FABRICATION OF AN ARRAY OF SILICON MICROSCALES FOR THE MONITORING OF CHEMICAL PROCESSES

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***Abstract* — We present the fabrication and demonstration of a two-dimensional array of scales micromachined in silicon. Each scale consists of a platform suspended by a spring. The mass on each scale is measured from a distance by a camera, which is imaging the fringe pattern that arises from optical interference in the gap under each spring scale. A diffractive lens at the bottom of the gap separates the fringe signal from unwanted reflections and directs this signal towards the camera. When a mass deflects a scale down, the gap narrows and the fringe pattern is getting tighter. Operation at temperatures as high as 700 C was demonstrated, which makes the device useful for the monitoring of high temperature chemical processes.**

Keywords : microscale, optical MEMS, diffraction.

I – Introduction

Some chemical processes can be monitored by measuring the mass of a recipient containing a liquid in a reaction with a gas, which is called gravitometry. Often this occurs in a reaction chamber or reactor, at high temperatures, where traditional methods for reading the position of a micromechanical sensor cannot be used (such as piezoresistors or electrostatic readout).

In this article, we demonstrate an array of microscales that can be read out optically and at a distance from the microscales themselves. The electronic component (consisting in this case of a light source and a camera) are placed outside the reaction chamber where the microscales are placed. This way the chamber can be warmed to several hundreds of degrees C without impairing the functioning of the electronics. Furthermore, such an optical readout setup can be realized with off-the-shelve components.

Another difficulty associated with operation at high temperatures is that bimorph stress will bend micromechanical structures consisting of several layers of different materials. This has the adverse effect of displacing the scale platforms, giving a wrong readout of the mass on each scale. To solve this problem we have fabricated a structure consisting exclusively of silicon and silicon oxide.

II –Operation principle

The construction consists of two parts with different functionalities: a mechanical part that is micromachined in a silicon-on-insulator wafer, and an optical part that is micromachined on a standard wafer. The two wafers are bonded together by means of high temperature fusion bonding.

The mechanical part of the scale consists of a centre platform, suspended by an annular spring, as shown in Figure 1. Each scale has an axial symmetry, and the spring is simply a thinner silicon torus around the platform. Increasing the mass placed on a platform bends the annular spring as the platform moves down. The optical wafer consists of 2-level diffractive lens etched in silicon. The optical wafer was thinned in some parts just under the scales, in order to limit the attenuation of light through silicon. The back of the spring is separated from the diffractive lens by a 8 µm gap, in order to allow vertical deflection of the platform-spring structure.

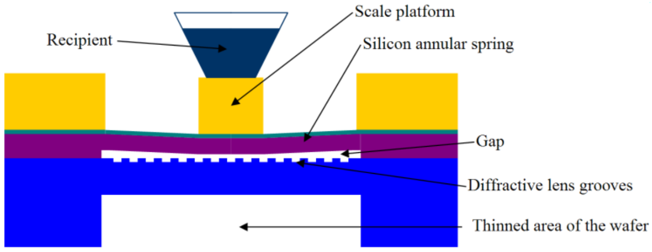

Figure 1. The different parts of a scale element: The optical wafer is shown in blue. The mechanical wafer consists of three layers, a 300 µm thick silicon substrate (yellow), a thin oxide layer (green) and a 35 µm thick silicon device layer (purple).

The basic principle is that increasing the mass in a recipient moves the underneath platform down, causing a progressive reduction of the gap situated under the scale platform and its spring. The principle for optical readout is very similar to that used to read the position of the microphone membrane described in [1].

Basically, the gap acts as a Fabry-Perot cavity where optical interference occurs. The light intensity reaching the camera from a place just under a scale depends on 1) the intensity of the light reflected by the Fabry-Perot cavity at this location and 2) the diffraction efficiency of the diffractive lens, both of which changes with the gap length. Thus the camera sees a fringe pattern consisting of concentric rings under each scale. This simple optical setup ensures a good contrast in the images, thanks to the diffractive Fresnel lens that separates the fringe signal from unwanted reflections and directs it off-axis towards the camera. When the scale platform moves down, the gap gets narrower in the middle of the scale, and the density of the rings increases, as illustrated in Fig. 2.

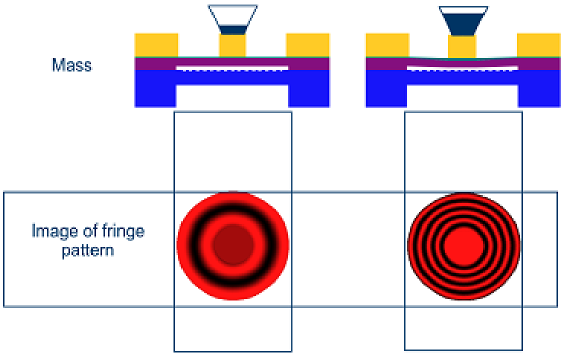

Fig. 2. The left scale has a low mass on top of it, and the annular spring is little bent. The scale shown on the right is loaded with a higher mass, bending the spring more and resulting in a fringe pattern underneath consisting of tighter rings.

III – Measurement setup

The measurement setup consists of a reaction chamber enclosing an array of recipients, each standing on its own scale, as shown in Fig. 3. A 4x4 scale array fabricated in silicon can be seen in Fig. 4. The vertical deflection of the scales is measured optically, by imaging the fringe pattern under each scale. The scale array is lit by a quasi-monochromatic light source, such as a LED. The fringe patterns are imaged by a camera, which is placed outside the reaction chamber. The camera can either be a standard and relatively inexpensive CCD, but for operation at temperature higher than 400° C, we will see that an InGaAs camera is required. Note that the light source and camera “see” the scale array through a window as they are placed outside the high temperature chamber.

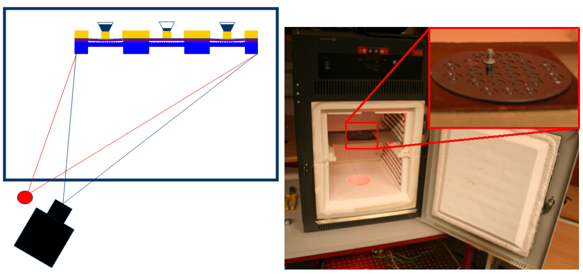

Fig. 3. Measurement setup (left): The light source is shown as a red dot, just over the camera, under the reaction chamber. The actual setup was enclosed in an oven with an opening under (right), and the scales were loaded with objects of known mass (such as the screw in the picture).

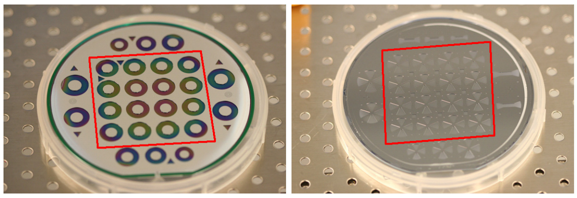

Fig. 4. Pictures of the fabricated devices: On the mechanical side (left), the scale platforms can be seen as circular areas enclosed by annular springs, while on the optical side (right), parts with a clover shape of the wafers were thinned. The red parallelepipeds represent the 4x4 scale array, while the structures outside were for test purposes only, and will be sawed away in a final version of the device.

IV – Fabrication

We have fabricated arrays of micro scales with a pitch of 13 mm, so that 16 scales fit on a 4-inch wafer, as seen in Fig. 4. If more scales are needed, it is possible to increase the wafer size or to assemble several sawed wafers side by side.

The array of scales were fabricated using fusion bonding of two silicon wafers, a silicon-on-insulator wafer on which was implemented the mechanical part of the scale, and a standard silicon wafer with the optical function of the device. The process was a combination of dry etches with different depths, in addition to the fusion bonding itself.

The micromachining process can be seen in Fig. 5. The mechanical wafer is a Silicon-on-Insulator wafer consisting of a 380 µm silicon substrate (shown in yellow in the figures) and a 43 µm silicon device layer (purple in the figure). Between the two silicon layers there is a 5 µm thick buried oxide layer. The optical wafer is a standard 300 µm thick double-sided polished silicon wafer. The first step of the process is to etch, using Reactive-Ion-Etching (RIE) technique, a gap under each scale. This gap defines the length of the Fabry-Perot cavity when no mass is present on the scale, and was chosen to be 8 µm. In parallel, the diffractive Fresnel lens is etched on the surface of the optical wafer, also using RIE. The two wafers are then bonded together with fusion bonding, which consist in aligning and pressing the two wafers in contact, before annealing the stack at 1050º C. The next step is to etch with deep RIE the substrate of the mechanical wafer, in order to define the platform of the scale. The resulting platform is suspended by the thinner device layer, which acts as a spring. Finally, the optical wafer is thinned to 100 µm using deep RIE. Not the whole area under the scale is thinned, but rather a clover shaped area, in order to keep as much structural strength as possible to the wafer stack, while giving a reasonably large “window” for imaging the fringe pattern under the scale.

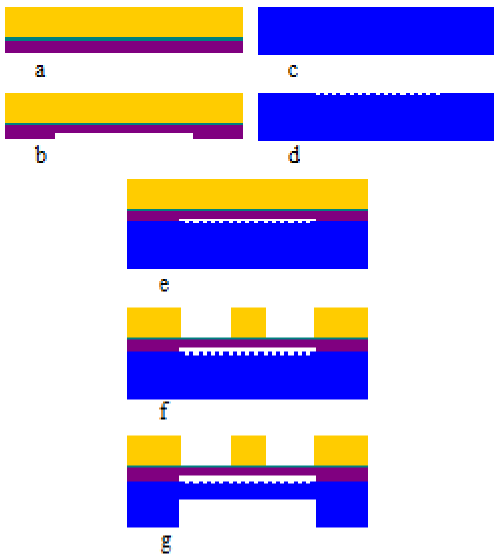

Fig. 5. Fabrication of the scale, seen as the cross-section of one scale element. Dimensions are not to scale. (a) The mechanical wafer. (b) Dry etching of a 8 µm gap in the device layer. (c) The optical wafer is a standard double-side polished wafer. (d) The diffraction pattern is dry etched onto the surface of the optical wafer. (e) The two wafer are aligned and bonded. (f) Deep Reactive Ion Etch defines the scale platform and spring area. (g) Thinning of the optical wafer with Deep Reactive Ion Etch.

V – Experimental results

A. Measurement using a CCD camera

In a first experiment, we used a CCD silicon camera to image the fringe pattern at the backside of the scale array. An image of the backside of the whole scale array can be seen in Fig. 6. The light source consisted of a halogen lamp connected to a fibre bundle, whose other end acted as a point source for illumination of the backside of the scale array. A band pass filter centred at 1067 nm was used to reduce the bandwidth of the illumination to 20 nm. The illumination wavelength was chosen to be at the edge at the silicon bandgap, so that light was still able to propagate without too much attenuation through the thinned parts of the optical wafer (100 µm thick), while remaining in the wavelength region where the CCD is sensitive to light. A wavelength of 1067 nm might not be ideal for this purpose, but was the closest available wavelength without having to order a custom filter.

Unfortunately, when the temperature of silicon increases, the edge of the bandgap moves towards longer wavelengths. We obtained good images of the fringe pattern at 200°C, even through the whole thickness of the optical wafer (ca. 300 µm), and at up to 400°C through the 100 µm thinned parts of the optical wafer. But above 400°C, transmission through silicon was too low to be able to extract the fringe pattern from the background noise. This is illustrated in Fig. 7.

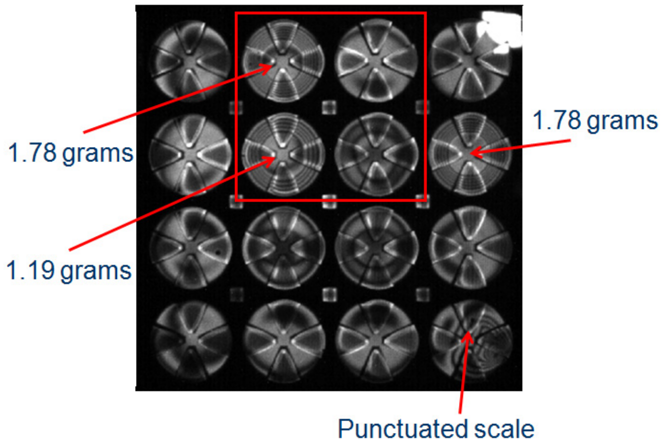

Fig. 6. Backside of the scale seen from the CCD camera, with different weights placed on several scales.

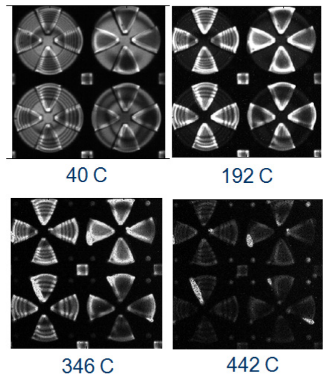

Fig. 7. A subset a 4 scales seen as temperature is increased from 40°C to over 400°C.

An automated program was used to count the number of fringes under each scale. The basic principle is to extract intensity curves from cross-sections of the fringe pattern, and to count the number of peaks and valleys. Such a measurement is shown in Fig. 8, where a mass of 2.38 grams gave 9 rings, that were counted with a resolution of 5 pixels per period. Only counting the number of peaks and valleys gives only a limited precision of the mass measurement. This can be improved by measuring, in addition, the lateral position of the peaks and valleys, even though this was not implemented in this experiment.

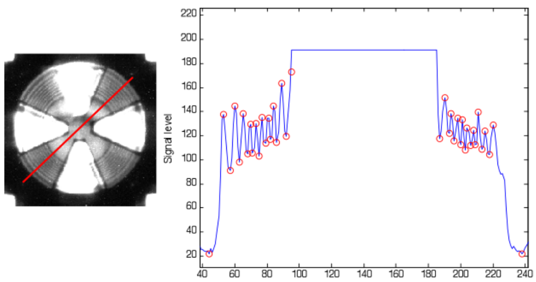

Fig. 8. Automated measurement of the number of rings in the fringe pattern under a scale. The signal level along the red line is plotted on the right graph.

In order to investigate the linearity and the cross talk between neighbouring scales, we have taken images of a subset of four scales while varying the mass on one of them, as shown in Fig. 9. We observed in the images taken no visible cross talk between neighbouring scales. In addition, the relation between the number of rings and the mass placed on a scale showed a good linearity, as shown in Fig. 10.

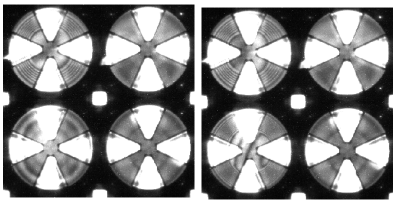

Fig. 9. Subset of four scales, with a constant weight of 1.78 grams on the upper left scale and a varying weight on the lower left scale: 0.55 grams (left) and 3.34 grams (right).

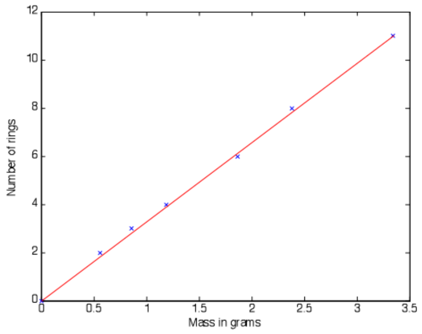

Fig. 10. Number of rings counted manually versus mass of objects.

B. High temperature measurement using an InGaAs camera

In order to increase the maximal temperature at which the fringe pattern can be imaged, we have used an InGaAs camera and an illumination at 1330 nm. This wavelength is not absorbed by silicon, while detected by the NIR-sensitive camera.

Because of the available camera lens, we had to place the scale array much closer to the camera lens (25 cm instead of the designed 40 cm) to be able resolve the rings in the fringe pattern. As a result, the diffractive lens was not focusing light on the camera aperture for all scale elements, and only light from parts of the scale array could be observed at the same time, as can be seen in Fig- 11. This problem could easily be solved by using an appropriate camera lens with higher magnification.

With the InGaAs camera, fringe patterns could be observed at temperatures as high as 700º C. However, the contrast of the fringe becomes lower as temperature increases, because the heating elements of the oven produce a large background signal that extends in the NIR-region as the temperature increases. This background signal can be measured by turning off the illumination of the scale array, and then subtracted from the images obtained with the illumination on. But at roughly 700º C, the camera was saturated by the background light and the fringe pattern became invisible.

490 C 620 C 700 C

Fig. 11. Image of the fringe pattern taken with an InGaAs camera.

Another possible limitation in the temperature at which the scale array can be operated, is mechanical stability. Because the structures consist of several layers of silicon and silicon oxide, with different thermal expansion coefficients, bimorph forces build up as temperature increases, with the result of bending the structures on the wafer. As a consequence, the position of the scale platform is modified, and thus the observed fringe pattern. This phenomenon can clearly be seen in Fig. 11, where the number of rings decreases from 4 at 490º C to 2.5 at 700º C. A possible solution to this problem is to calibrate each scale at different temperatures. But a more ideal solution would be to attenuate the bimorph forces by reducing further the number of layers the scale array consists of. In our construction, we had an aluminum layer on top of the scale array, which was used as a mask for Deep Reactive Ion Etching of the scale platform. This layer could easily be removed at the end of the scale array processing. Similarly, there is an oxide layer on top of the annular springs, which also could easily be removed.

VI - Conclusion

We have fabricated and demonstrated an array of microscales that is read out optically from a distance and that can be operated at high temperatures. We have implemented a method that derives the scale deflection by counting the number of circular fringes under each scale. This method shows good linearity from 0 to 3.5 grams. We have not observed crosstalk between neighbouring scales. Using a standard CCD camera, we could operate the scale up to 400ºC. At higher temperatures, the silicon structure under the scales becomes opaque for short wavelengths and a near-infrared-sensitive camera must be used to be able to see the fringes. Using an In-Ga-As camera we demonstrated operation at up to 700ºC. At such high temperatures, both the thermal stability of the structures and the thermal emission from the chamber gradually degrade the performance of the system.

References

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