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Towards Micromechanical Sensors with (La,Sr)MnO_3 Epitaxial Films

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Abstract

The rich spectrum of functionalities exhibited by oxide thin films is an appealing feature for the development of micro and nanomechanical devices [1,2]. MEMS made of heterostructures of crystalline oxide materials having targeted physical properties may be applied as sensors having different integrated functionalities. In this work, we explore the feasibility of manganite thin film based epitaxial MEMS for magnetic micromechanical sensing. We investigate the electromechanical properties of LSMO freestanding structures for future applications in the field of micromechanical magnetic sensors.

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Keywords: MEMS; microstructures; magnetic field sensors; oxides;

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1. Introduction

Manganese oxides of general formula $RE_{1-x}M_xMnO_3$ (RE = rare earth, M = Ca, Sr, Ba, Pb) have remarkable structural, magnetic and transport properties due to the mixed valence (3+/4+) of the Mn ions. For example, LSMO exhibits a transition from a high temperature paramagnetic semiconducting or insulating phase to a low temperature

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ferromagnetic phase at around 360 K [3]. MS with oxides can be employed for studying the physical properties of these materials and for the development of sensors that take advantage from the use of crystalline materials having high resistance to harsh environments and engineered strain. The spontaneous magnetization of LSMO below its transition temperature can provide an effective coupling mechanism with an external magnetic field. This coupling can be detected by measuring the mechanical resonance frequencies of the MS. This is analogous to what occurs in Magnetic Force Microscopy (MFM) [4], where surface mapping of magnetic domains is achieved by measuring the position-dependent shifts of the eigenfrequency of a cantilever having a magnetized tip. Here, we characterize the mechanical properties of LSMO based cantilevers in view of the development of micro and nanomechanical resonant sensors.

Nomenclature

LSMO	$La_{0,7}Sr_{0,3}MnO_3$
STO	$SrTiO_3$
MS	Micromechanical Structures
MEMS	Micromechanical Systems
PLD	Pulsed Laser Deposition
MFM	Magnetic Force Microscopy

2. Thin film deposition and device fabrication

LSMO films were grown on STO(001) substrates by PLD from stoichiometric target in oxygen pressure (13 mPa) at a laser fluency of 0.8 J cm^{-2} and 1 Hz repetition rate. Substrate temperature was fixed to 850°C during deposition and 20 min annealing in oxygen pressure (2.6 10^4 Pa) at 600°C was performed after the growth. LSMO films present surface roughness of about 2–3 nm *rms*. The transition temperature of our films depends on the La/Sr ratio and oxygen stoichiometry, which mainly depends on the laser parameters and oxygen partial pressure during the growth or annealing conditions, respectively. For these reasons, the critical temperature and the R(T) characteristics of the films are scattered around the nominal bulk one (~360K). MS fabrication was performed by standard optical photolithography (Megaposit SPR 220-4.5 photoresist) and Ion milling (Ar ions 500 eV, 0.2 Acm^{-2}) (Fig. 1). Free-standing structures were fabricated using diluted HF (4,8% in water solution), with mild agitation, which removes STO without etching the LSMO film [5,6].

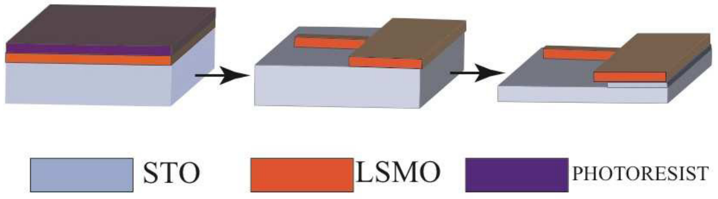

Fig. 1: Fabrication process of suspended microstructures.

3. Electrical and mechanical characterization

We measure the mechanical eigenfrequency of these MS by a custom system based on optical lever technique able to detect oscillations of MS at different temperatures (-10 °C to 200 °C) and in controlled environment (air, vacuum or pure gasses). Furthermore, electrical measurements are performed in a standard four-probe configuration. Fig. 2 shows a typical Resistance vs Temperature curve of a LSMO film. This curve presents a change of slope due to the magnetic phase transition at 350K, which is almost what we expected for bulk samples.

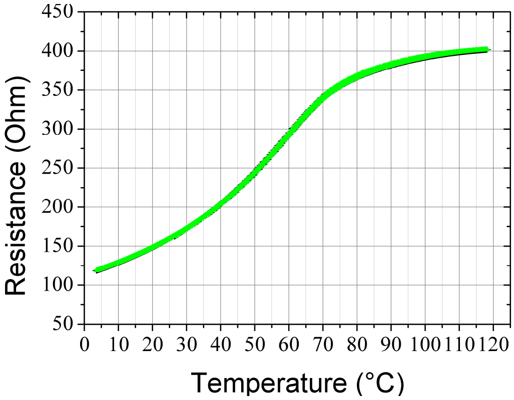

Fig. 2: Electrical Resistance vs Temperature characteristic of a 100 nm thick LSMO film in the 0-120°C temperature range.

For final applications as magnetic sensor, mechanical measurements among cantilevers having different dimensions are performed and compared with standard single-clamped bar theory, considering a straight and homogeneous structure with a Young's modulus value typical for this material [7]; theoretical values are also compared with ones derived from Finite Element Analysis as, for example, is presented in Fig. 3. The results of both studies are reported in the following table:

Table 1. Eigenfrequency of different LSMO cantilever.

Dimensions (μm^3)	Theoretical eigenfrequency (*kHz*)	Measured eigenfrequency (*kHz*)
(a) 50 x 10 x 0.16	47.6	47.8
(b) 50 x 20 x 0.3	86.6	170.5
(c) 100 x 20 x 0.3	21.6	50.8

As reported in table 1, sample (a) is in good agreement with the theoretical eigenfrequency value. On the contrary, the experimental values for samples (b) and (c) are quite different from the expected ones. The difference between theory and experiments increases with the cantilever width. A possible explanation can be given considering the presence of internal stresses accumulated inside the material due to the heteroepitaxial growth. These stresses curve the freestanding structure with the final effect of increasing its stiffness [8]. Curved cantilevers (*i.e.* bent along their transversal direction) are observed both in freestanding MS clamped from the base to the (strained) LSMO film (fig. 4a) and on MS detached from the original substrate (fig. 4b), where any stress transmitted from the clamped (still stressed) part of the LSMO film is suppressed. From these observations, we can assume that stress could have been accumulated inside the material during the deposition process and can be attributed to defects during deposition or gradients in the oxygen stoichiometry. The role of internal stress is crucial in the study and application of these structures, because of the great effect that a small amount of it can have on the actual dynamical behaviour of MS.

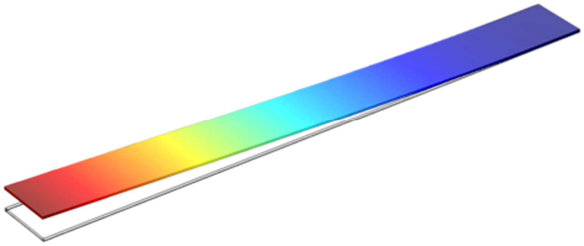

Fig. 3: Example of Finite Element Analysis of single-clamped LSMO cantilever.

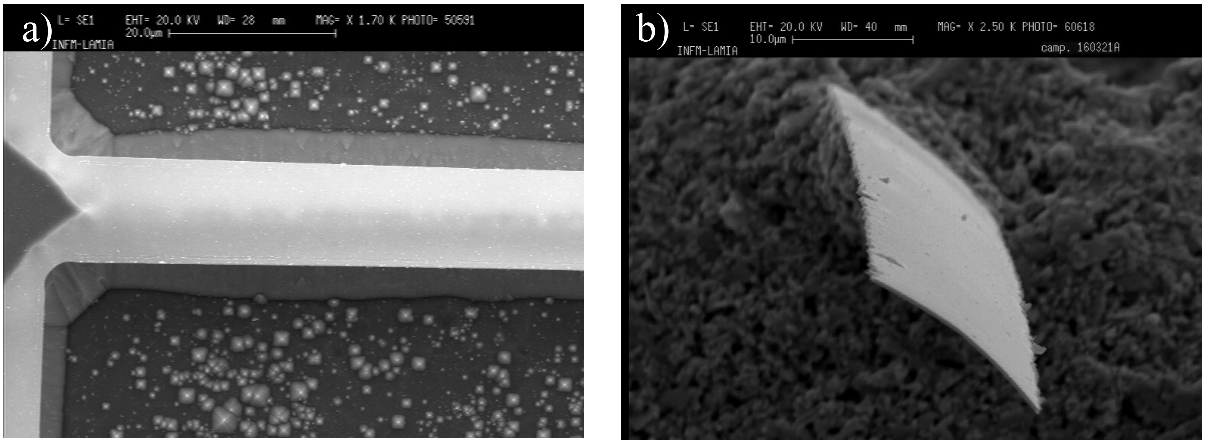

Fig. 4: SEM images of LSMO freestanding MS (a) with the base clamped to the substrate, (b) removed from substrate and glued to a Si chip by silver paste.

Further investigations in this direction are necessary to evaluate and control stress distribution is these structures.

4. Conclusions

In this work we performed electrical and mechanical characterization of LSMO freestanding MS focusing on the central role that internal accumulated stress plays in their dynamical behaviour, such as mechanical resonances. Knowledge of the basic properties that drive LSMO MS is the first step for developing complex sensors with these materials.

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