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Poling Condition Optimization for $CaBi_4Ti_4O_{15}$ /Pb(Zr,Ti)O_3 Sol-Gel Composite

$CaBi_4Ti_4O_{15}$ /Pb(Zr,Ti)O_3 の分極条件最適化に関する研究

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1. Introduction

In recent years, the safety of infrastructure is regarded as an important issue. To improve safety, in addition to periodical off-line non-destructive inspection (NDI), on-line NDI during operation during operation is recommended. One of the main NDI methods is ultrasound NDI using ultrasonic transducers, but it is technically difficult to use ultrasonic trabsducers during operation due to high tempearature.

High-temperature ultrasonic transducers using sol-gel composite materials have been developed for improving high temperature resistance.[1-4)] Sol-gel composite materials are composites made by ferroelectric powders and sol-gel solution. $CaBi_4Ti_4O_{15}$ (CBT) / Pb(Ti,Zr)O_3 (PZT), which consists of CBT powder and PZT sol-gel solution, has been developed for high temperature applications above 500℃.[3)] Normally, polarization of the sol-gel composite is performed by positive DC corona discharge since a high electric field can be applied without dielectric breakdown. However, since CBT powder phase has high coercive electric field, it is necessary to polarize at high temperature to obtain high piezoelectricity. Therefore, it took a long time to polarize due to heating and cooling time. If CBT/PZT can be polarized at RT, manufacturing cost can be reduced and CBT/PZT becomes more practical.

In past study, RT poling of CBT/PZT was attempt using pulse voltage discharge.[4)] It was assumed that pulse voltage could supply higher energy than DC voltage. As a result, RT poling of CBT/PZT itself was successfully accomplished. However, for ultrasonic transducer application, signal to noise ratio (SNR) was not satisfactry at elevated temperatures. Suspected reason was poling conditions were not optimised yet. In this research, RT poling conditions of CBT/PZT was investigated to improve SNR at high temperatures.

2. Fabrication of CBT/PZT films

CBT/PZT sol-gel composite films were fabricated on 3 mm thick titanium substrates by sol-gel spray method. PZT sol-gel solution and CBT piezoelectric powder were mixed. The mixture was ball milled more than one day in order to achieve proper viscosity for spray coating. After the mixture was sprayed onto the titanium substrates, heating process, drying process at 150℃ for 5min and firing process at 650ºC for 5min, was performed. Spray coating and heating processes were repeated until target thickness was achieved. In this research, target thickness was 50μm. Thereafter, 1 cm diameter platinum top electrode was manufactured by commercial platinum paste.

3. Poling process

In this study, poling condition optimization was attempt. In order to supply higher energy and suppress arc discharge, new pulse voltage source machine, which could supply higher voltage and lower current compared with another machine, was designed and developed. The parameters were distance and pulse voltage machine in this time. There were two conditions regarding the distance between the needle and the film surface; 2.0 cm and 2.5 cm. Also, there were two conditions regarding pulse voltage machine; old pulse voltage source (21 kV) and new pulse voltage source (40 kV). Three cases were investigated at this moment as following;

(i) Distance: 2.5 cm, source voltage: 21 kV (old machine)
(ii) Distance: 2.0 cm, source voltage: 21 kV (old machine)
(iii) Distance: 2.0 cm, source voltage: 40 kV (new machine)

4. Experimental results

To evaluate SNR at high temperature, ultrasonic measurement in pulse-echo mode were operated at various tempertures, between RT and 400 °C. Each sample with a silver top electrode was heated by a hot plate. The measurement results was recorded by a digital oscilloscope. The ultrasonic response in time domain at 400 °C for case (i)-(iii) is shown in **Figs. 1-3**, respectively. From these results, it seems that case (iii) had highest SNR at 400 °C.

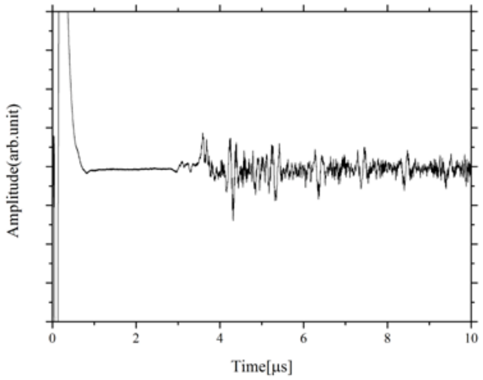

Fig. 1 Ultrasonic response at 400°C of CBT/PZT sample poled by case (i) conditions.

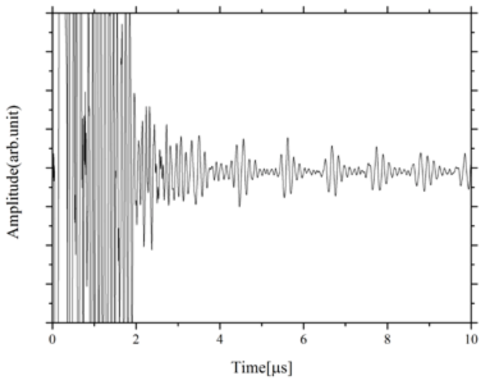

Fig. 2 Ultrasonic response at 400°C of CBT/PZT sample poled by case (ii) conditions.

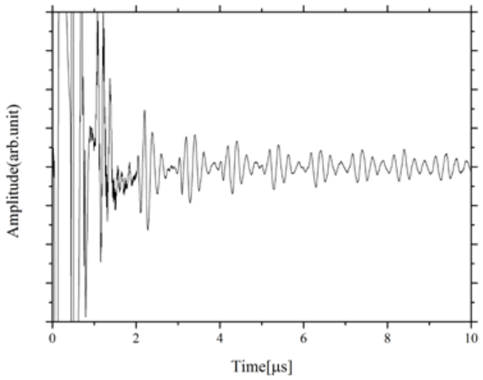

Fig. 3 Ultrasonic response at 400°C of CBT/PZT sample poled by case (iii) conditions.

Since the sample poled by case (iii) conditions could operate at 400 °C satisfactory, that samples was set inside an electrical furnace and high temperature measurement was performed up to 600 °C to investigate SNR at elevated temperature further. The measurement result at 600 °C is shown in **Fig. 4**. At 600 ℃, the SNR was significantly deteriorated even though multiple reflected echoes are still confirmed. Derioration of silver top electrode would affect the signal deteritioration.

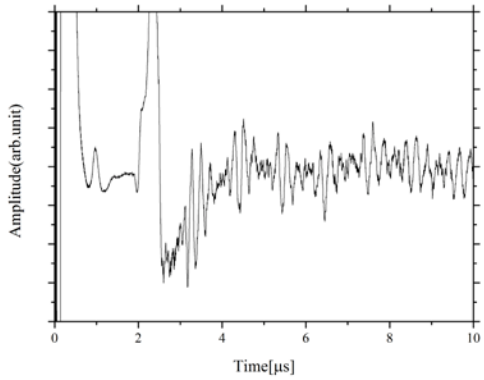

Fig. 4 Ultrasonic response at 600°C of CBT/PZT sample poled by case (iii) conditions.

5. Conclusions

Room temperature poling conditions of CBT/PZT was investigated to imprive SNT at high temperatures. Two parameters were used in this time; distance between needle and CBT/PZT film surface and pulse voltage source machine. Pulse voltage source machine difference was mainly voltage source difference and new machine could supply about twice high voltage, though other conditions, such as current, were also different. As a result, distance and machine difference influenced SNR at elevated temperatures. Further research will be conducted for poling condition optimisation.

References

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