 10^{-12} C/N [23]. Therefore, this polling has not yet obtained an optimal parameter, due to the number of polling parameters themselves (such as temperature, sample thickness and DC KV voltage). Besides that the PZT material has 3 phases (tetragonal, rhombohedral and PbTiO$_3$ phase), while the best composition is in the MPB area. The MPB area represents the existence of 2 phases (tetragonal and Rhombohedral) with the same content, as indicated by Jafe [15].

CONCLUSION

The synthesis of PZT material using solid state reaction method has been successfully carried out with the following parameters, grinding the base material for 4 hours, sintering at a temperature of 950 °C for 4 hours. Crystal structure analysis in synthesized products using the fitting process (Highscore program) shows the tetragonal perovskite crystal system with the composition of PbZr$_{0.52}$Ti$_{0.48}$O$_3$ (P4mm spacegroup, and rhombohedral PbZr$_{0.58}$Ti$_{0.42}$O$_3$ (R3c space group), as well as the presence of other tetragonal phases PbTiO$_3$. Tetragonal and rhombohedral PZT phase content and PbTiO$_3$ for 7000 psi compaction were 56.3%, 22.9% and 20.8%, while for 5000 psi were 32.8%, 51.9% and 15.3% respectively.

This shows the greater compaction, the greater the tetragonal phase content and vice versa for the rhombohedral phase, with this content showing the composition not in the MPB area, but giving an effect on the polling results of the synthesis results.

The use of Flyback TV to obtain high voltage is quite feasible to use in the piezoelectric material polling process along with the application of a voltage divider electronic circuit, in order to obtain a voltage of around 4kV DC.

The initial PZT material polling process has been successfully carried out in 100 °C silicone oil for 15 minutes and 25 minutes. d33 measurement without polling was obtained by 5 x 10^{-12} C/N, while the poll results were quite significant with the highest increase of 61 x 10^{-12} C/N for a sample of 7000 psi. Increasing PVDF in PZT material shows a decrease in d33 value.

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