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Development of lead-free single-element ultrahigh frequency (170–320 MHz) ultrasonic transducers

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ABSTRACT

This paper presents the design, fabrication and characterization of single-element ultrahigh frequency (UHF) ultrasonic transducers in which the center frequency ranged from 170 to 320 MHz. The center frequency of >300 MHz is the highest value of lead-free ceramic ultrasonic transducers ever reported. With concern in the environmental pollution of lead-based materials, the transducer elements presented in this work were lead-free $K_{0.5}Na_{0.5}NbO_3/Bi_{0.5}Na_{0.5}TiO_3$ (KNN/BNT) composite thick films. All transducers were evaluated in a pulse-echo arrangement. The measured $-6\,dB$ bandwidth of the transducers ranged from 35% to 64%. With the optimized piezoelectric properties of the composite film, the insertion loss of the UHF transducers was measured and determined to range from -50 to $-60\,dB$. In addition to the pulse-echo measurement, a 6 μm tungsten wire phantom was also imaged with a 205 MHz transducer to demonstrate the imaging capability. The measured $-6\,dB$ axial and lateral resolutions were found to be 12 μm and 50 μm , respectively. The transducer performance presented in this work is shown to be better or comparable to previously reported results even though the frequency is much higher.

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1. Introduction

Ultrasonic imaging is one of the most important medical diagnostic tools used today. Compared to other clinical imaging modalities such as magnetic resonance imaging (MRI), X-ray computed tomography (CT), and nuclear imaging, ultrasound provides images with comparable quality and resolution at low cost without exposing patients to ionizing radiation. With its noninvasive characteristics, the use of ultrasonic imaging is expanding into new clinical applications. High frequency (HF) ultrasound has found many applications in the diagnosis of arterial diseases [1–3], problems in the anterior segment of the eye [4,5], and characterization of skin lesions [6,7] due to its enhanced spatial resolution. It has also played a significant role in small animal preclinical imaging [8,9].

In addition to imaging applications, other applications of HF ultrasound have been investigated. As the frequency is increased to beyond 100 MHz, the beam dimension would approach the cellular level. Ultrasound beams at ultrahigh frequencies (UHF), frequencies higher than 100 MHz, have thus been dubbed "ultrasound microbeams". It has been found that ultrasound microbeam can be used to acoustically trap and sort small particles (in micron size) and cells [10–12]. For such applications, UHF transducers with desirable performance are needed. Tradition-

ally, zinc oxide (ZnO) has been the material for UHF (200 MHz-1 GHz) transducer design [13] because it has a low dielectric constant ($\varepsilon^{S} \sim 4$) and it can be deposited in very thin layers (\sim 1 μ m) with excellent uniformity on a substrate. However, due to its low electromechanical coupling coefficient $(d_{33} \sim 10-26 \text{ pm/V}, k_t \sim 0.28)$, other piezoelectric thick/thin films with higher electromechanical coupling coefficients are needed to offer better performance. Because of the high voltage (receiving) coefficient ($d_{33} \sim 33$ pC/N, $\varepsilon^{S} = 5$, $g_{33} \sim 280 \times 10^{-3} \text{ V m/N}$), piezoelectric polymer (e.g. polyvinylidene fluoride (PVDF)) or polyvinylidene copolymer (e.g. fluoride-trifluoroethylene (P(VDF)-TrFE)) has also been used for many commercial high frequency (in 100 MHz range) single element transducers [14]. With a low dielectric permittivity ($\varepsilon^{S} \sim 14$), lithium niobate (LN) offers a viable alternative [15].

In the fabrication of ultrahigh frequency transducers, one of the technical challenges is the preparation of the piezoelectric element [16]. Piezoelectric material thickness on the order of only a few microns must be prepared. Among various thick film fabrication methods, a composite sol–gel technique [17] has attracted much interest to develop dense and crack-free ceramic films [18,19]. For the transducer element material, since the traditional lead-based ceramics (>60% lead in PZT by weight) cause environmental pollution by releasing the lead during the fabrication process, the trend is to develop relatively environmental friendly lead-free material systems [20].

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Among the lead-free material systems, (K_{0.5}Na_{0.5})NbO₃ (KNN)based and bismuth sodium titanate (Bi_{0.5}Na_{0.5})TiO₃ (BNT)-based materials are promising candidates. The KNN-based ceramics have been found to exhibit high piezoelectric ($d_{33} \sim 210-270 \text{ pC/N}$) and electromechanical coupling coefficients ($k_t \sim 0.47$) [21,22] while the BNT-based ceramics shows strong ferroelectricity ($E_c \sim 37 \text{ kV/}$ cm; $P_r \sim 38 \,\mu\text{C/cm}^2$) [23,24]. Both material systems have extensively been used for various electromechanical applications [25–28]. Combining the merits of two systems, a KNN/BNT thick film synthesized by the composite sol-gel technique has been developed [29]. In an initial attempt, a 193-MHz high-frequency ultrasound transducer fabricated using the KNN/BNT film was reported to exhibit a -6 dB bandwidth of 34% [30]. Although the work on the 193-MHz KNN/BNT film transducer was published, the sensitivity of the transducer was poor because of the inhomogeneous quality of the film. With the weak signal of the previous transducer, no insertion loss (sensitivity) could be measured and reported.

In the present work, with the maturity of the fabrication technique, the film quality can be retained even when the film thickness is <5 μm . This manuscript describes the lead free transducer operating at the frequencies exceeding 300 MHz. It is novel because the >300 MHz is the highest center frequency of lead-free ceramic ultrasonic transducers ever reported. Besides achieving much higher transducer frequency, the insertion loss of the transducers can also be measured and reported. Compared to the previous work, the present work is a breakthrough in both material and transducer points of view.

In this work, results on ultrahigh frequency transducers in a frequency range of 170–320 MHz designed and fabricated with thinner KNN/BNT films (<5 μm) are reported. Both -6 dB bandwidth and insertion loss of these transducers were measured. The measured transducer characteristics were shown to be comparable to the simulation. To demonstrate their imaging capability, a 6- μm tungsten wire phantom was imaged with a 205 MHz transducer to determine the axial and lateral resolutions. It was found that the present transducer performance is better or comparable to previously reported results even though the frequency is much higher.

2. Methods

In this section, the transducer material, design and fabrication process are described.

2.1. Transducer element

KNN/BNT composite films were fabricated using a composite sol–gel technique. Based on the traditional sol–gel method, the technology of composite and vacuum infiltration was utilized. To develop the KNN/BNT composite films, KNN ceramic powders were prepared using a conventional solid-state reaction method and BNT precursor solution was synthesized by a sol–gel process. At the annealing temperature of 750 °C, the KNN/BNT films with the KNN powder – BNT solution weight ratio of 0.40 were found to exhibit the optimized structural, dielectric and ferroelectric properties [29]. Thus, the composite films with this designated composition were chosen as the transducer element in this study. To further improve the film density and homogeneity, fine KNN powder was obtained by ball-milling for long time (10 h) and a vacuum infiltration process with BNT precursor solution was applied after each coating of the composite film.

2.2. Transducer design and fabrication

KNN/BNT composite film ultrasonic transducers were fabricated using conventional transducer technology. The composite

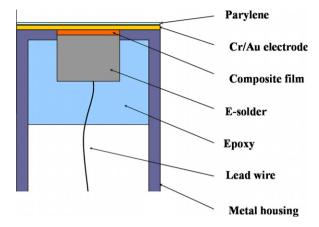


Fig. 1. Schematic diagram of the transducer.

films with thicknesses ranging from 3 to 5 µm were used to fabricate the ultrahigh frequency transducers (>170 MHz). A crosssectional view of the design is shown in Fig. 1. Firstly, a very lossy (an attenuation of 112 dB/mm at 30 MHz [31]) conductive epoxy (E-Solder 3022, Von Roll Isola Inc., New Haven, CT) was applied to the transducer element as the backing layer material by centrifuging it at 1200g for 15 min. After curing at room temperature overnight and lapping the backing layer, the sample was diced into small posts with dimensions of 0.2×0.2 mm². The composite film with the support of the backing layer was removed from the silicon substrate without any damage using a wet etching method [32]. The acoustic stack was released after immersing in 20% concentrated KOH solution at 80 °C for a few minutes. Afterwards, a 1.5 cm-long lead wire was connected to the backing layer using E-Solder 3022 epoxy. The transducer element was fixed inside a metal housing using epoxy (EPOTEK 301, Epoxy Tech., Billerica, MA). After curing the epoxy, a chrome/gold layer of \sim 1500 Å in total thickness was sputtered across the active element and the housing to serve as a common ground. A parylene layer was then vapor-deposited on the front face of the transducer to serve as an acoustic matching layer as well as a protection layer. The transducer prototype was assembled with an SMA connector for further electrical treatments and measurements to facilitate electrical poling and pulse-echo characterization.

A photo of final transducer prototype is shown in Fig. 2. The transducers were poled with an electric field of 210 kV/cm using a high voltage power supply (Bertan Associates, Inc., Syosset, NY) at 115 °C for 10 min so as to induce the piezoelectric performance



Fig. 2. Photograph of the final transducer prototype.

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