proceedings

Eurosensors Paris 2017

MDPI

Proceedings

Mechanical Characterization of (La,Sr)MnO_3 Microbridges for Thermometric Applications †

Federico Remaggi [1,2], Luca Pellegrino [2,*], Nicola Manca [1,2], Cristina Bernini [2] and Daniele Marré [1,2]

[1] Physics Department, University of Genova, 16146 Genova, Italy; federico.remaggi@spin.cnr.it (F.R.); nicola.manca@spin.cnr.it (N.M.); marre@fisica.unige.it (D.M.)

[2] Consiglio Nazionale Delle Ricerche-Istituto SPIN, 16152 Genova, Italy; cristina.bernini@spin.cnr.it (C.B.)

* Correspondence: luca.pellegrino@spin.cnr.it; Tel.: +39-10-6598-751

† Presented at the Eurosensors 2017 Conference, Paris, France, 3–6 September 2017.

Published: 28 August 2017

Abstract: MicroElectroMechanical Systems (MEMS) made of heterostructures of crystalline oxide materials with 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 thermometric micromechanical sensing. We investigate the mechanical properties of $La_{1-x}Sr_xMnO_3$, with x ≈ 1/3, freestanding microbridges as a function of temperature for applications in the field of micromechanical temperature sensors.

Keywords: uncooled bolometers; microbridges; micropirani

1. Introduction

The rich spectrum of functionalities exhibited by oxide thin films is an appealing feature for the development of micro and nanomechanical devices [1,2]. 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, $La_{1-x}Sr_xMnO_3$ with x ≈ 1/3 (LSMO) is a conducting oxide exhibiting a phase transition from a high temperature paramagnetic phase to a low temperature ferromagnetic phase at around 360 K [3]. As a consequence, the Temperature Coefficient of Resistance (TCR), defined as 1/R × (dR/dT) shows quite a large value (≈2% K^{-1}) nearby room temperature (Figure 1) compared to metals (platinum TCR ≈ 0.385% K^{-1}). This property suggested to employ LSMO-based microbridges for thermometric-related applications such as uncooled room temperature bolometers [4] and Pirani sensors [5]. While the referred studies focused on the resistive properties of LSMO freestanding microbridges (FSM), here we discuss the potentialities of micro-mechanical resonators based on LSMO FSM for mechanical-based thermometric applications. FSM with oxides can be employed both 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. Here, we characterize the thermomechanical properties of LSMO based microbridges in view of their employment as resonant sensors.

2. Material and Methods

Sample preparation starts from films grown on STO (001) substrates by Pulsed Laser Deposition (PLD) in oxygen pressure (13 mPa) at a laser fluency of 0.8 J cm^{-2} and 3 Hz repetition rate. Substrate temperature was fixed to 850 °C during deposition. Thermal annealing in oxygen pressure (2.6 × 10^4 Pa) at 650 °C for 30 min was performed after deposition. LSMO films present surface roughness of about 0.5 nm *rms* as measured by Atomic Force Microscopy. The transition temperature of our films depends on the La/Sr ratio and oxygen stoichiometry. These quantities are influenced by the deposition process, so the critical temperature (~360 K) and the R(T) characteristics of the films are generally slightly scattered around the reference bulk ones.

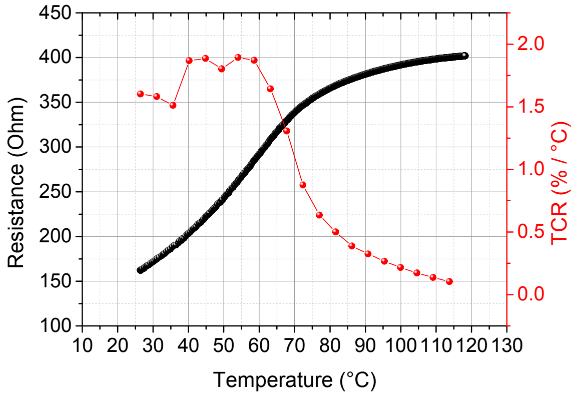


Figure 1. Electrical Resistance vs Temperature characteristics of a 100 nm thick LSMO film in the 25–120 °C temperature range (**left**) and respective calculated TCR (**right**).

FSM fabrication was performed by standard optical photolithography (Megaposit SPR 220-4.5 photoresist) and Ion milling (Ar ions 500 eV, 0.2 Acm^{-2}). Free-standing structures were fabricated using diluted HF (4.8% in water solution), with mild agitation, which removes STO without etching the LSMO film and affecting its transport properties [6,7]. We measure the mechanical eigenfrequency of these FSM by a system based on the optical lever technique able to detect the mechanical oscillations of microstructures at different temperatures (−10 °C to 200 °C) and in controlled environment (air, vacuum or pure gasses). Electrical measurements are performed in the same system in a standard four-probe configuration. An example of gold-coated LSMO FSM is shown in Figure 2.

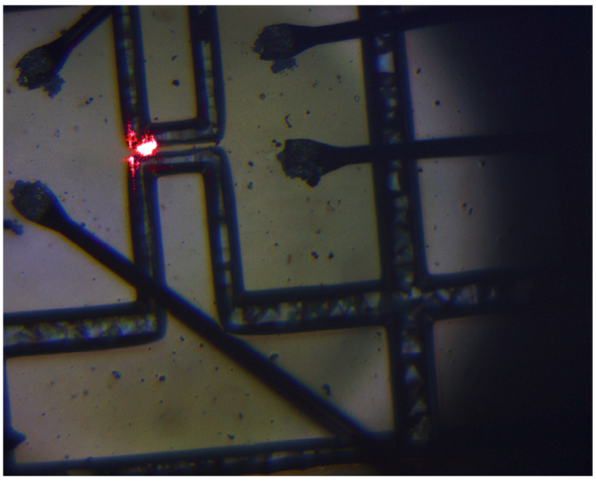
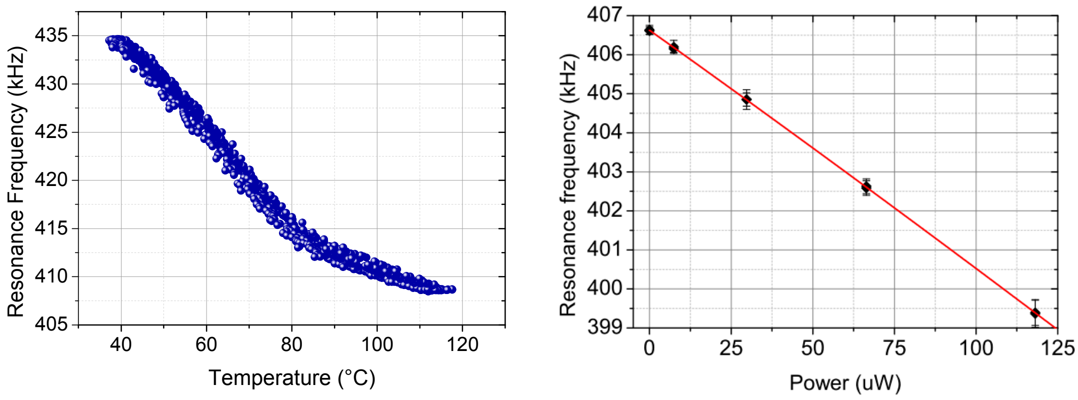


Figure 2. Optical microscope image of a typical LSMO FSM coated with a 30 nm thick gold layer to improve optical reflectivity. The detecting laser spot and the current/voltage pads are also visible.

3. Results

The measured resonance frequency of the FSM is higher than that expected considering an ideal homogenous double-clamped beam structure with no *built-in* stress (~60 kHz). This fact can be attributed to the stress induced by the slightly different lattice parameter of the deposited LSMO films with respect to the $SrTiO_3$ substrate and to the subsequent induced curvature of the thin freestanding film structures that both influence the flexural stiffness of the microbridges. We measure the mechanical eigenfrequency of LSMO FSM as a function of temperature by both heating the whole sample through a Peltier heater and heating the FSM only by Joule heating *via* an electrical current bias throughout the microstructure. In the first case (Figure 3a), we noticed an overall decrease of the resonance eigenfrequency with an inflection point in a temperature range compatible with the one reported in Figure 1, a possible sign of the phase transition occurring in our LSMO FSM. We believe that the observed monotone decrease of the eigenfrequency is linked to the stress relaxation of the pre-stressed microbridge upon the increase of temperature.



(**a**) (**b**)

Figure 3. Resonance frequency (first flexural mode) of a LSMO bridges of dimensions 150 × 15 × 0.3 μm^3 measured in ambient pressure conditions as a function of (**a**) external temperature (cycle ≈ 100 °C → 40 °C → 120 °C in about 12 min); (**b**) dissipated Joule power calculated as I^2 × R, where R is the electrical resistance probed across the microbridge by four probe method. Linear fitting function is reported in red. Sample stage is kept at ambient temperature.

The resonance frequency versus applied power dissipated in FSM by Joule heating is reported in Figure 3b. In order to compare the two measurements, we estimate the temperature induced inside the microbridge by finite element analysis (Comsol Multiphysics 4.3b). The temperature profile along the FSM is maximum at the centre due to the presence of the pads that act as heat sinks, as reported in the inset of Figure 4. Figure 4 shows the measured resonance frequency of Figure 3b reported as a function of the simulated average and maximum temperatures of the microbridge.

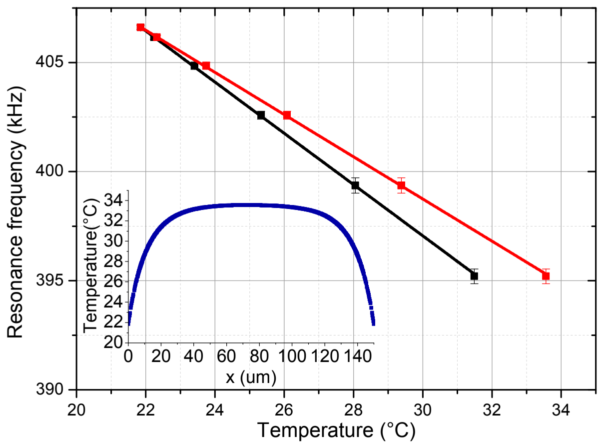


Figure 4. Resonance frequency (first flexural mode) of a LSMO bridges of dimensions 150 × 15 × 0.3 μm^3 obtained as a function of calculated maximum (red) and average (black) temperature applied by Joule heating. Inset shows the temperature distribution calculated along the length of the microbridge.

In order to compare the frequency shift obtained by Joule and uniform heating, we calculated the normalized temperature frequency shift defined as

$$\kappa_{T,n} = \frac{\Delta f}{\Delta T} \cdot \frac{1}{f_n}, \quad (1)$$

where f_n is the eigenfrequency of the system at the chosen temperature of 40 °C. $\Delta f/\Delta T$ is evaluated by a linear fit of the data reported in Figure 4 and in the range 40–60 °C of Figure 3a, where the frequency behavior can be approximated as linear. The value of $\kappa_{T,n}$ for uniform heating, calculated from the measurements of Figure 3, is $\kappa_{T,n} \cong 1 \cdot 10^{-3} K^{-1}$. In the case of Joule heating we obtain $\kappa_{T,n} \cong 3 \cdot 10^{-3} K^{-1}$ (average temperature) and $\kappa_{T,n} \cong 2.5 \cdot 10^{-3} K^{-1}$ (maximum temperature).

4. Discussion

In this paper, we presented preliminary mechanical characterization of LSMO FSM as a function of temperature in view of thermometric applications. We showed how the resonant frequency of these microstructures is influenced by temperature possibly via stress relaxation mechanisms due to thermal expansion of the constituent materials and to the change of the physical properties of LSMO itself across its phase transition. The observed difference in the mechanical response with uniform and Joule heating can be accounted to the different stress relaxation distribution occurring during the heating process or to different pre-stress conditions of the microbridge. Further investigations are needed to clarify these issues and settle the feasibility of a new generation of thermomechanical sensors based on LSMO.

Acknowledgments: We acknowledge University of Genova through "Progetti di Ateneo" and PRIN 2010NR4MXA "OXIDE".

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Pellegrino, L.; Biasotti, M.; Bellingeri, E.; Bernini, C.; Siri, A.S.; Marré, D. All-oxide crystalline microelectromechanical systems: Bending the functionalities of transition-metal oxide thin films. *Adv. Mater.* **2009**, *21*, 2377–2381, doi:10.1002/adma.200803360.
2. Manca, N.; Pellegrino, L.; Kanki, T.; Yamasaki, S.; Tanaka, H.; Siri, A.S.; Marré, D. Programmable mechanical resonances in MEMS by localized Joule heating of phase change materials. *Adv. Mater.* **2013**, *25*, 6430–6435, doi:10.1002/adma.201302087.
3. Haghiri-Gosnet, A.-M.; Renard J.-P. CMR Manganites: Physics, thin films and devices. *J. Phys. D Appl. Phys.* **2003**, *36*, R127–R150, doi:10.1088/0022-3727/36/8/201.
4. Liu, S.; Guillet, B.; Aryan, A.; Adamo, C.; Fur, C.; Routoure, J.-M.; Lemarié, F.; Schlom, D.G.; Méchin, L. $La_{0.7}Sr_{0.3}MnO_3$ suspended microbridges for uncooled bolometers made using reactive ion etching of the silicon substrates. *Microelectron. Eng.* **2013**, *111*, 101–104, doi:10.1016/j.mee.2013.02.024.
5. Le Bourdais, D.; Agnus, G.; Maroutian, T.; Pillard, V.; Aubert, P.; Bachelet, R.; Saint-Girons, G.; Vilquin, B.; Lefeuvre, E.; Lecoeur, P. Epitaxial manganite freestanding bridges for low power pressure sensors. *J. Appl. Phys.* **2015**, *118*, 124509, doi:10.1063/1.4931885.
6. Ceriale, V.; Pellegrino, L.; Manca, N.; Marré, D. Electro-thermal bistability in $(La_{0.7}Sr_{0.3})MnO_3$ suspended microbridges: Thermal characterization and transient analysis. *J. Appl. Phys.* **2014**, *115*, 054511, doi:10.1063/1.4864222.
7. Manca, N.; Pellegrino, L.; Marré, D. Reversible oxygen vacancies doping in $(La_{0.7}Sr_{0.3})MnO_3$ microbridges by combined self-heating and electromigration. *Appl. Phys. Lett.* **2015**, *106*, 203502, doi:10.1063/1.4921342.