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DEVELOPMENT OF PIEZO-SMART MATERIALS AND DEVICES

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ABSTRACT

Higher forms of materials, which can perform functions such as sensing, actuating, control and intelligence, are termed as "Smart Materials". Some examples of the smart materials are piezoelectric materials, Shape Memory Alloy (SMA), electro-rheological fluids, magneto-strictive materials etc. Among all these active materials piezoelectric materials are most widely used because of their fast electromechanical response, low power requirement and relatively high generative forces. Piezoelectric ceramics are widely used for various applications such as actuators and sensors, sonar transducers, ultrasonic motor, accelerometers, in drug delivery systems etc. These materials can be prepared with a wide range of properties by varying the compositions with different dopants. In NAL, PZT powders of 5A and 5H grade were prepared under National Programme on Smart Materials (NPSM). The powders were processed by wet chemical technique. Compositions near Morphotropic Phase Boundary (MPB) were studied by varying Zr/Ti ratio as well as the effect of different dopants such as lanthanum, neodymium and their combinations. The powders were characterized for ferroelectric, piezoelectric and dielectric properties. Few Multi-layered (25 layers) stacks were fabricated by using the above powders. The displacement and the free strain of the actuators were evaluated. A maximum displacement of 20μm and free strain of 0.1% was obtained at 650V.

Keywords: Lead zirconate titanate, morphotropic phase boundary, wet chemical, hysteresis loop, Multilayered stack.

1. INTRODUCTION

Lead Zirconate Titanate (PZT) is an important material used for "smart" applications i.e. for both sensing as well as for actuating purpose. It is a solid solution of lead zirconate ($PbZrO_3$) and lead titanate ($PbTiO_3$). It possess excellent piezoelectric properties such as high piezo-electric charge constant (d_{33}), high coupling coefficient, high dielectric constant etc. and are used as actuators and sensors, as sonar transducers, as accelerometers, in inkjet printers etc. The highest piezoelectric coupling coefficients as well as maximum permitivity are obtained for compositions near morphotropic phase boundary (MPB) which fall in between the tetragonal and rhombohedral phases [1-3]. The properties of PZTs are further enhanced by addition of suitable dopants. The powders are generally prepared by various methods like mixed oxide, sol-gel, co-precipitation, spray drying, hydrothermal synthesis etc. Synthesis of PZT powders by conventional "mixed oxide" route requires a fairly high sintering temperature which causes significant loss of lead oxide [4]. Other disadvantages of this process are compositional inhomogeneity, impurity pickup during milling and large particle size of the powder. All these factors degrade the properties. Therefore, several chemical methods are followed for preparation of PZT powders, which generate very fine, stochiometric,

homogeneous powders [5-9]. Among these methods wet-chemical, co-precipitation [9,10] and sol-gel [11] techniques are most popular.

In this paper, the properties of PZT-5H and PZT-5Apowders prepared in NAL and the technology of fabrication and characterisation of multi-layered stacks are presented. The powders were prepared by wet-chemical method to achieve compositional homogeneity, stoichiometry and fine particle size distribution which is necessary to obtain better properties. Few multilayered stacks were fabricated and the percentage free strain was also measured.

2. EXPERIMENTAL PROCEDURE

Analytical grade lead nitrate (99.5%) zirconium oxychloride (99.9%), titanium tetrachloride(99%) and lanthanum nitrate (99.9%) were used as starting chemicals for the preparation of PZT powders. The combined salt solution with 3% excess lead along with dopant solution were converted into a mixed hydroxide by increasing the pH between 7.5-8.5 by adding dilute ammonia solution. The hydrous precipitate was dried and calcined in the temperature range of 750-900°C for 1-4 hours. After calcination, the powders were de-agglomerated by grinding and PZT phase formation was confirmed by X-Ray Diffraction (XRD) technique (M/s. Phillips, Holand). The average particle size d_{50} of the calcined powders were analyzed using the particle size analyzer (Sedigraph 5100, M/s. Micromeritics, USA). The calcined powders were granulated using 2 wt. % polyvinyl alcohol (PVA) solution, uniaxially pressed into pellets and sintered in the temperature range of 1050-1250°C for 2h in a closed lead rich atmosphere to minimize lead oxide loss. Sintered pellets were leveled, polished and electroded with silver paste and were poled in a dc field of 2kV/mm in a silicone oil bath for 30 minutes and the linear piezo-electric strain coefficient (d_{33}) was measured using a piezo-meter (Model PM-35, M/s.Take control, UK).

3. RESULTS AND DISCUSSION

The particle size distribution curve of calcined, PZT powders is shown in **Fig.1**. The curve indicates a narrow size distribution of the particles and d_{50} was found to be 1μm. A typical SEM picture of chemically etched PZT sintered pellet is shown in **Fig.2**. It is observed from the micrograph that grains are almost uniform in size in the range of 1-2μm.Variation of sintered density with temperature is shown in the F**ig 3.** It is observed that the density of the samples gradually increases with increase in the sintering temperature and a maximum density of 98% of theoretical value is obtained from the sample sintered at 1250°C.

3.1 Piezoelectric and dielectric properties:

The variation of piezoelectric and dielectric properties with respect to temperature is shown in F**ig.4** and **Fig.5** respectively. From **Fig.4** it is observed that the d_{33} value increases with increasing sintering temperature. A maximum d_{33} value of 598 pC/N is obtained at 1250°C. The maximum value of d_{33} can be co related to the sintered density of the samples i.e. greater the sintered density better is the piezoelectric properties. Due to better sintered density at higher sintering temperature the samples are less porous and there is a good contact among the grains, which enhance the dipole orientation during poling and consequently increases the piezo properties. From F**ig.5,** it is seen that the dielectric constant increases with increase in the sintering temperature up to 1200°C and slightly decreases at 1250°C.

4. Fabrication of multilayered stacks

Multi-layered (25layered) PZT stacks were fabricated using the above powder(Fig.7). The powders were consolidated into blocks (26x21x14 mm^3) and sintered in a lead rich atmosphere to get finally a dense sintered block. These blocks were cut into thin strips (18x 12x0.55 mm^3). After leveling, polishing and electroding the strips were poled and their piezo properties were evaluated. Strips with almost equal d_{33} values were selected for fabrication of multi layered stack. Using a strain gauge sensor the static displacement of the actuator was measured to be 20 μm at 650 Volts. The induced strain was found to be 0.1%.The plot of the displacement Vs applied voltage is almost linear (Fig.8). The operating voltage can be brought down significantly by reducing the thickness of the layers.

5. CONCLUSIONS

PZT-5A and PZT-5H grade powders were successfully synthesized at NAL. Few multilayered stacks were fabricated from these inhouse prepared powders and their performance were evaluated. The free strain was measured by applying dc voltage up to 650V. The maximum displacement was found to be 20μm and the free strain was 0.1% .

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TABLES

Table-1: Properties of NAL PZT-5A and 5H grade powders

PROPERTIES	NAL-5A	NAL-5H
Density (gm/cc)	7.6-7.7	7.6-7.7
Piezoelectric charge constant d_{33} (pC/N)	375-430	590-610
Relative dielectric constant (K)	1250-1330	1700-1790
Dissipation Factor (tanδ)	0.025-0.042	0.025-0.035
Particle size (median diameter, d50) µm	0.6-1.2	

Table -2: Sintering study of NAL-5H powder

Sample	Sintering Temperature / soaking time.	Sintered Density (%)	d_{33} (pC/N)	Capacitance (pF)	Dielectric constant (K)	Tanδ
PZT-5H	1100°C /2hrs	91	350	836	1590	0.0520
	1150°C /2hrs	94.8	491	834	1817	0.0364
	1200°C /2hrs	97.5	537	894	1918	0.0245
	1250°C /2hrs	98	598	873	1701	0.0228

FIGURES

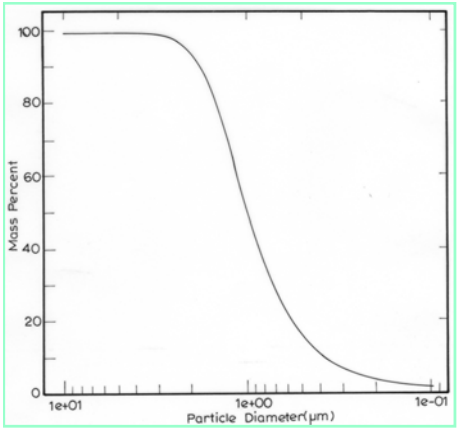

Fig.1. A typical particle size distribution of calcined PZT powder

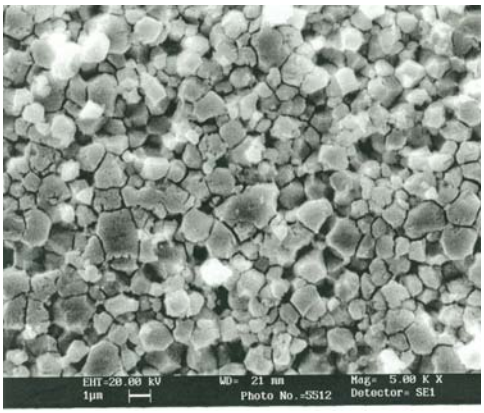

Fig.2. SEM photograph of sintered PZT body

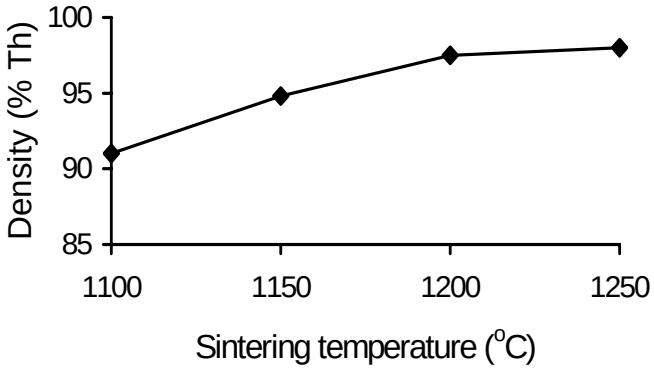

Fig.3: Variation of density with sintering temperature

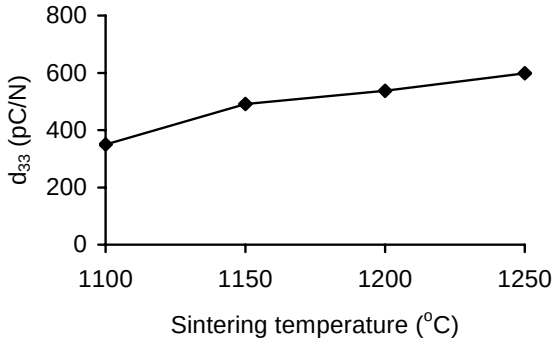

Fig.4: Variation of d_{33} with increase in temperature

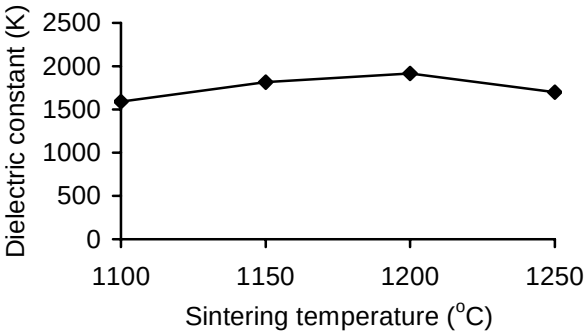

Fig.5: Variation of dielectric constant with increasein temperature

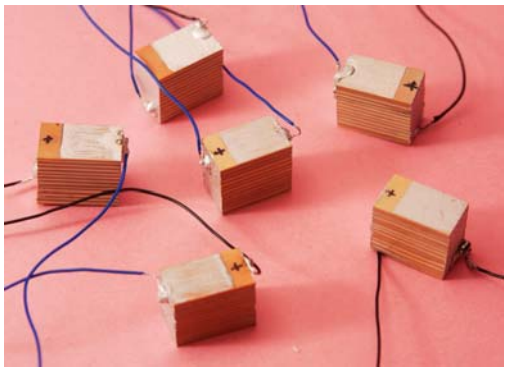

Fig.7. Picture of PZT multilayered stacks

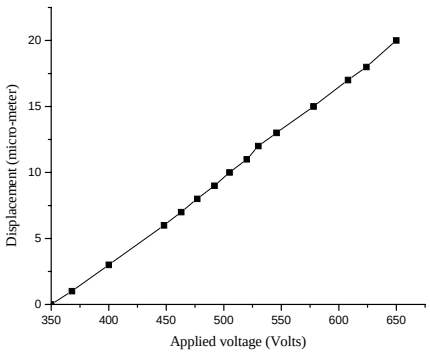

Fig.8. Plot of displacement Vs.Applied voltage.