Resonant Langasite Microsensor for Atomic Force Microscopy

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***Abstract*— Quartz length-extension resonators have already been used to get atomically-resolved imaging by frequency-modulation atomic force microscopy. New piezoelectric materials such as Langasite could be appropriate for this application. Theoretical study is reported on length extension resonators in this material. In this paper, an attempt to fabricate micro resonators in Langasite temperature-compensated cuts is prospected. The pointed tip of the micromachined cantilever can be used for atomic force microscopy applications.**

I. Introduction

The main component of an atomic force microscope (AFM) is the force sensing cantilever (CL). There are three parameters that can be used to characterize a CL: the stiffness k, the eigenfrequency f_0, and the quality factor Q. When atomic resolution in vacuum is desired, the preferred method is frequency modulation AFM. The spring constant k and the quality factor of quartz tuning forks have motivated the attempt to achieve atomic resolution [1]. Piezoelectric tuning forks have the advantage of self sensing [2]. The use of optics is not necessary and operation in ultrahigh vacuum and low temperatures are easily implemented.

Materials such as Langasite (LGS), Langatate (LGT) and Gallium Orthophosphate ($GaPO_4$) crystals are more piezoelectric than quartz crystal and they also have a much better temperature behavior. It has been shown that a temperature-compensated cut exists for $GaPO_4$, LGS and LGT length-extension resonators [3-6]. Consequently LGS crystal could be used to replace quartz crystal in several applications. For instance, quartz length-extension resonators (LER) have already been used to get atomically-resolved imaging by frequency-modulation atomic force microscopy [7-8]. Properties of the LGS devices are compared with quartz crystal's ones. LGS crystal belongs to the same crystal class as quartz (32) and its chemical composition is $La_3Ga_5SiO_{14}$. In order to miniaturize the resonators in LGS temperature-compensated cuts, we attempt to use chemical etching method [9]. Until now, only few studies on such etching of LGS were reported in the literature [10-11]. Most of the studies were devoted to polishing [12] and there are few informations on the opportunity of wet etching to manufacture resonant devices.

II. Theory

A. Temperature-compensated cuts for resonators

An analytical model (AM) of length-extensional vibration has been built [3]. In this model, resonant frequencies f_n are determined from the equation of motion. For a free-free beam, we get:

$$f_n = \frac{n}{2 \cdot l} \frac{1}{\sqrt{\rho \cdot s_{22}}} \quad (1)$$

where n is an odd positive integer, l the length of the beam, ρ the mass density of the material and s_{22} the compliance along the Y axis.

Thanks to this model, the theoretical temperature-compensated cuts of LGS could be determined. For a X-cut, the angles of the temperature-compensated cuts are $\theta = -7.1°$ and $\theta = 61.9°$ assuming that the angle θ is the angle of rotation around X-axis. $\theta = 0$ coincides with a beam aligned along Y-axis. There is no real first order temperature-compensated cut for quartz crystal. Experimental proof of the existence of the LGS compensated cut for length extensional mode in rectangular beam has been given in [5]. Measurements have shown that the 2^{nd} order frequency-temperature dependency is smaller for the angle $\theta = -7.1°$.

B. Sensitivity of the FM-AFM

The frequency modulation AFM operates in non-contact mode. Classically, the piezoelectric rod is excited in order to vibrate at its resonant frequency. The frequency shift of the resonant frequency is used as the feedback signal for the tip-surface interaction. The system can be characterized by the oscillating current. The sensitivity S_{cal} of the current to the displacement of the frequency-modulation atomic force microscope is given by [8]:

$$S_{Cal} = 4\pi \cdot f \cdot E \cdot d_{12} \cdot t\left(1 - \cos\frac{l_e \pi}{2l}\right) \quad (2)$$

where f is the LER resonant frequency, E the Young's Modulus in the beam direction, d_{12} the used piezoelectric coefficient, t the thickness and l_e the half length of the electrode (1.1 mm [8]).

Table 1 gives a comparison of LGS and Quartz crystal characteristics. The Young's modulus E is also 1.5 times higher in LGS. LGS resonators should have a sensitivity three times better than the quartz crystal ones because of the piezoelectric coefficients.

TABLE I. COMPARISON OF QUARTZ AND LANGASITE LER CHARACTERISTICS [13].

LER: 2.76 mm×70 µm×130 µm	Quartz		LGS	
θ [degrees]	5°	0°	-7.1°	61.9°
E [10^{10} N/m²]	8.47	7.87	12.67	14
d_{12} [10^{-12} C/N]	2.22	2.3	5.54	3.84
S_{cal} [nA/nm] ref [8]	227	212	705	568

III. EXPERIMENTS: LENGTH-EXTENSION MICRO-RESONATOR FOR AFM

A. Design of the masks

LGS monocrystal is commercially available as square wafers with a size of 1.5 in., which makes it useful for bulk micromachining and batch fabrication. The fabrication of the resonator is performed on an X-cut wafer because, as seen in *§ II-A,* there is a temperature-compensated orientation for this cut.

Fig. 1 shows the disposition of the different LERs in the Chromium-glass mask. The length of the beam is oriented by the angle θ from Y orientation, with θ not exceeding +/- 10°.

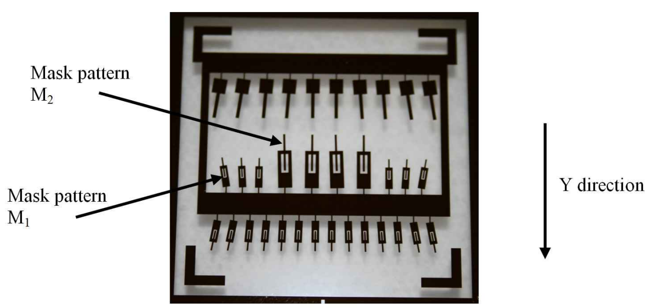

Figure 1. Chromium-glass mask.

The dimensions of the beams are assumed to be 1 300 µm for the half length and 100 µm for the width of M_1 LER. The dimensions of the resonator M_2 are 2 times greater. The dimensions on the mask are slightly greater to take into account the under-etching.

B. Fabrication

The complete fabrication of resonators passes through a standard photolithographic process that includes:

- cleaning of samples
- electrode deposition
- masking process
- spinning and patterning of the photoresist
- mask opening.

Fig. 2 shows the design of the LERs before the chemical etching.

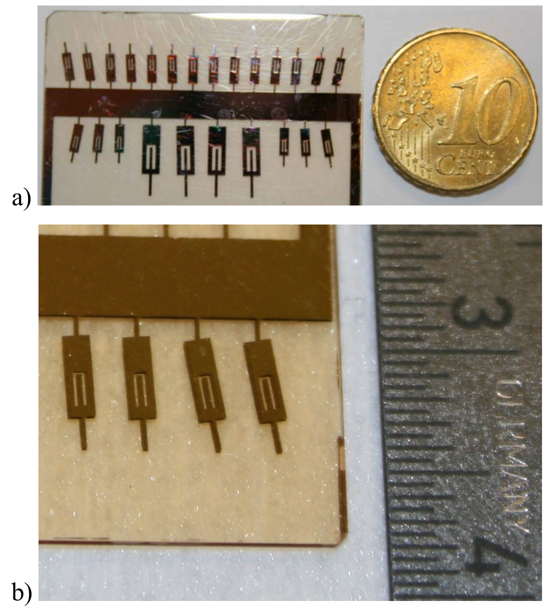

Figure 2. a) LGS plate resulting from the photolithographic process, b) Details of small LER patterns.

An HCl based solution has been chosen to perform the chemical etching [10]. This choice has been made based on the etch ratios of such a solution as well as the fact that this acid doesn't induce film formation at the surface of the LGS samples.

C. Results of the fabrication

Fig. 3 shows the LERs after chemical etching. The 225 µm thick plates were left in an isotherm HCl bath for 6h30 at a controlled temperature of 60 °C.

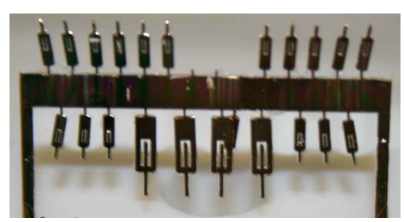

Figure 3. LER's after wet etching.

1) Shape

It was noted that the hole in the middle of the small LER's (M_1-type) could not be opened. A dissymmetry between the two halves of the beam was also observed. It will lead to the design of a second version of the mask.

Fig. 4a presents a LER of M_1-type. The graduations seen in Fig. 4a are in millimeters. The half beam included in the holder is not free. In fact, the half beam and the holder are linked together by a thin LGS layer. Fig. 4b is a scanning electron microscope image of a M_2-type LER.

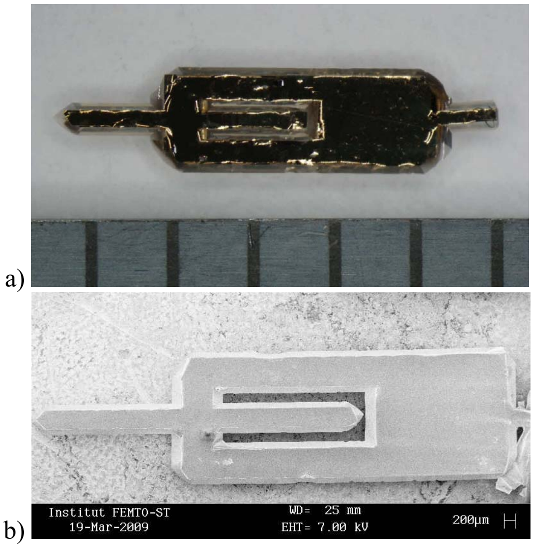

Figure 4. a) M_1-type LER (not open), b) SEM image of an M_2-type LER.

Details of the chemical etching are presented in Fig. 5. A marked difference occurs between the mask pattern and the final shape of the structure. The changes in shape of the micromachined resonators are due to the anisotropy of the chemical etching. Etched structures are bounded by cristallographic planes that possess the smallest dissolution rates. Consequently, the lateral sides of the beams are not constituted by vertical walls but by inclined blocking facets and the cross-sections of the beams are then not squares.

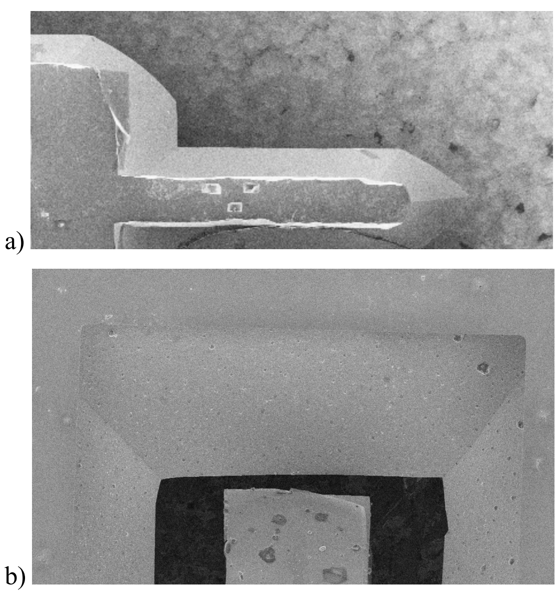

Figure 5. a) Blocking facets, b) Facets instead of vertical walls.

2) Tip

Due to the fact that the etching is anisotropic, a tip is obtained at the end of the beam (Fig. 6). This could be used as an AFM-tip or for scanning microdeformation microscopy [14-15].

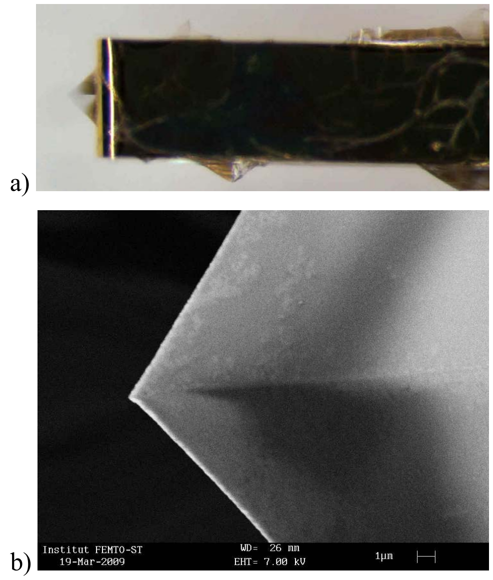

Figure 6. a) Close up on the tip of a LER, b) SEM image of the tip.

3) Difficulties

The wafer cleaning and the electrode achievement are critical steps of the process. Holes in the electrodes will cause etch pits at the surface of the structure (Fig. 7).

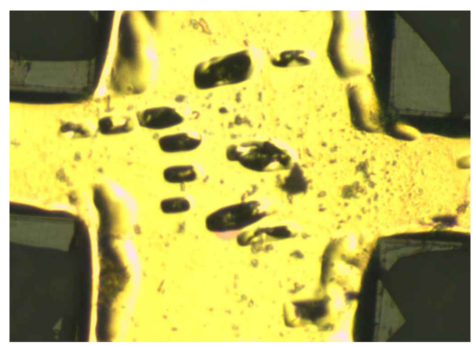

Figure 7. Etch pits.

Electrode ungluing during the etching process can lead to irregular edges as seen in Fig. 8.

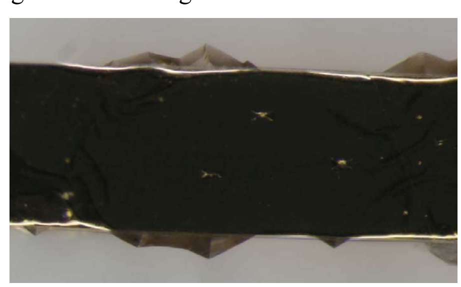

Figure 8. Irregular edges.

D. *Experimental measurements of the frequencies*

Fig. 9 presents the amplitude of the impedance according to the frequency obtained using a network analyzer. The eigenfrequencies of the LERs have been measured at room temperature. Several modes of vibration of an M_2-type LER are observed in the frequency range.

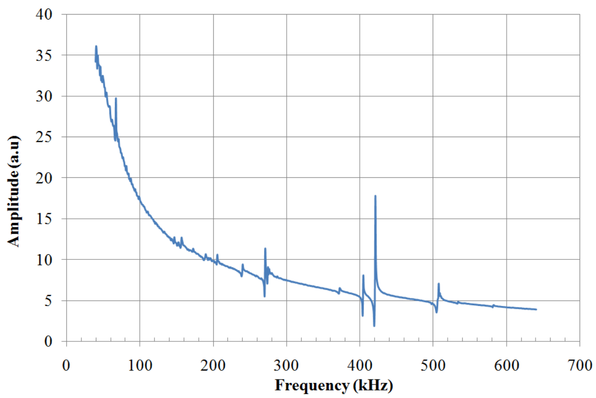

Figure 9. Spectrum for an M_2-type LER.

The resonant frequency of the 1st length extensional mode is at about 420 kHz which is in agreement with the analytical model result (Fig. 10). The theoretical calculus for a beam with 5.5 mm long gives a frequency of about 412 kHz. This frequency shift can be explained by the beam shape and the clamping conditions.

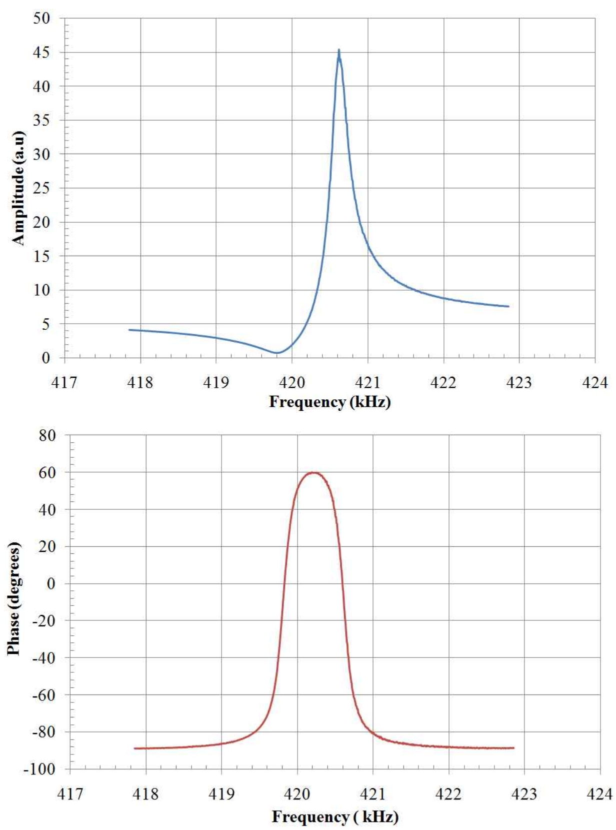

Figure 10. Amplitude and phase at the eigenfrequency for an M_2-type LER.

The motional parameters and the quality factor are given in table 2. The measurements have been obtained at atmospheric pressure and room temperature. The quality factor is about 2 000.

TABLE II. MOTIONAL PARAMETERS AND Q FACTOR OF AN M_2-TYPE LER.

F (kHz)	420
R (Ω)	7 675
L (H)	6
C (fF)	24
Q	2 070

Fig. 11 presents the amplitude of the impedance of an M_1-type LER. The 1st length extensional mode is obtained around 900 kHz. Because of the link between the half beam and the holder (see § III), this vibration frequency corresponds to a clamped-free beam mode. The considered length of the beam is then equal to 1.3 mm. Theoretical and experimental frequencies are in good agreement. The quality factor of the M_1-type resonator is around 1 000.

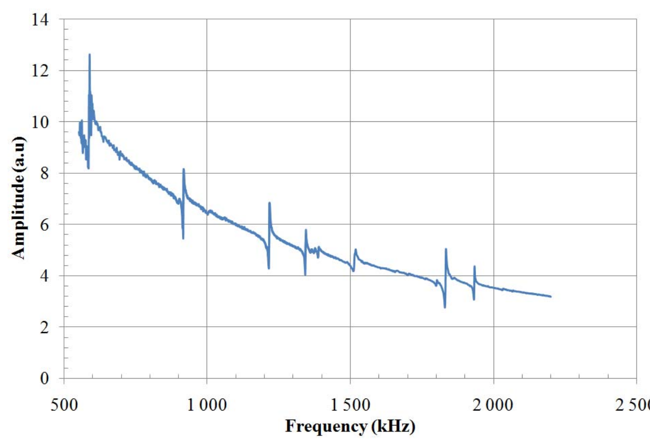

Figure 11. Amplitude spectrum for an M_1-type LER.

IV. CONCLUSION AND PERSPECTIVES

The feasibility of original vibrating length-extension resonators in LGS crystal by chemical etching has been demonstrated. Theoretical and experimental frequencies are in good agreement.

This contribution shows that LGS is a new promising material in order to obtain tips for near field microscopy applications.

Some future technical and theoretical works could be investigated to improve the performances of the LERs. A second version of the mask has to be designed considering the dissymmetry of the beam lengths, the blocking facets and the tip. A finite element model that would take into account the exact shape of the structure and the tip should be achieved to obtain more precise results for eigenfrequencies and motional parameters. The dimensions of electrodes have to be optimized to eliminate the parasite modes.

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