



Contents lists available at ScienceDirect

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc

Original Article

Innovative method to produce large-area freestanding functional ceramic foils

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ARTICLE INFO

Keywords:

Freestanding functional foils
NaCl sacrificial layers
PZT ceramic films
Ferroelectric

ABSTRACT

Using thick and thin films instead of bulk functional materials presents tremendous advantages in the field of flexible electronics and component miniaturization. Here, a low-cost method to grow and release large-area, microscale thickness, freestanding, functional, ceramic foils is reported. It uses evaporation of sodium chloride to silicon wafer substrates as sacrificial layers, upon which functional lead titanate zirconate ceramic films are grown at 710 °C maximum temperature to validate the method. The freestanding, functional foils are then released by dissolution of the sacrificial sodium chloride in water and have the potential to be integrated into low-thermal stability printed circuits and flexible substrates. The optimization of the sodium chloride layer surface quality and bonding strength with the underlying wafer is achieved thanks to pre-annealing treatment.

1. Introduction

Functional ceramics with ferroelectric properties have many applications in electronics as transducers, actuators and sensors [1] that can be used for example in mobile phones [2], structural health monitoring [3], medical imaging [4], robotic surgery [5], touch screens [6], micro-positioning [7] and active vibration control [8]. The use of thick and thin films as opposed to bulk functional ceramics has many benefits in terms of cost, size, weight and sensitivity, which greatly enhances the usability of transducers, actuators and sensors [1]. Therefore, having to integrate a bulky substrate, which serves no other purpose than growing the functional film, is an inconvenience for electronic components fabrication and an obstacle to their miniaturisation. And since growing functional ceramics generally requires a minimal temperature around 500 °C, it is preferable to integrate freestanding microscale foils on low-thermal stability printed circuits or polymers for flexible electronics, rather than growing them directly in-situ. Solutions such as thinning bulk functional ceramics [9], removing the growth substrate [10] or transferring the functional ceramic foil to a new carrier substrate [11,12] using respectively grinding, etching or a laser process, have been suggested. However, thinning functional bulk materials to less than 30 μm is long, tedious and very challenging as they are generally friable ceramics and the process results in subsurface damage [9]. Moreover, procedures to remove the substrate such as isotropic etching are very costly in terms of time and equipment and consumables [10].

Finally, the laser transfer is time-consuming, limited in feature size and requires expensive transparent growth substrates, generally a glass material with a low coefficient of thermal expansion (α_{CTE}). Besides, there is the technical challenge to focus the beam of a very expensive-to-operate laser at the functional ceramic layer/glass growth substrate interface to transfer the functional layer to a new carrier without creating cracks in the ceramic film. Indeed, the high energy density produced by the laser and the difference between the α_{CTE} of the functional film and the growth substrate, are likely to generate stresses and cause cracks [11,12].

In the past, sodium chloride (NaCl), a non-oxide ceramic, has been used as a cheap, bulk, sacrificial material to achieve freestanding foils of materials such as Ni [13]. Besides, it was deposited onto a suitable carrier surface using an evaporation technique [14–16]. However, to the best of our knowledge, this work is the first successful attempt to deposit functional ceramic films on evaporated, sacrificial, NaCl layers and then release them as microscale, freestanding foils thanks to a simple dissolution of the NaCl interface in water. The chosen functional material was a lead zirconate titanate (PZT) ceramic, developed at Cranfield University and previously studied in literature [17,18], as it is one of the most widely used ferroelectric materials, commonly in the morphotropic phase boundary composition of $Pb_{1.1}(Zr_{0.52}Ti_{0.48})O_3$ [19,20].

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<https://doi.org/10.1016/j.jeurceramsoc.2018.03.051>

Received 19 September 2017; Received in revised form 27 March 2018; Accepted 30 March 2018
0955-2219/ © 2018 Published by Elsevier Ltd.

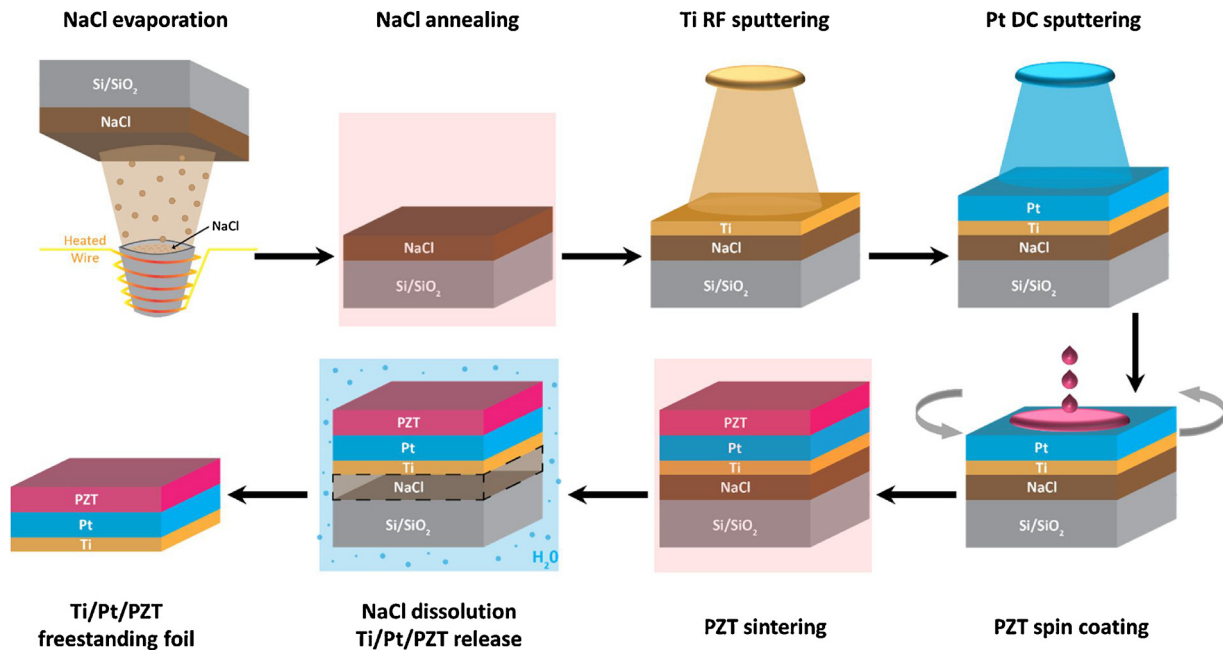


Fig. 1. Schematic of the manufacturing process of the Ti/Pt/PZT freestanding foils.

2. Experimental section

A schematic summarizing the whole manufacturing process to obtain Ti/Pt/PZT freestanding foils is presented in Fig. 1. First, NaCl release layers were deposited onto 500- μm thick Si substrates covered with a 300-nm thick top SiO₂ layer, using an Edwards thermal evaporator with a tungsten basket heater and an alumina crucible (B8A-3 \times .030 W from Testbourne Ltd, UK). In a typical procedure, the base pressure was maintained at 1×10^{-4} Pa and the current was successively set to 30 A for 60 s, 50 A, and 40 A for 1250 s to outgas, melt and deposit 250 nm of NaCl (Sigma Aldrich $\geq 99.5\%$), respectively. Afterwards, the NaCl-coated substrates were annealed in a Carbolite box furnace in ambient air at 450, 550, 650 or 750 $^{\circ}\text{C}$, during 1 or 3 h. Afterwards, a tape test and a dissolution test into H₂O were carried out to assess the NaCl/SiO₂ bonding strength and the ability to release a functional freestanding foil, respectively. Annealed NaCl layers were coated with electrodes composed of 8 nm of Ti and 250 nm of Pt, using a Nordico radio frequency/direct current (RF/DC) sputter system. Ti and Pt were respectively RF and DC sputtered under argon. Subsequently, 6- μm thick PZT layers were deposited on the surface of the electrodes using a standard composite sol gel methodology described elsewhere [17,18]. Finally, Ti/Pt/PZT foils were released almost instantaneously from the wafers thanks to a simple immersion in H₂O. In some cases, a Nitto Denko - Revalpha 3195 V tape strip was previously bonded to the surface of the PZT layer. The structure and microstructure, were characterised using X-ray diffraction (XRD, Siemens D5000) and scanning electron microscopy (SEM, Phillips XL30). Moreover, the Na and Cl content in the freestanding foils was determined by energy dispersive spectrometry (EDS, Oxford Instruments). Finally, the capacitance loss and ferroelectric properties were studied with a precision component analyser (Wayne Kerr 6425) and a Radiant Technology RT66C Ferroelectric tester, respectively. For the latter characterisation, the PZT film was either Si/SiO₂ wafer-supported and sandwiched between two Pt electrodes, or freestanding and sandwiched between a Pt electrode and a liquid metal eutectic drop of indium, gallium and tin, commonly referred to as Galinstan [21,22], and brought to the PZT surface with a hypodermic syringe. The samples were stored in a vacuum desiccator between each process and/or characterisation step to avoid absorption of atmospheric moisture.

3. Results and discussion

In a preliminary test, a Pt film was deposited on top of an NaCl non-oxide ceramic layer without any previous annealing of the latter, which caused the whole coating to delaminate from the SiO₂ surface. This meant that the stress generated at the Pt/NaCl interface exceeded the binding energy at the NaCl/SiO₂ one. Indeed, since the wafer was held at ambient chamber temperature during the NaCl deposition, the thermal energy was too low to induce a satisfactory NaCl grain consolidation or a chemical bonding at the NaCl/SiO₂ interface. The influence of the water contained in the hygroscopic NaCl layer was assumed to be insignificant as it should have been extracted when the chamber was evacuated down to a pressure of 1.33×10^{-5} Pa before the Pt sputtering.

Hence, in order to optimise adhesion of NaCl to SiO₂, and its surface quality to grow a PZT ceramic film upon, post evaporation annealing was performed as described in Table 1. The corresponding samples are referenced as “NaCl T/t”, with T the annealing temperature in $^{\circ}\text{C}$ and t the dwell time in hours. Three possible beneficial effects were expected [23–25]: a densification of NaCl improving the mechanical integrity of this layer, a grain growth lowering the surface roughness and giving a higher quality interface for the Ti/Pt/PZT film deposition, and the formation of a sodium silicate ceramic phase (Na_xSi_yO_z) promoting a strong chemical bond at the NaCl/SiO₂ interface.

According to the results summarised in Table 1, the samples annealed at 450 and 550 $^{\circ}\text{C}$, regardless of the dwell time, were soluble in water but mostly peeled off the SiO₂ layer during a tape test,

Table 1
Annealed NaCl samples ID, annealing temperature and time, and dissolution and tape test results.

Sample ID	T/ $^{\circ}\text{C}$	t/h	Dissolution into H ₂ O	Peeling during tape test
NaCl 450/1	450	1	Yes	Yes
NaCl 450/3	450	3	Yes	Yes
NaCl 550/1	550	1	Yes	Yes
NaCl 550/3	550	3	Yes	Yes
NaCl 650/1	650	1	Yes	No
NaCl 650/3	650	3	No	No
NaCl 750/1	750	1	No	No
NaCl 750/3	750	3	No	No

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