MECHANICAL PROPERTIES STUDY OF SILICALITE MICROCANTILEVERS

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Zeolites constitute a family of highly interesting technological materials, on account of their framework structure, with pores of subnanometric size, and their remarkable properties in catalysis and adsorption [1]. Given the interesting properties of zeolite coatings, a significant effort has been carried out to combine the experience gathered in growing zeolite films and the fabrication methods used in the electronic industry to prepare microstructured supports, especially on Si substrates [2].
The final target of this work is the development of a highly selective and sensible nanoporous resonator for the early detection of explosives. The specific and tunable adsorption properties of zeolites combined with their chemical, thermal and elastic properties make them suitable candidates as sensible coatings. However, the mechanical properties of well-intergrown zeolite polycrystalline membranes must also fulfil the transducer requirements in order to develop a resonator with a good quality factor. Indeed, the resonant behaviour of zeolitic layers has not been previously attempted in the literature.
In this work c-oriented silicalite (Sil-1) polycrystalline layers have been synthesized on Si wafers and used as structural layers for micropatterning [3] to develop bulk Sil-1 cantilevers (see Figure 1). To the best of our knowledge this is the first time that a zeolite-only cantilever is proposed. Then, the resonance of these cantilevers excited with a piezoelectric material has been optically detected and characterized. Table 1 compiles the experimental results obtained. From these data, the intrinsic value of Young Modulus for well defined uncalcined SIL-1 polycrystalline cantilevers have been estimated (30.09±5.29GPa).
The microporous framework structure release by the organic template removal has been attempted by several methods. In addition to standard calcinations at 480ºC under controlled atmosphere, ozone oxidation, oxygen plasma, and sulphuric acid leaching has also studied. Thermogravimetric analysis (see Figure 2) of free standing Sil-1 membranes indicates that O_3 oxidation at 200ºC during 6 hours enables the complete organic removal. SEM analysis is being carried out to ensure the structure integrity (see Figure 3), before the estimation of the new young modulus.

Acknowledgments
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References:

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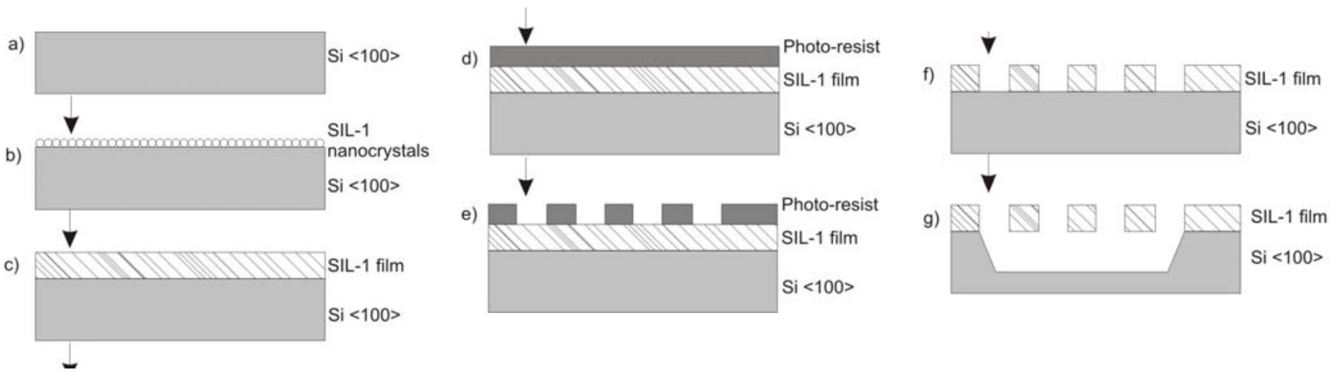

Figure 1. a) Starting Si wafer; b) spin coating of SIL-1 nanocrystals (4% wt. in ethanol); c) hydrothermal synthesis of SIL-1 film; d) deposition of a TI-35 ES reversal photo-resist; e) UV photolithography, reversal bake process and resist development, f) BHF etching of the SIL-1 layer; g) TMAH etching of Si underneath and bulk Sil-1 500 μm length cantilever released as shown in the SEM micrograph.

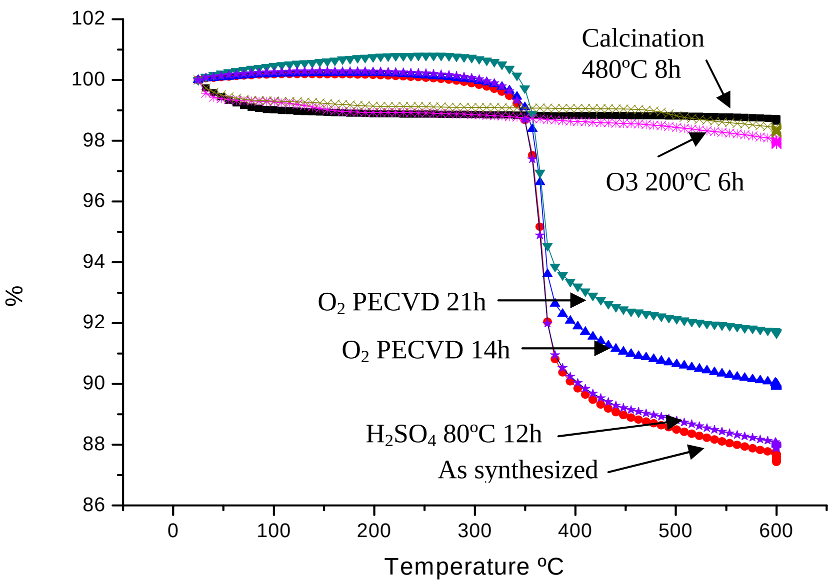

Figure 2. Thermo-gravimetric analysis of SIL-1 free standing membranes subjected to different organic template removal methods

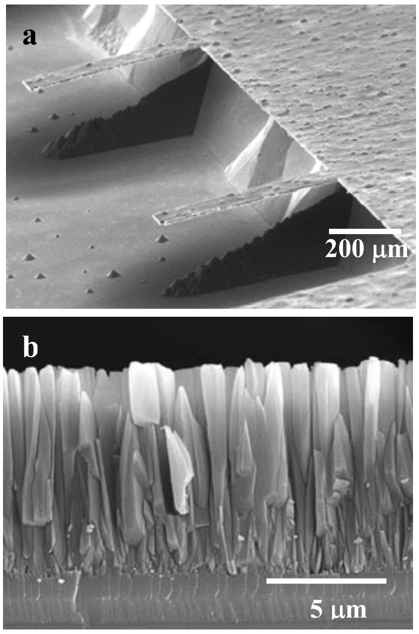

Figure 3. (a) As synthesized μcantilevers 500 μm length. (b) Sil-1 layer, crystal orientation "c".

Table 1. Resonant frequency measurements for cantilevers resonating perpendicular to the SIL-1 surface (parallel to "c" crystallographic axis).

Sample	Length (μm)	Width (μm)	Thickness (μm)	f_0 (kHz)	Young Modulus GPa
1	500	90	8	18.03	30.27
2	1000	90	8	4.83	34.75
3	400	90	8	28.80	31.61
4	1000	60	8	4.54	30.75
5	400	60	8	26.60	26.97