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Cantilever resonator based on the electrostriction effect in Gd-doped ceria

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ABSTRACT

Thin films of $Ce_{0.8}Gd_{0.2}O_{1.9}$, which have recently been shown to demonstrate giant electrostriction, are proposed as the active material for miniature cantilever resonators and actuators. In the absence of strain, these films have an electrostriction coefficient within the range of 2–10 kPa/(kV/cm)^2, as compared with the somewhat larger values for the best commercial electrostrictors (e.g. $PbMn_{1/3}Nb_{2/3}O_3$, 62 kPa/(kV/cm)^2. At the same time, $Ce_{0.8}Gd_{0.2}O_{1.9}$ films can generate stress >70 MPa which is competitive with materials currently in use and only limited by the strength of the film-substrate interface. In this report, we investigate two aspects of the practical application of $Ce_{0.8}Gd_{0.2}O_{1.9}$ as a resonator: the fabrication conditions and the frequency dependence of the electrostrictive behavior. We show that the films can display electromechanical response with frequencies up to 6 kHz. With respect to fabrication, we show that $Ce_{0.8}Gd_{0.2}O_{1.9}$ films have a number of technological advantages when compared to the lead titanate based materials currently in use: (a) they can be deposited on a variety of metal contacts and substrates, including silicon; (b) they do not require high temperature processing; and (c) because $Ce_{0.8}Gd_{0.2}O_{1.9}$ has cubic symmetry, it can in principle be used as a polycrystalline film with arbitrary texture and does not require poling. In addition, neither Ce nor Gd nor their oxides are toxic; the oxides have very low vapor pressure; and the cations, being highly charged do not diffuse into Si. Consequently, $Ce_{0.8}Gd_{0.2}O_{1.9}$ films may be readily and advantageously integrated into existing semiconductor fabrication technologies.

1. Introduction

Miniature electromechanical devices, the backbone of modern microelectromechanical systems (MEMS) [1–3], convert electrical energy into physical displacement using either multi-part electric motors or electromechanically active materials. The latter display better performance [4,5]. However, the best electromechanically active materials, piezoelectrics and electrostrictors, require high temperature processing (>600 °C) [4,5], which complicates their integration into Si-based microfabrication. Furthermore, the most efficient piezoelectrics and electrostrictors are derivatives of lead titanate and their presence in Si-based microfabrication requires insulating diffusion barriers and Pt alloy electrodes [6–8]. Increasing demand for environmentally friendly materials has promoted numerous studies on lead-free piezoelectrics and electrostrictors in order to find replacements for lead titanate based ceramics. Among these is AlN in both its single crystal and nanocrystalline forms. AlN is suitable for certain MEMS applications, such as mass sensing [9]. It is characterized by strong high frequency response, comparative ease of preparation and compatibility with microfabrication techniques [10]: the piezoelectric coefficients are $e_{33}^{AlN} \leq 1.34$ C/m^2 (134 kPa/(kV/cm)), $e_{13}^{AlN} = -0.6$ C/m^2 (−60 kPa/(kV/cm)) [11], with correspondingly low resulting stress. However, in view of the broad range of MEMS requirements, e.g. for actuators and other high stress, low frequency applications, the development of additional high performance, electromechanically active materials that are compatible with microfabrication processing, as well as being non-toxic, remains a serious research goal.

We have recently reported that thin films of Gd-doped ceria, $Ce_{0.8}Gd_{0.2}O_{1.9}$ (GDC), one of the most important oxygen ionic conductors, incorporated in a glass-based cantilever [glass\100 nm of Cr, Al, or Ti\300–700 nm $Ce_{0.8}Gd_{0.2}O_{1.9}$\100 nm of Cr, Al, Ti or Au], exhibit a large electrostriction effect [12]. With <5 V applied between the metallic contacts, the layer of $Ce_{0.8}Gd_{0.2}O_{1.9}$ can generate stress of a few tens of MPa. The saturation electric field of $Ce_{0.8}Gd_{0.2}O_{1.9}$ is not yet known but it is definitely at least 110 kV/cm (∼70 MPa stress), which makes this material potentially competitive with the best commercially available materials. In the same study [8], we also observed that the cantilever exhibited a

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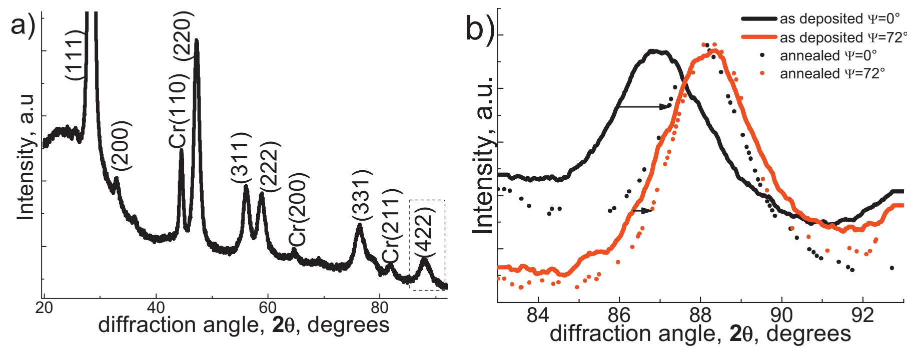

Fig. 1. (a) Indexing of the XRD pattern of a 690 nm $Ce_{0.8}Gd_{0.2}O_{1.9}$ film deposited on a glass substrate covered with a 120 nm thick Cr layer as the bottom electrode (peaks labeled Cr). (b) The (4 2 2) peak of the $Ce_{0.8}Gd_{0.2}O_{1.9}$ film with tilt angles, $\psi = 0°$ and 72° measured both before (solid lines) and after (dotted lines) 6 h of annealing at 400 °C.

well-defined mechanical resonance. However, issues related to the possible practical application of $Ce_{0.8}Gd_{0.2}O_{1.9}$ thin films for actuators and resonators were not explored. These include:

1) The electrostrictive response in $Ce_{0.8}Gd_{0.2}O_{1.9}$ is described as being related to defect rearrangement [12]. This is expected to be a much slower process than the electromechanical response in perovskite-type piezoelectrics (GHz) and electrostrictors (a few tens of kHz) [13]. Therefore, the frequency range within which $Ce_{0.8}Gd_{0.2}O_{1.9}$-based devices are able to operate is of primary practical interest.
2) The technological compatibility of $Ce_{0.8}Gd_{0.2}O_{1.9}$ actuators with Si microfabrication remains to be clarified, in particular, details concerning processing temperatures and suitable electrode materials.

In this report, we address both issues and demonstrate that $Ce_{0.8}Gd_{0.2}O_{1.9}$ films can function as the active layer in cantilever mechanical resonators operating at frequencies up to ~6 kHz and that these films are indeed compatible with the constraints of Si microfabrication technology.

2. Experimental results and discussion

2.1. Cantilever preparation

$Ce_{0.8}Gd_{0.2}O_{1.9}$-based cantilevers were prepared from either ≈150 μm thick glass slides or ≈250 μm thick *n*-Si^{++} (100) wafers (STV Telecom) [12]. The substrates were cut into 8–9 mm wide, 40–50 mm long rectangles. This cantilever length was chosen in order to provide deflection of a few tens of microns, thereby optimizing measurement accuracy. Glass slides have a lower elastic modulus (70 GPa) than Si wafers (130 MPa), and the cantilever deflection is correspondingly larger. When Si was used, it served as the bottom electrical contact. A 10 nm layer of Ti was RF sputtered on the Si substrate at room temperature prior to the deposition of $Ce_{0.8}Gd_{0.2}O_{1.9}$ in order to eliminate the thin insulating SiO_2 layer that grows naturally on the Si surface during film deposition. However, this stage can be omitted at the expense of some decrease in performance due to a voltage drop in the SiO_2 layer. In the case of glass substrates, 100 nm thick Al, Ti or Cr layers, deposited by sputtering, were used as the bottom electrode. 300–700 nm thick films of $Ce_{0.8}Gd_{0.2}O_{1.9}$ were deposited on the bottom electrode using RF magnetron sputtering at room temperature in Ar + O_2 atmosphere as described in Refs.[14,15]. According to X-ray diffraction (XRD, Rigaku TTRAXIII) the as-deposited polycrystalline films were in a pure fluorite phase with a strong (1 1 1) texture (Fig. 1a); a clearly expressed columnar structure normal to the film plane could be observed (SEM, Leo Supra) (Fig. 2a and b), which is typical for $Ce_{0.8}Gd_{0.2}O_{1.9}$ prepared by sputtering. The (4 2 2) XRD peak position, measured as a function of inclination angle, $\psi = 0°$, 72° (Fig. 2b) showed that the as-deposited films were under compressive strain (0.1–0.4%) which depended on the bottom contact (Al – minimum, Ti/Si – maximum). Although films with in-plane compressive strain may display a much larger electrostriction effect than strain-free films (reaching 500 MPa stress) [12] the reproducibility was found to be poor because of the inevitable partial stress relaxation at the film-substrate interface. The extent and rate of the relaxation strongly depend on the bottom contact layer. Films deposited on Al relaxed almost completely (to below 0.1% strain) within a few hours while films on Ti/Si do not relax spontaneously. However, the film-substrate interface of the strained films may undergo catastrophic failure during voltage application, especially if high field-induced stress (>80 MPa) is developed. Therefore, in order to compare film

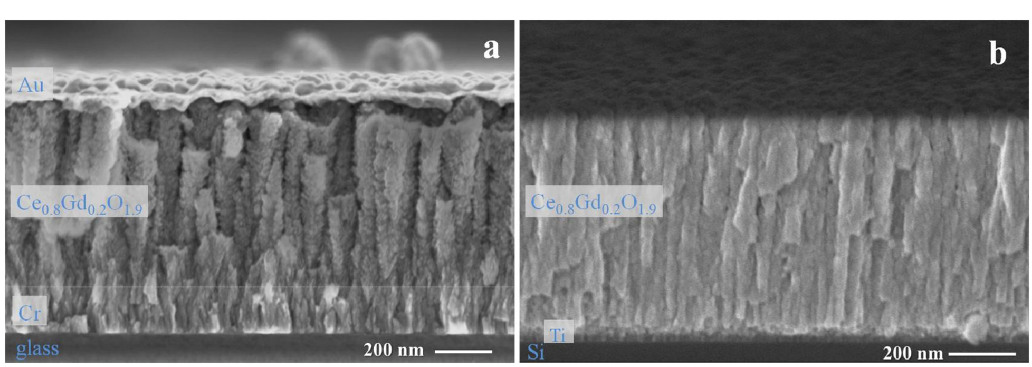

Fig. 2. SEM images of the cross-section of: (a) [150 nm Cr/650 nm $Ce_{0.8}Gd_{0.2}O_{1.9}$/70 nm Au] layered structure deposited on a 150 μm thick glass slide; (b) [10 nm Ti/610 nm $Ce_{0.8}Gd_{0.2}O_{1.9}$] layered structure deposited on a 250 μm thick *n*-Si^{++} (1 0 0) wafer prior to top contact deposition. The Cr bottom layer in (a) is not readily distinguished from the ceramic film due to its only marginally lighter color and smaller grain size.