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Fabrication and characterization of annular-array, high-frequency, ultrasonic transducers based on PZT thick film

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ABSTRACT

In this work, low temperature deposition of ceramics, in combination with micromachining techniques have been used to fabricate a kerfed, annular–array, high–frequency, micro ultrasonic transducer (with seven elements). This transducer was based on PZT thick film and operated in thickness mode. The 27 μm thick PZT film was fabricated using a low temperature (720 °C) composite sol–gel ceramic (sol + ceramic powder) deposition technique. Chemical wet etching was used to pattern the PZT thick film to produce the annular array ultrasonic transducer with a kerf of 90 μm between rings. A 67 MHz parallel resonant frequency in air was obtained. Pulse-echo responses were measured in water, showing that this device was able to operate in water medium. The resonance frequency and pulse-echo response have shown the frequency response presented additional resonance mode, which were due to the lateral modes induced by the small width-to-height ratios, especially for peripheral rings. A hybrid finite-difference (FD) and pseudospectral time-domain (PSTD) method (FD–PSTD) was used to simulate the acoustic field characteristics of two types of annular devices. One has no physical separation of the rings while the other has 90 μm kerf between each ring. The results show that the kerfed annular-array device has higher sensitivity than the kerfless one.

1. Introduction

Ultrasound transducers are widely used today especially in the field of medical imaging systems. The growing demand of higher-resolution imaging requires desirable materials, fabrication techniques and design of structures for creating effective high frequency ultrasound transducers. Lead zirconate titanate (PZT) is a suitable material for ultrasound transducer because of its high piezoelectric constant, relative permittivity and electromechanical coupling coefficient [1]. High operating frequencies above 30 MHz are needed to achieve high resolution images for various applications such as ophthalmology, dermatology and intravascular imaging [2].

The thickness-mode resonant vibration frequency is inversely proportional to the thickness of the PZT structure [3]. Therefore, thicknesses of 10–100 μm are required to achieve sufficiently high frequencies and spatial resolution. Fabrication of the thick PZT film represented a significant technical challenge using thin-film technology such as physical vapour deposition, chemical vapour deposition, sputtering or pulsed laser deposition, due to the slow deposition rates and high levels of stress generated during the process, which can lead to cracks in the film [4]. The use of conventional bulk ceramic processing with subsequent machining and bonding has a significant difficulty of producing film smaller than 100 μm, because of the high brittleness of the film at this thickness [5]. Other thick film fabrication techniques based on sintering of ceramic particles, such as screen printing, are limited by high temperature processing which can easily result in damage to the bottom electrode, substrate and PZT film itself [6]. In our previous work using a combination of conventional sol–gel processing and PZT powder processing thick films between 2 and 30 μm were fabricated using spin coating methods at lower temperatures (720 °C), compatible with silicon [7].

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Table 1
Composition of PZT slurry.

PZT slurry	Quantity
PZT sol	30 ml
PZT powder	45 g
Dispersant KR 55	0.9 g
Cu_2O/PbO	0.3105/1.926 g
Zirconia balls milling media	200 g

The most commonly used ultrasound transducers are single elements [8]. However, the image field-of-view is limited when using this kind of transducers. To overcome this problem, multi-element transducers such as linear and annular arrays allow for the focal distance to be varied through delayed excitation of the array elements, which significantly increases the depth-of-field [9]. A simple type of multi-element ultrasonic transducer is a kerfless array, where there is no physical separation between elements [10]. However, crosstalk between adjacent elements can significantly degrade performances [11]. One effective way of reducing crosstalk is to mechanically isolate the individual elements [12]. The fabrication of kerfed multi-element arrays is challenging due to the dimensions of the kerfs required at high frequencies [13]. In this work a kerfed annular-array, high-frequency ultrasonic transducer with seven elements was designed and fabricated using the PZT composite slurry/sol deposition technique combined with micromaching fabrication technology. The performance of this device, including resonance behavior and acoustic responses, was analyzed.

2. Materials and methods

2.1. PZT sol and composite slurry

The PZT sol used in this work was prepared from the precursors of Lead (II) acetate trihydrate, Titanium (IV) isopropoxide, Zirconium (IV) propoxide, Niobium (V) ethoxide, Antimony (III) ethoxide, Manganese (II) acetate and 2-methoxyethanol (2-ME). The details of preparation procedures were described in our previous work [14]. The general formula of the sol used was: $Pb_{1.1}[Nb_{0.02}Sb_{0.02}Mn_{0.02}(Ti_{0.48}Zr_{0.52})_{0.94}]O_3$. The PZT thick film was formed through deposition of a composite slurry prepared from PZT sol and PZT powder. The PZT slurry was prepared by mixing PZT powder (Pz26, Ferroperm Piezoceramics, MEGGITT, Denmark), 2-ME based PZT sol, sintering aid of CuO_2–PbO (4.7 wt%) and dispersant KR55 (Ken-React Lica 38, KenRich) in a nitrogen environment, then ball-milled on a roller for 24 h. In the PZT slurry the sol binds the PZT particles together and reduces the sintering temperature, due to the presence of nanoscale material, which in turn decreases lead volatilization [15]. The sintering aid enhances densification and leads to an increase in piezoelectric properties of the PZT film formed [16]. The composition of the PZT slurry is shown in Table 1.

2.2. Fabrication of the annular array

Fig. 1 shows the procedures for creating the PZT thick-film, annular-array ultrasonic transducer. The 60 nm ZrO_2 diffusion barrier was formed on the silicon substrate by spin coating of a zirconium isopropoxide sol and crystallization in a furnace, prior to the deposition of the Ti/Pt electrode and the PZT film, to prevent Pb, from the PZT layer, diffusing into the silicon wafer to form Lead silicates. Patterned bottom and top electrodes of Ti/Pt (8 nm/100 nm) were deposited using photolithographic lift off techniques with RF magnetron sputtering. During the formation of the PZT thick film, the substrate with patterned bottom electrode was spin-coated (2000 rpm for 30 s) with two layers of composite slurry followed by six layers infiltration of diluted PZT sol (50 vol% in 2-ME). This process was repeated six times, leading to a final film thickness of about 27 μm.

The PZT thick film was patterned by masking and selectively exposing the PZT to chemical etchants. The photoresist and etching solutions were AZ4562 (Clariant UK Ltd., Horsforth, Leeds, UK) and HCl (4.5 wt%)/HF (0.5 wt%)/H_2O (95 wt%), respectively. The PZT was etched in a solution of HF/HCl at 60 °C for 10 min. The patterned PZT thick film was then sintered at 720 °C for 20 min prior to depositing the Ti/Pt top electrode. The SiO_2 and Si materials underneath the active PZT thick film structure were removed using RF reactive ion etching (RIE, 30 min) and deep reactive ion etching (DRIE, 140 min). Each element was poled at 8.4 V/μm at 200 °C for 5 min.

2.3. Electro-acoustic characterization

The electrical impedance of each element was characterized using a HP4395 spectrum analyzer (Agilent Technologies, Inc., Santa Clara, CA, United States) to determine the resonance frequency and the electromechanical coupling coefficient (thickness mode) of the individual rings. Performances of the transducer were also evaluated through measurement of the acoustic field radiated by each element. They were excited using an AVTEC AVG-3B-C impulse generator (Avtech Electrosystems Ltd., Ottawa, Canada) with a 50 MHz impulse and the acoustic response was measured in water using a broadband needle hydrophone with a 40 μm-large active area (Precision Acoustics Ltd., Dorchester, UK).

3. Results and discussion

3.1. Structure of the annular array

The annular-array transducer was composed of 7 rings with a mechanical separation of 90 μm at the surface of the PZT film, and an outer diameter of approximately 2 mm. Each individual electrode had an area of 0.212 mm^2 to ensure electrical impedance matching. Fig. 2a shows an overview of the 7-ring transducer with the top ground electrode faintly visible as a vertical line in the lower half of the device. One can see that some PZT ceramic remains between rings in some areas, particularly near the base of the kerfs where it is difficult for the etchant to enter and waste product to be removed. During the etching process an intermediate product of PbClF will be formed [17]. The residue of PbClF can act as a barrier that affect the etching uniformity, particularly in the narrow place such as the PZT residue near the base of kerfs observed in this work. The individual electrodes and connection lines are clearly visible from the backside of the PZT film (Fig. 2b), where the silicon has been removed by DRIE.

Fig. 3 shows the homogeneous microstructure of the PZT thick film after etching. The PZT film exhibited slight, sub-micrometer surface cracking (Fig. 3a), but these cracks were non-connecting such that the top electrode deposited on the PZT surface was not disrupted and all of its area remained active. The cracks also do not extend through the entire thickness of the PZT film, as can be seen in Fig. 3b, such that no element was electrically shorted. The porosity of this PZT thick film is ~13% which was deduced from the scanning electron microscopy micrographs of film fracture cross section. The variation of the density of a material with porosity can be considered linear. The relationship between density of the PZT thick film and its porosity can be presented by the following equation [18].

$$\rho = \rho_0(1 - P) \quad (1)$$