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In-situ optical monitoring of single-crystal silicon membrane etching

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Abstract We present a simple yet efficient technique to monitor membrane thickness during etching of silicon in anisotropic etching bath. This technique uses a mechanical holder to protect the front side of the wafer and the measurement of light absorption to obtain remotely the thickness of a reference zone in the etched wafer. The original feature in our set-up is that we measure the absorption in two different bands of wavelength, one where the silicon is strongly absorbing and the other where it is not, improving the robustness of the measurement. Actually, this principle allows for effectively compensating the fluctuation in the optical path, and after calibration provides real-time information on the membrane thickness, which proves to be particularly useful for fabricating membranes below 40 µm.

Keywords wet etching ; KOH ; membrane ; optical absorption

1 Introduction

Since the heydays of silicon micromachining, anisotropic chemical etching has been one of the favored way to produce silicon micro-systems (Petersen 1982). If this technique allows to easily control the lateral dimensions of the holes, using the (111)-planes that essentially behave as an etch-stop, it lacks an easy way to stop the etching along the depth for producing membrane with accurate thickness. Many techniques have been proposed to obtain this control, and they may be split in two categories: those introducing an etch-stop along the depth and those that don't. In the first category we find the boron diffusion (Bassous and Lamberti 1989) and the electro-chemical stop (Jackson et al 1981) techniques that use doping for introducing the etch-stop. This step complicates the process substantially and leave a doped silicon membrane that is undesirable in many devices. SOI wafers do not present this drawback and provide a clean etch-stop on the buried SiO_2 layer. These techniques have the advantage to fix the membrane thickness before etching occurs and will be useful for guarantying an accuracy even smaller that the total thickness variation (TTV) of the wafer.

However for cost or operational reason they can not always be used, and other processes have been developed that do not rely on modified silicon wafer. In that case, the accuracy of the thickness of the etched membrane is ultimately limited by the TTV of the wafer. Accordingly, a good etching control technique should allow an accuracy better than the TTV guaranteed by the wafer manufacturers, that is better than 5 µm for prime 100 mm wafers (SEMI TC 2013). The timed etch of Si wafer is the simplest process that does not use any physical mechanism to stop the etching. Its accuracy is limited by the uncertainty of the etching rate, and, for example, if the etching rate is known with an uncertainty of 2% or worse – as will commonly happen in a lab environment – after etching about 500 µm of silicon the error is at least 10 µm. For (100)-cut wafer, a refinement of the timed etch technique (Zhang 1998) uses stripes with side perpendicular to the <100>-direction to monitor the depth of the hole by measuring the overetching of the stripe. This technique greatly improves the accuracy of the etched depth by removing the uncertainty on the etching rate but is not very practical to implement because it requires the visual inspec-

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tion of the wafer with a microscope during the etching. To reduce the uncertainty of the timed etch it is possible to regularly monitor the etch depth using a contact or an optical profilometer. The depth measurement is very cumbersome because the sample has to be taken out of the KOH solution, rinsed and dried thoroughly for a safe and accurate contact or optical reading. After the measurement, replacing the cooled holder in the solution decreases its temperature affecting significantly the etching rate and making the accurate prediction of etching depth difficult. Finally we note that these techniques only measure the etched depth and they require an accurate knowledge of the wafer thickness to obtain the remaining membrane thickness. Intrinsically they are unable to compensate the wafer-to-wafer and lot-to-lot random deviation of the wafer thickness, forcing to precisely measure each wafer before etching. Otherwise, as the manufacturer tolerance are not so tight on wafer thickness, for example 100 mm prime wafers are usually given with a tolerance of ±20 µm (SEMI TC 2013), this would translate in similar error in membrane thickness.

To alleviate these problems, techniques have been devised for monitoring the real thickness of the etched membrane (not only the etch depth) and directly inside the etching solution to simplify the process. It is for example possible to use infra-red interferometry and study the fringes when the membrane thickness decreases (Tosaka et al 1995). However the set-up is complex, requiring a spectrometer, and moreover it was observed that the bubbles generated during KOH etching produced large noise for an accurate operation. A simpler approach use the absorption of visible light by the silicon to probe the membrane thickness (Stoller et al 1970). As the light intensity reaches a certain level we can stop the etching by removing the sample from the KOH. However this technique depends significantly on the source characteristics and its accuracy reaches ±2 µm (Stoller et al 1970) at best - and more commonly ±5 µm. An improvement on the source stability made use of a laser and a photodetector (Pons et al 1990), but it is plagued by a lot of noise coming from the bubbles and the presence of interference in the membrane making the technique difficult to use for in-situ monitoring. The absorption technique has been refined further by using a broad spectrum source and a spectrometer (Mescheder and Koetter 1998) but then it looses much of its simplicity and becomes expensive and complex to set-up. We propose a simpler yet effective method also based on absorption that can compensate for most source of noise while still being fast and affordable (Chollet and Hwai 2005).

2 Experimental set-up

Our idea is to improve the accuracy of the simple absorption measurement method by using a broad spectrum illumination and recording the light passing through the silicon membrane simultaneously in the visible part of the spectrum, where the silicon absorbs significantly, and in the near infra-red (NIR), where it absorbs very little.

The intensity, I, of the light crossing an attenuating layer depends on the thickness of the layer, h, following Bouguer's law (Tagirov and Tagirov 1997) at a particular wavelength λ:

$$I(\lambda) = I_0(\lambda)\ \exp\left(-\alpha_{\mathrm{Si}}(\lambda)\, h\right) \tag{1}$$

where $\mathrm{I}_0(\lambda)$ is the intensity of the light just below the surface of the layer and $\alpha_{\mathrm{Si}}(\lambda)$ the attenuation coefficient at the wavelength λ for the silicon. Because we are dealing with single crystal silicon, without scattering centers, the attenuation is mostly due to the absorption coefficient of silicon. This equation is valid for a single wavelength but in our case we would need to use an integral and take into account the absorption coefficient dispersion.

The equation revels that the intensity of the light measured after the layer (transmitted light) not only depends on the absorption inside the layer and its thickness, but also on the intensity $I(0)$. This intensity includes factors such as the intensity of the light source, the roughness of the layer surface, the layer index of refraction (Fresnel loss), and the medium outside the material between the source and the detector. Going a bit forward we may write $I(0)$ as a product of different factors $I(0) = I_0 \eta r \alpha_{med}$ where I_0 is the source light intensity, η the attenuation due to the light diffracted by the surface roughness, r the reflection at the layer surface, and α_{med} the attenuation due to the media present in the optical path (etching bath, waveguide, bubbles...). A first trial shows quickly that simply using the transmitted light intensity for monitoring the layer thickness is not satisfactory because most of the factors vary with time making the transmitted intensity a poor variable for monitoring the layer thickness. A good solution for alleviating this problem is to use a reference signal that goes through the same optical path as the measurement signal and will thus experience much of its fluctuation while it is little absorbed by the material. Then taking the ratio of the two signals would remove most of the environmental noise. Actually, for silicon it is possible to use a signal in the visible spectrum that will show strong absorption, while a signal in the NIR spectrum will show very little. In that case, the ratio of

the detectable signal may be written as :

$$\frac{I_{\mathrm{Vis}}}{I_{\mathrm{NIR}}} = \frac{A_{\mathrm{Vis}} \int_{\mathrm{Vis}} \Re_{\mathrm{Vis}} I_0 \eta r \alpha_{med} e^{-\alpha_{\mathrm{Si}} h} d\lambda}{A_{\mathrm{NIR}} \int_{\mathrm{NIR}} \Re_{\mathrm{NIR}} I_0 \eta r \alpha_{med} e^{-\alpha_{\mathrm{Si}} h} d\lambda} \quad (2)$$

where A_{Vis}, A_{NIR}, $\Re_{\mathrm{Vis}}$ and $\Re_{\mathrm{NIR}}$ are the gain and the responsivity for the visible and NIR photodetectors respectively. The interesting feature of our set-up is that most of the variables that vary with time in the equation are actually varying proportionally in the two wavelength bands. Actually, the main problem affecting α_{med} is with the bubbles that appear in the bath and perturb strongly the transmittance, mostly because of their lensing effect that will divert light from the photodetectors. This lensing effect is very similar with the two wavelength bands if they are not too far apart, and thus the refractive index of the KOH solution about the same. I_0 is affected by the current intensity fluctuations in the halogen lamp. However, measurement show that the fluctuation around a stable operating point affect the complete lamp spectra in a similar way Korhonen et al (2009), particularly when the fluctuation are not too large which can be guaranteed by a stabilized power source. Normally, the diffraction due to roughness η is highly dependent on wavelength (in some cases it depends with $1/\lambda^4$) and any change of roughness will affect differently both portions of the spectrum. However, during the etching the roughness of the silicon surface is remaining constant (save for a short time following the etching start) if the wafer has been properly prepared (Haimi and Lindroos 2003). In that case η is no more time dependent and only varies with the KOH bath used.

In the set-up, to measure the light going through the Si-membrane in two bands of wavelength, we shine it on a pair of photo-detectors. One photodetector is a Silicon photodiode sensitive from the beginning of the visible spectrum to about 1 μm, and the other is a GaInAs photodiode placed below the Si photodiode and thus responsive from about 1 μm to 1.7μm. The advantage of the stacked photodetector (K1713-09 from Hamamatsu Photonics) used in our set-up is double: it provides a single optical path and the Si photodiode effectively filters out all the light below 1 μm where the InGaAs is slightly sensitive. This later point provide intensity measurement in truly disjoint bands increasing the robustness of the method.

Actually, as the temperature inside the bath may exceed 90°C, we could not directly place the photo-detector close to the wafer. Thus, we have used a glass optical fibre to pick the light from the front side of the wafer and carry it outside the etching solution to the photodetector. A relatively large sensitive area is required to average the property of the etched surface and be less sensitive to local variation of roughness for example. Accordingly, a fiber with 1 mm core diameter has been chosen as a good compromise between collecting efficiency and stiffness for obtaining short bending radius. Polymer core fiber could have been used to increase the fiber compliance, however the large absorption in the NIR in polymer precludes its use here. We verified that the jacket of the fiber withstands the hot KOH solution without visible alteration for long period of etching. The additional advantage of the optical fiber is that it helps remove most of the stray light. Actually, the optical fiber acts as a spatial filter, only propagating the light that falls on its front facet within a solid angle defined by the numerical aperture NA= $n \cos\theta$. For the glass fiber used, NA=0.22, resulting in an acceptance angle of only 17˚ in water. We note that any motion of the fiber will change the coupling across the whole spectrum, affecting the two wavelength bands in essentially the same way. This feature makes the ratio of detected intensity robust to displacement of the fiber, facilitating the use of the set-up.

The holder is designed to etch a single-crystal silicon membrane from the backside (Kung et al 1991). The micromachined structures on the front-side, which are usually highly sensitive to KOH (e.g., polysilicon or metals), are protected by a mechanical holder that leaves access at the back for the etchant. The holder is built of high performance PEEK polymer and it includes a backside illumination with a tungsten-halogen lamp. The lamp emits light from the lower end of the visible spectrum to the NIR. The broad spectrum of the source will help decrease the effect of Fabry-Pérot in-

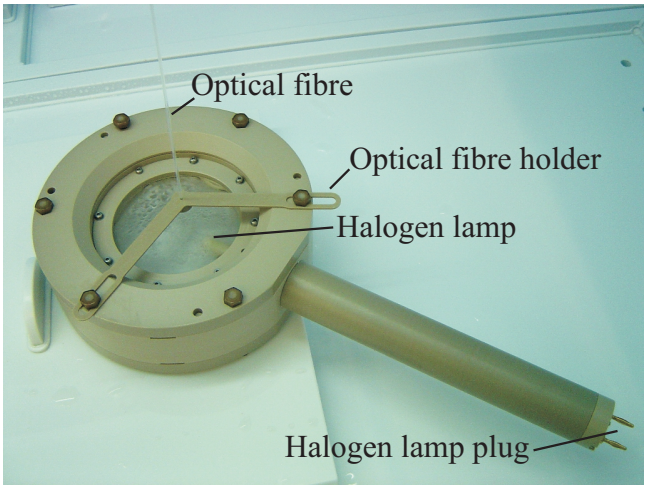

Fig. 1 Picture of the holder with the fiber-optic for optical monitoring of membrane thickness. The holder is a modified version of a commercial system sold by AMMT Gmbh.

terference when the membrane becomes thin. The commercial holder has been fitted with an adjustable fiber holder to keep the end of the optical fiber in front of the wafer as shown in Figure 1.

The choice of the different elements in the set-up is strongly dictated by their spectral characteristics shown on the diagram in Figure 2, where we have figured the emission spectrum of the lamp and the dispersion of the absorption of Si and of the detectivity of the photodetectors.

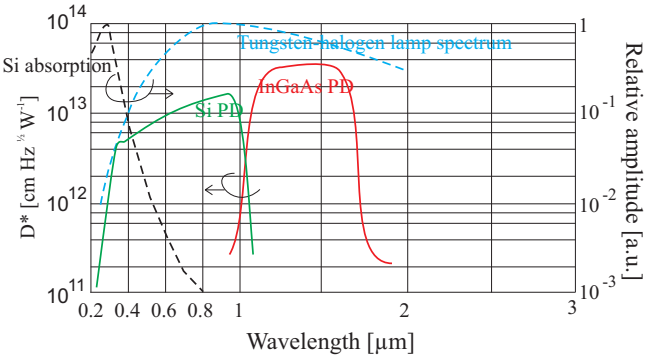

Fig. 2 Dispersion of the characteristics of the main elements in the set-up.

Finally, the set-up is completed with a signal conditioning circuit where we convert the photodiodes signal to numerical data using a custom made amplifier (with precision multi-turn potentiometer to set the gain) connected to a USB analog to digital converter from National Instrument. The signal is then sampled 4 times per second with a LabView script running on a computer.

3 Experiments

We have conducted different tests of silicon membrane etching using a hard mask of silicon nitride 0.2 μm thick deposited by LPCVD on a slightly oxidized, (100)-oriented, n-doped, double side polished, silicon wafer of 100 mm diameter. The wafer is patterned by standard photolithography and RIE etching with CF_4/O_2 chemistry. During the experiments we use a standard bath of 33%-KOH solution kept at a temperature of 80°C in a large temperature-controlled bath with nitrogen bubbling for stirring. A square aperture is patterned in the center of the wafer and is used for optically monitoring the membrane thickness. The wafer is securely placed in the holder and the fiber end placed about 3 mm above the central aperture surface to allow easy escape of the hydrogen bubbles formed during the etching. On the front side of the wafer inside the holder we have taken precaution to keep bare the surface of silicon above the membrane. In that way the light intensity is not perturbed by interference that could occur in thin-films, affecting the sensitivity of the measurement. The membrane aperture is 6 mm wide, much larger than the light collecting zone of the fiber (including the effect of the NA) to avoid alignment trouble and obtain a good sensitivity. Actually we have scanned the fiber across the membrane surface after it has been etched and we found a lateral position tolerance of more than 1 mm, enough for simple manual positioning of the fiber.
The curves in Figure 3 show the intensity of the light passing through the membrane during the last hour of the etching, that is about the last 60 µm of the membrane. We show here the signal from the InGaAs photodiode (labeled NIR), the Si photodiode (labeled Vis), and the normalized ratio between them (labeled Vis/NIR). The quick oscillation observed on the curves are believed to be due to the movements of the hydrogen bubbles appearing on the etched surface and the step-like change are either due to larger bubble detachment or to motion of the fiber in the bath. Of course, existing fluctuations of the light source intensity are also appearing in this signal.

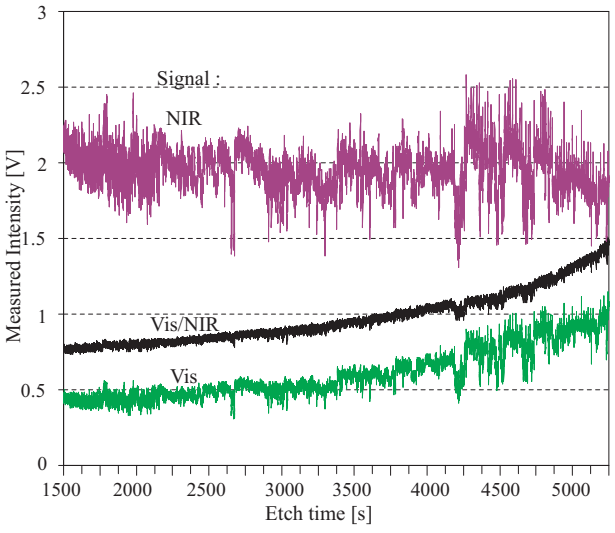

Fig. 3 Intensity after the membrane during the last hour of etching in 33% KOH at 80°C. (NIR) the intensity measured by the InGaAs detector, (Vis) the intensity measured by the Si detector, (Vis/NIR) the normalized ratio of the two previous signals (The ratio has been multiplied by 2 and offset up to allow an objective comparison with the Vis signal).

These curves clearly show the relevance of our approach, as the average Vis signal intensity increases significantly during the etching (that is, when the mem-

brane becomes thinner), whereas the NIR signal remains largely unchanged. Moreover we see that by taking the ratio between the two signals Vis/NIR we reduce the signal to noise ratio by a factor of 3 to 4 compared to the Vis signal alone. It should be noted that simply averaging the visible signal can not compensate for the large jumps that can be seen particularly towards the signal end, whereas they are highly compensated in the intensity ratio. The remaining fluctuations on the ratio signal are probably due to the difference in light absorption in the visible and NIR wavelength in water, making the ratio of α_{med} in the two wavelength bands vary slightly with the presence or absence of a bubble.

The complete analysis of all the parameters affecting the attenuation and the detection in Eq.2 and those governing the light collection is cumbersome. For relating the intensity ratio value to a specific thickness we have performed a calibration step for etching in our standard KOH bath. We placed a wafer in the set-up continuously monitoring the intensities and we regularly stopped the etching process for checking the etched depth using a profilometer. We used a mechanical chuck to position the wafer in the same spot on the profilometer for increasing the accuracy of the etched thickness measurement. Additionally, each profile measurement was repeated 10 times and averaged to decrease the influence of random errors. Finally, before the membrane was fully etched out, we cut through the wafer and used an SEM to measure the remaining thickness of the membrane (in the range of a few µm), allowing to beat the accuracy limit imposed by the TTV of the wafer. We estimated that by using this cumbersome and lengthy procedure the accuracy of the etched depth measurement is about 1 µm, enough for calibrating our tool whose goal is to obtain an accuracy below the TTV of the wafer. The proper mounting of the wafer and the fiber after this step was each time ascertained with the signal coming from the NIR detector which has to be around 2V in our case when it is placed in a cold KOH bath free of etching bubbles. As described above, we could verify that the ratio between the two detected intensities is not sensitive to the fiber position, as soon as the fiber is close enough from the surface to be sure that all the collected light is passing through the membrane. Actually, it is necessary to change the gain of the visible photodetector for different ranges of membrane thickness and we have particularly focused on the range below 40 µm where the gain could be fixed to a single value. The tabulated series of calibration data is then fitted to a fifth order polynomial which can then be used for obtaining the thickness from the value of the intensity ratio.

To estimate the resolution of our system, we have then etched a series of membranes with thicknesses below 20 µm, and compared the results obtained with our calibrated system with the results obtained by measuring the etched depth with a stylus profilometer using a standard procedure. The horizontal error bars on the profilometer measurement correspond to the TTV of the standard wafer (5µm) as for measuring the membrane thickness we had to rely on the thickness of the wafer from which we subtracted the etched depth measured with the profilometer. This complicates the use of this method as reference, but it seems clear that the resolution of our technique is at least better than the standard TTV of the wafer as expected. From our experience the system is still reliable for membrane as thick as 40µm, although, as the membrane get thicker, using a simple timed etch may give results with sufficiently low relative error.

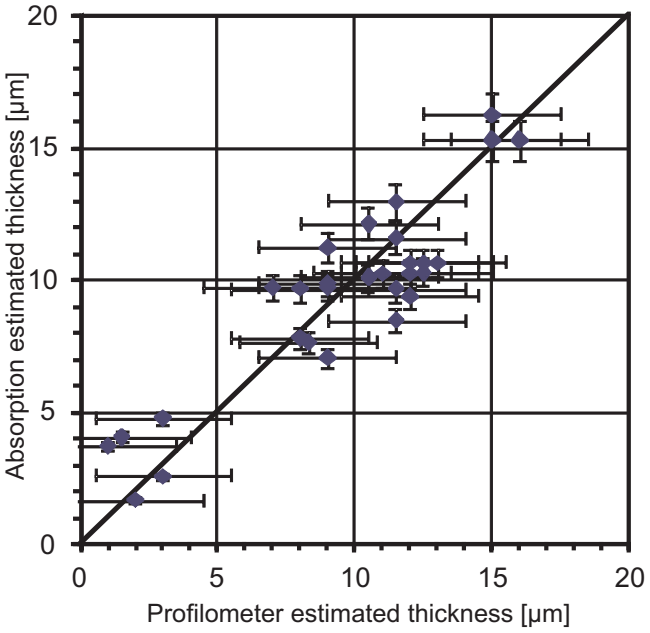

Fig. 4 Comparison between membrane thickness estimated using etched depth measurement and optical absorption measurement.

4 Conclusion

We have presented a simple and economic way to obtain single-crystal silicon thin membranes with precise thickness, that requires only one initial calibration step when it is used in a standard KOH bath. Of course the technique won't compensate for intrinsic thickness variation across the wafer (TTV) but by monitoring in

situ the membrane thickness it will allow speeding-up significantly the realization of thin silicon membranes that can be unattended until the last minute.

We may note that we used n-type silicon wafer throughout our work to avoid the light induced increase in etching rate that appears in p-type wafers. An evolution of the set-up could use a fiber to illuminate the wafer back instead of the lamp placed here directly inside the holder. In that case it would be possible to use a mechanical shutter to limit for very short time the wafer illumination by the halogen lamp. Finally, the sensitivity of the device may be increased by tailoring the source spectrum in the visible wavelength band. Actually, as we can see in Figure 2 if the source is limited in the visible between 0.3μm and 0.5μm, the sensitivity to absorption in silicon will be much larger, and the resolution of the device should increase.

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