

The Research on the Microstructure and Electrical Properties of Nb₂O₅ Doped PBSFCZT Ceramics

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Abstract. In order to improve the electrical properties of PZT piezoelectric ceramic material, the effect of sintering temperature on the microstructure and electrical properties of the Nb₂O₅ doped PBFSZT ceramic material. The crystalline phase and microstructure of the PBSFCZT+0.5% Nb₂O₅ ceramic were analyzed at the different sintering temperatures (1120°C, 1150°C, 1180°C and 1210°C) by x-ray diffraction (XRD) and scanning electron microscopy (SEM) respectively. The results of the experiment show that the amounts of the Nb₂O₅ was 0.5%, the grain size of the PBSFCZT piezoelectric ceramic materials decreased correlated with the increasing of the sintering temperature. When the sintering temperature at 1180°C, the grain growths well and the grain-boundary intersection in tight, an excellent comprehensive electrical properties of the PBSZT ceramics material are as follows : $\epsilon=3626$, $\tan\delta=1.7$, $d_{33}=478\text{PC/N}$, $K_p=0.669$. These results proved that appropriate softness doping can decrease the sintering temperature and improve the piezoelectric properties of the PBSFCZT ceramic material.

Introduction

With the development of the high technology, the different country researchers focus on the new material technology. Since 1942, the first piezoelectric ceramic BaTiO₃ has been invented, the research on the piezoelectric ceramic become more and more important. Because of the good mechanical properties and reliability piezoelectric properties of the piezoelectric ceramics, it has been regard as an important mechanical, heat, light sensitive function material. It has been widely used in the sensors, acoustic transducer, micro-displacement device and other electric components, etc [1,2]. In present, there are two major methods to improve the properties of the piezoelectric ceramics: one is conventional mixture oxide doping, another one is technology improvement. In this paper, the effect of the sintering temperature on the microstructure and properties of Nb₂O₅ doped PBSFCZT ceramics has been researched.

Experimental Procedures

In this experiment, the $\text{Pb}_{1-x}\text{Ba}_x[(\text{Sb}_{1/2}\text{Fe}_{1/2})_{1/2}\text{Ca}_{1/2}]_{0.02}(\text{Zr}_{53}\text{Ti}_{47})_{0.98} + 0.5\%\text{Nb}_2\text{O}_5$ are prepared by a conventional mixed oxide sintering method. The reagent grade raw powders of Pb₃O₄, BaCO₃, Sb₂O₃, Fe₂O₃, ZrO₂, TiO₂, Nb₂O₅, CaCO₃ are used as starting materials. And then the powders are weighed according to stoichiometric ratio. The powders mixed by ball milling for 16 hours with 99% alcohol. After ball milling, the mixture is pre-sintered at 860°C for 4 hours. After that, the obtained oxide powder was ball milling again. After complete the previous processes, the powder was formed into $\text{Ø}12.88\text{mm} \times 1.2\text{mm}$ plates with 7wt% paraffin. Then the plates were subjected to a process of debinding at 700°C for 500 minutes and then sintered at 1120 oC, 1150 oC, 1180oC and 1210oC for 2h respectively. Finally, the ceramic plates are coated with sliver electrodes on both sides and polarized at 75 oC in silicon oil bath by applying DC electric fields at 2.3KV/mm for 30min. The polarized ceramic plates are then tested with dielectric loss ($\tan\delta$), electromechanical coupling coefficient (k_p), piezoelectric constant (d_{33}) and dielectric constant (ϵ) after aging at room temperature for 24h.

The phase structure of the samples was analyzed by X-ray diffraction(XRD) and the fractured surface were observed by scanning electron microscopy(SEM). The piezoelectric constant d_{33} was measured by quasi-static d_{33} meter(ZJ-6A,China).The electromechanical coupling coefficient K_p and the electromechanical quality factor Q_m were calculated from the resonance and antiresonance frequencies using precise impedance analyzer(HP4194, Angilent).

Results and Discussion

Analysis of Electrical Properties

The electrical properties (dielectric loss ($\tan\delta$), electromechanical coupling coefficient(K_p), piezoelectric constant (d_{33}) and dielectric constant (ϵ)) of PBSFCZT samples with the sintering temperature changed from 1120°C to 1210°C for 2h are listed in Tab.1.

Tab.1 The electric properties of PBSFCZT samples sintered at 1120°C--1210°C

Samples	Temperature	c	ϵ	f1	f2	kp	d33	$\tan\delta$
1	1120°C	2346	5646	181.1	235.2	0.723	652	1.6
2	1120°C	1192	2915	183.9	234.2	0.701	426	1.8
3	1150°C	1457	3231	180.8	227.3	0.686	467	1.6
4	1180°C	1602	3625	182.8	226.6	0.669	478	1.7
5	1210°C	1498	3194	182.8	220.8	0.634	438	1.7

From the Tab.1, it has been found that the samples of the PBSZT ceramics have the same electrical properties when sintered at the same temperature. But when the samples sintered at 1120°C, there is some difference in the samples, sample 1 have an good electric properties, the sample 1 posses the satisfied electrical properties not only in the dielectric loss, electromechanical coupling coefficient, piezoelectric constant and dielectric constants, which are as follows: $\tan\delta=1.8\%$ $K_p=0.723$, $d_{33}=652\text{pC/N}$, $\epsilon=5646$, the electrical properties higher than other samples about 30%. The electrical properties of the samples gradually improving with the sintering temperature increased. When the samples sintered at 1180°C, the PBSFCZT samples have an good electrical properties: $\tan\delta=1.7\%$ $K_p=0.669$, $d_{33}=478\text{pC/N}$, $\epsilon=3625$.

Analysis of XRD

Fig.1 shows the X-ray diffraction (XRD) patterns of PBFSZT doped 0.5% Nb_2O_5 ceramic samples at different sintering temperature(1120°C,1150°C,1180 °C and 1210°C) for 2h.

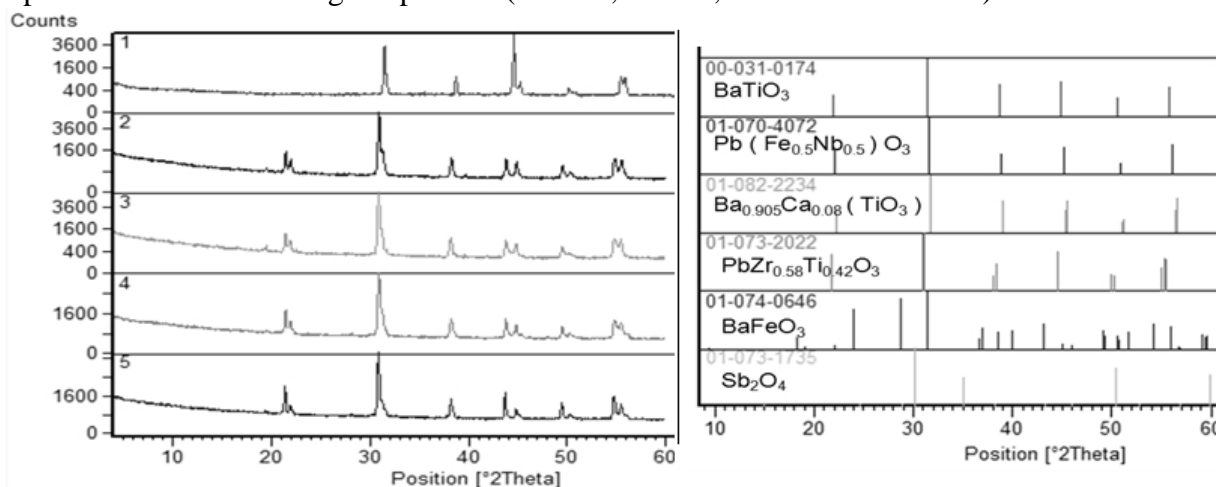


Fig.1 The XRD images of the PBSFCZT samples from 1 to 5

It can be found from the Fig.1, a comparative pure perovskite structure was obtained, but at the same time some pyrochlephases were found. From the samples 2 to 5, there are some BT, PFN,

PZT, BCT, BF and Sb_2O_4 phases. But different from the the other samples, the sample 1 has no obvious double diffraction peak around the 21° ; this is because the sample 1 has no BF phase exist. But the diffraction peak of the secondary phase gradually disappeared with the increased of the sintering temperatures, the electrical properties of the sample will become better[3,4]. The scanning regions range from 10° to 60° .

Analysis of SEM

Fig.2 is the SEM photography of the PBSFCZT doped 0.5% Nb_2O_5 ceramic samples sintered at different sintering temperatures($1120^\circ C$, $1150^\circ C$, $1180^\circ C$ and $1210^\circ C$).

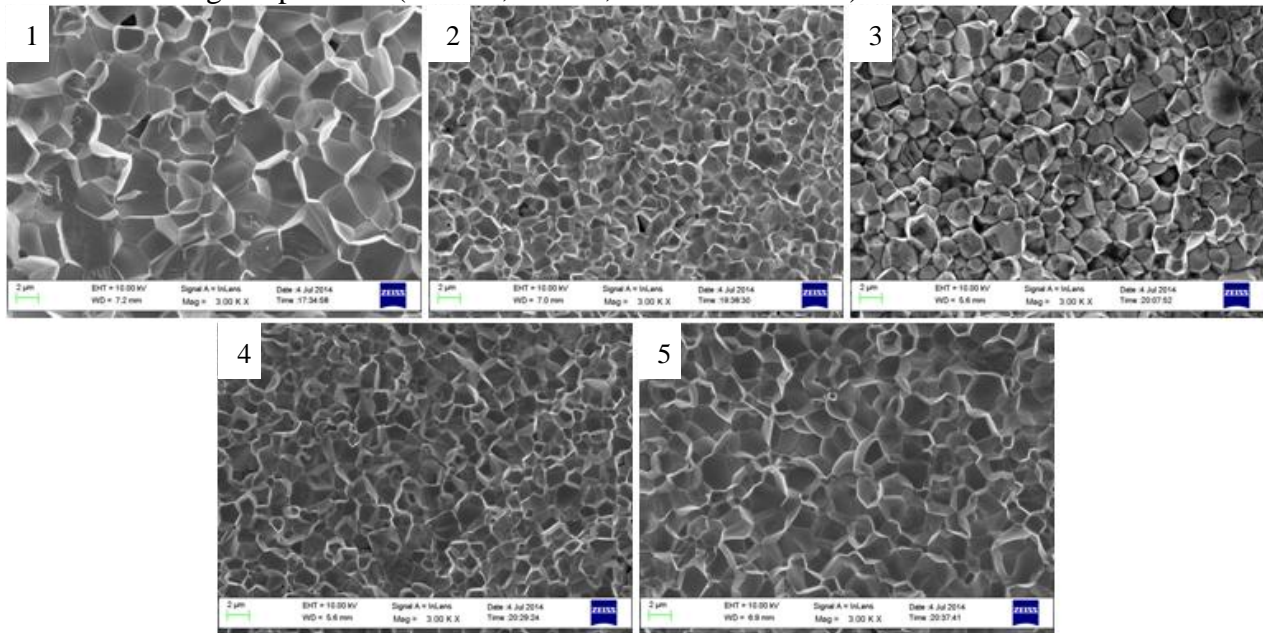


Fig.2 The SEM images of the PBSFCZT samples from 1 to 5

From the Fig.2, it can be seen that the grain size of sample 1 is obvious bigger than other samples. The grains size of the ceramic sample from 2 to 5 have a significant change with the sintering temperature vary from $1120^\circ C$ to $1210^\circ C$, the grain size of the samples increased with the increasing of the sintering temperature. Sample 5 exhibit the largest grain size and are over developed when sintered at $1210^\circ C$. Wang xiaohui et al[5] research shows that, with the size of grain change, the cell parameters also change related to grain size. Furthermore, that will influence the electric properties of the samples. Experiment results shows that, when sintered at $1180^\circ C$, grains grows well, the intersection between grain and grain combined well, there is no segregation phase appearance. Correlated to electrical properties results, the sintering temperature have an obvious influence on the piezoelectric ceramic. With the increasing of sintering temperature, the electric properties first increased but after gradually decrease. The Nb_2O_5 doped in PBSFCZT samples which can make the samples sintered easier[6]. The decrease of grain size can cause the increase of dielectric constant of samples. The reason of sample 1 have an excellent comprehensive electrical properties, because of the sample in the sintering furnace location is not same, which contribute to the obvious difference.

Conclusion

The influences of different sintering temperatures on the microstructures and the electrical properties of the PBSFCZT doped 0.5% Nb_2O_5 ceramic samples are investigated. In this paper, it has been observed that, different sintering temperatures will influence the grain size and electrical properties of the PBSZT doped 0.5% Nb_2O_5 ceramic samples respectively. Through appropriate adjustment of the sintering temperature, the grains size of the PBSZT ceramic samples materials will become fine and the porosity of the bulk material will decrease. when the sintering temperature

is 1180°C, both the electromechanical coupling coefficient and the piezoelectric constant of samples will be improved, which are as follows: $\tan\delta=0.017$, $k_p=0.669$, $d_{33}=478\text{pC/N}$, $\varepsilon=3626$.

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