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Nanofabrication of Diffractive X-ray Optics

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Abstract

Hard X-ray Nano imaging is one of the best candidate for the futuristic development in the area of imaging used for different kind of applications especially in the bio imaging area such as tomography and CT Scan, for that reason the zone plate for hard x-ray regime exploited for diffraction and its physical conceptual phenomenology has been opted for that reason, in the current application the whole insertion instrument is going to be installed in a so called synchrotron for light accelerating, as has been reported as a novel project in NANOMAX IV, in Sweden.

The idea backed up here is to explain different aspect for the structure realization in terms of fabrication and characterization of the zone plate, especially the one regraded for surface micromachining. To make a precise description on what is coming out as the master thesis, it worth to say that the effort has been made to report the fabrication process for the best performance in terms of resolution and diffraction efficiency which in turn are related to the dimensions of the zone plate, multiple steps are going to be held for the fabrication and characterization process, such as patterning, deposition, etching, optical microscopy, SEM, AFM and other metrologies.

Regarded to the work dedicated to the thesis, development has been done three steps for structure realization that define for the Silicon backside etching or substrate thinning, named as optical lithography, deposition by means of electron beam evaporation method and finally for the structure thinning the method used for etching has been assisted by chemical or wet etching to be performed using the known chemical etchant named KOH (Potassium Hydroxide), obviously during the fabrication process optical microscopy exploited for better surface imaging and observing. Having reported all the physical and chemical steps used for nanofabrication, the parameters used during the processes affecting each step have been evaluated. Such as etching rate and using auxiliary chemical agents for the kinetic of the process. It worth to say that one of the best instrumentation could be helpful for better evaluation or characterization of the etched surface is a measurement device for thickness and roughness evaluation, called contact or optical profiler, of course regarded to the case study and the application the usage

will be different, especially here we use optical profiler for the surface roughness and etched structure measurement based on the interferometer physics and contact profiler as a mechanical instrument for the nanofabrication process evaluation during the deposition and patterning. Different surface roughness parameters and thickness are acquired when different etching process regraded to different type and amount of parameters has been studied, with respect to what has been mentioned the ultimate idea that addressed here are going to be exploited when the desire option is to have better resolution and diffraction efficiency for different type of high aspect ratio index for the zone plate, it is considered for frequent attempts to use different substrate like tungsten and diamond, the most pertinent candidate for the substrate that we are interested in here is silicon and studying its effect an already etched structure when diffractive optics is considered for hard X-ray beamline during the experience in the synchrotron ring for light acceleration.

We present the reports done for different samples in terms of doping and structure realization for chemical etching used KOH as chemical etchant, the fact was to optimize the depth and surface roughness as much as possible using different state of arts such as adding alcohol to the solution during etching and study the etching depth when the sample is positioned in different geometry inside the solution for etching, also the temperature effect as detrimental and enforcing factor to alter the etching rate and roughness also has been studied, as long as the parameters affecting the etching mechanism the crystal being etched and its characteristic also mentioned for the comparison to other crystal planes, different aspects such as surface tension and adsorption density have illustrated that the etching mechanism has trade-off module for parameters alteration and the optimized behavior must be discovered as a intermediate phase for changing the amount of alcohol and etchant concentration together with changing the internal parameters; finally analysis done on the surface structure using optical profiler elucidate the morphology of the structure needed for back-side thinning of the structure so as to achieved the best for trenches assembled as building block foundation of the zone plates in a sense that the best factor for transmission pertinently suits the photonic energy in the range of Kev will be the one corresponding to the depth of silicon substrate admitted for light transmittance around 900 nm or red-infrared spectra.

Chapter 1

Introduction (Objective & outline)

The perspective of the master thesis represented is addressed to manifest the development of the nanofabrication process used for the realization of some instrumentation to be inserted as optical devices in the area of synchrotron technology and its application. Precisely in tomography and imaging systems.

The idea is that how different nanofabrication process in terms of material properties and usage in optical system can be opt so as to achieve the best optical characteristics such as diffraction efficiency and focusing when talking about imaging. Nanofabrication of diffractive x-ray optics is the term that we worked on to optimized different aspect in nanofabrication process based on different parameters when talking about material and structure realization. The best achieved parameters to be reported here in detail where to optimize the surface roughness and depth of the structure precisely in a sense that other parameters like the transmittance and refractive index were considered during the test.

Based on the project the development was to work on Silicon backside thinning or etching as an intermediate step during the hard X-ray zone plate fabrication to be inserted in the NANOMAX IV generation of synchrotron project figure1, the detailed layout for the previous and current project is depicted in the photo.

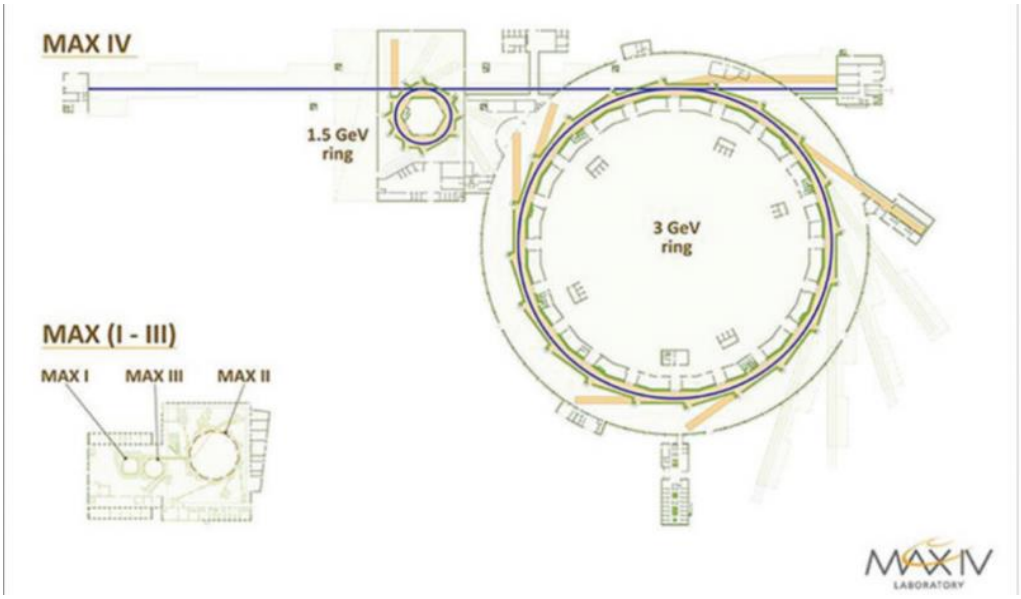


Figure1: MAX IV 3 GeV ring, composing of 168 meter as the diameter, used for light acceleration, the previous generations also manifested as the internal project carried out beforehand [1]

Different nanofabrication process such as optical lithography, electron beam evaporation and chemical or wet etching has been reported and the ultimate test for depth measurement also derived by optical profiler or better to say interferometer technology, the chemical solution used for the etching KOH as an etchant plus the effect of alcohol named IPA also evaluated and reported in detail.

The idea is that by the progression of science and essential needs for the future human being discovering of the environment and himself the needs for getting into more precise measurements of the surrounding factors are going to be increased, one of which that we are very much interested in is to find a way so as to overcome the unknown problems especially related to the dangerous attacks encountering by humankind or the ecology, named disease, for that end the most novel instrumentation continuously needed so as to recognize and encounter them, of

course at the first step during the history of long time attempts people were interested in imaging, and imaging is the easiest and better to say the prominent solution for them to feel what is going on around them. Nowadays by the development of instruments in parallel to the biological progression for immunoassay and curing, the needs is going further to help biology to discover more in terms of scale recognition and speed of biological entities analysis for being in some hidden location inside the body. The ideas turns out to be more fascinating when physics come into help by introducing modern area of physics, named nanoscale physics, and this is the way to ignite the path inside the dark point of biology, named NANOIMAGING. Obviously like any part of a scientific approach the first step is to implement the instrumentation and install all the needed physics behind that. For that end high degree of attempts and efforts come together to make investment in biological and economic point of view, such as the big project currently held in Europe named (XFEL), Free electron laser is an instrumentation implemented inside a very big ring named synchrotron ring that is used for synchrotron radiation, working in the energy scale of Kev when talking about energy of photons accelerated inside that [2].

The working project currently is dealing with the one famous project in Sweden named, NANOMAX, which is a generation of synchrotron project working on the items of hard x-ray experimental station, set for the performance of 7-30 Kev, with diffraction limited resolution of 10-100 nm, of course the design is for the resulting coherence properties of x-ray beams, this ideas is prominent when the usage is invested into Nano focusing area of research for biological studies such as CT-scan, life science, cultural heritage are of most importance issues to be considered in their studies [3]

All of what has been mentioned as progression in the area of optics are coming into fact when the optical properties of structure is ruled by DIFFRACTION, rather than reflection or refraction, and the concept is ran into the point of diffractive x-ray optics, to be more precise about the idea of the incident energy it is worth to consider the x-ray energy in terms of its domain into two separated regimes called HARD AND SOFT X-RAY spectra figure2.

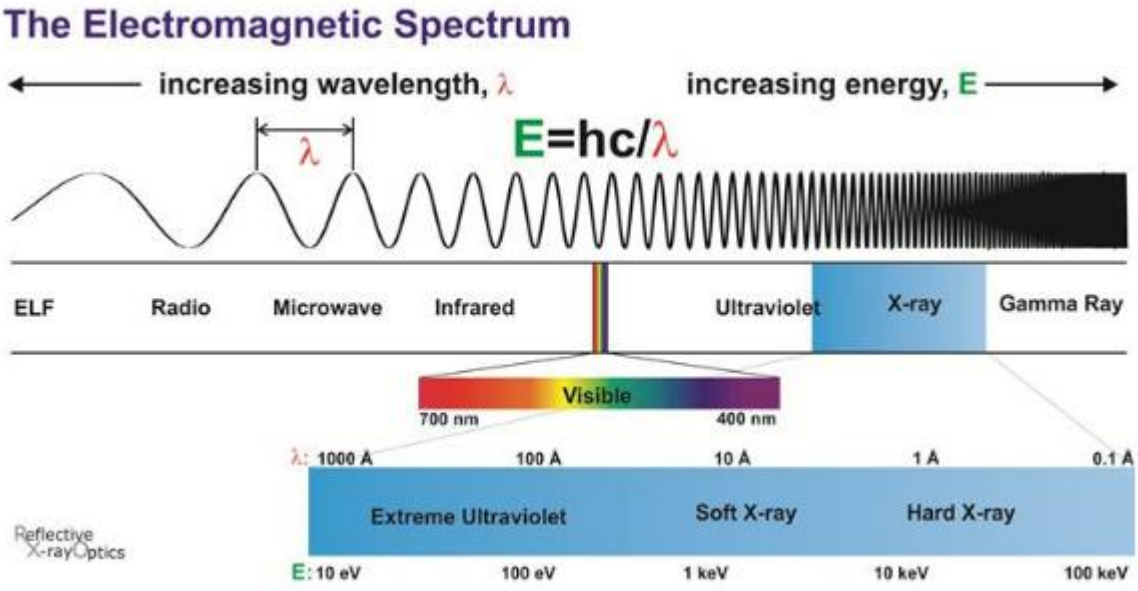


Figure 2: The short-wavelength/high-energy region of the electromagnetic spectrum spanning the extreme ultraviolet (EUV) to the hard X-ray presents a particular challenge to exploit the range needed for recognition among soft and hard x-ray optics close to each other but with different performance in refractive optics [4]

considering all of the properties of the optical properties of these spectra the idea is that how we can insert an optical instrumentation figure3, into the scientific sight or better to say SYNCHROTRON STORAGE RING to control the wave front properties and the get the best performance from that, such as diffraction efficiency and resolution, that recently has been exploited by famous companies in this area for super resolution performance. [5]

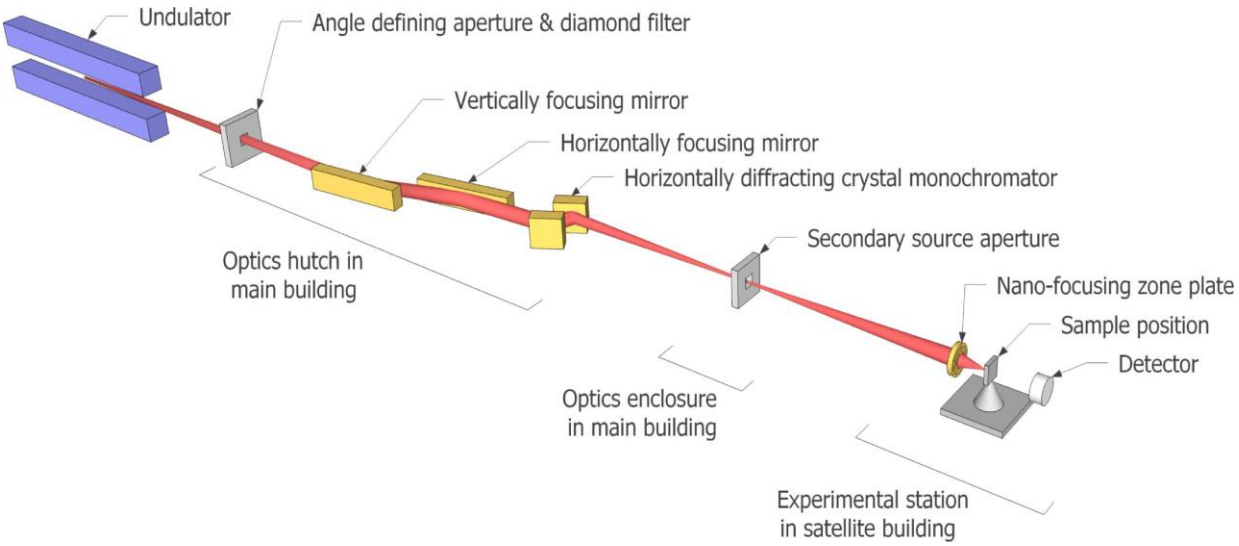


Figure 3: The proposed apparatus adopted for the *Nanomax IV* beamline, the mentioned end station for Nano imaging also identified for the project, the zone plate as the ultimate check point through the beamline has been focused for working as the case study in focusing optimization, 85 m from the source was the first attempt for diffraction-limited beam [6]

To put all around optics in the field of diffractive x-ray optic together the best viewpoint for the optical structure to be identified is ZONE PLATE, zone plate is aimed at focusing, and using diffraction for the goal of Nano focusing optics for x-ray microscopy, and the physical concept behind that is based the diffraction and interfaces when hard x-ray is coming as an incident wave and optimization of x-ray focusing efficiency.

Nanofabrication of diffractive x-ray optics addressed for the set of methodologies used when encountering the idea of nanostructure realization and optical properties of materials to be optimized in terms of resolution and efficiency, the bridge that best cross link the material and optical properties of the zone plate are addressed as:

width of the finest ring of the zone plate for the resolution and thickness of the structure for the efficiency or better to say diffraction efficiency, the remained challenging factor that best controlling and playing the role in optical performance is the substrate or foundation thickness, it means that optimization in terms of the size of the substrate in turn is playing a rule for the transmittance and absorption coefficient of the whole zone plates figure 4. The work is dealing with thinning of the substrate and using different methodologies in terms of Fabrication process for better controlling the substrate thickness and roughness. From now on in a very advance point of view the works were to finding a best method to improve the dimensional parameters and in this thesis the focus is on the different part of the nanofabrication process used for that end, thinning or etching of the substrate is performed by means of nanofabrication sequential work in terms of patterning, deposition and etching, that must be chose in each step which method is the best to start the preformation.

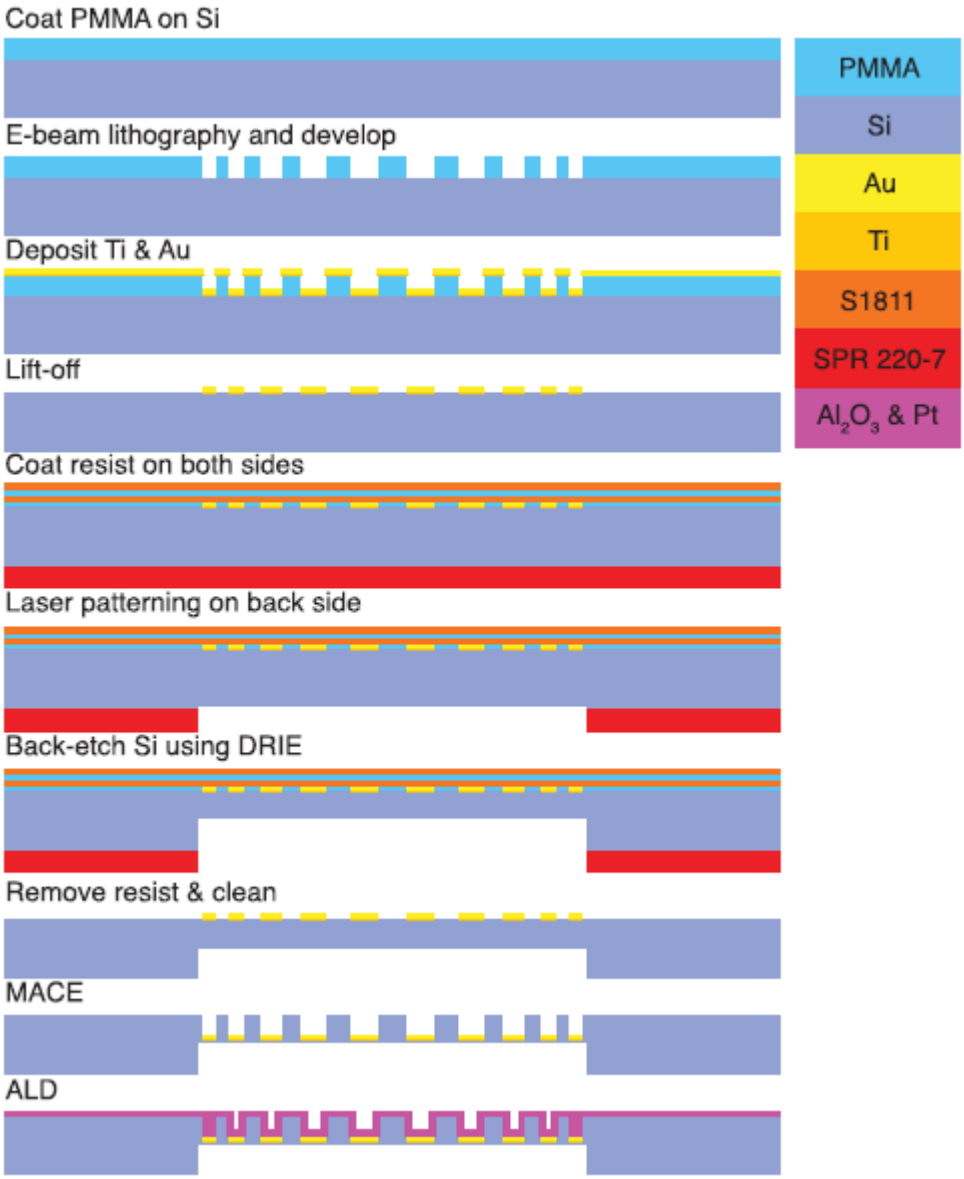


Figure 4: Process flow for zone plate fabrication, the target step regarded to the work dealing with chemical etching is replaced for the Back-etch Si using DRIE, to evaluate the results when wet etching is introduced for thinning the silicon substrate [7]

It is worth to be mentioned that the work can be considered as a sub branch work dealing with surface micromachining and optical MEMS, like micro mirror application in industry and high technology in modern area of research and development [8], however we are just dealing with optimization of parameters for back side thinning of the zone plate substrate so as to facilitate better structure.

Talking about the so called characterization or metrology of the structure, some ideas are come into the fact when reflectance and absorbance are measured by photoluminescence spectroscopy and for the ordinary study of x-ray absorption measurements also is used, however the work here is to study the structure step by step using optical microscopy, contact profiler and interferometry or better to say optical profile meter for thickness and roughness evaluation of the substrate, for that end the strategy is to work on the optimization of the silicon back side etching which is a work that is as an intermediate part in the great work for hard x-ray Nano imaging that is implemented by nanofabrication of zone plates when the aspect ratio is of high degree for better resolution and diffraction efficiency. We are working on the hard x-ray zone plates for the easy to use in terms of easy alignment, controlling the energy tuning, of course high efficiency is desired to make use of the increased coherence in the synchrotron station. Having work with different materials and structures, ultimately it was adjusted as an effort to work for the thinning of the silicon substrate when is deposited by metals and then dealing with the etching parameters to optimize the parameters in terms of thickness and roughness, precisely the objective here is to use KOH or potassium hydroxide as an etchant for the process of wet or chemical etching and then study the effect of different parameters during the etching, also it has been denoted that one of the best candidate that we are dealing with during the experience responsible for the reaction mechanism and roughness of the structure was to study the etching using alcohol as an auxiliary agent and observe its effect on the etching process. The ultimate work for thinning the substrate so as to implement the process for the electroplating step is the one with very nice wide trenches figure 5 as shown below:

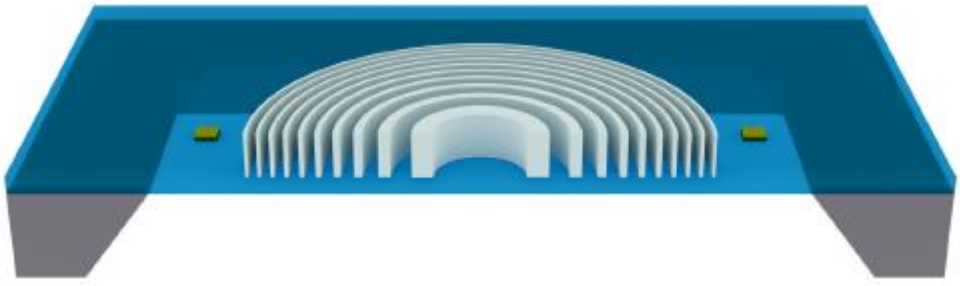


Figure 5: Backside thinned silicon substrate as a building block plate for zone plate growth on the front side (upper side), the backside is well trenched enough for the sufficient transmission of diffracted light incidence form the zone plate structure [9].

Considering the framework the outline will proceed like what as follows:

In the following chapter the fundamental aspects of zone plate and the optical phenomena will be introduces based on the diffractive x-ray optics, then the approach will be dedicated to the theoretical features of the whole work done during the fabrication and characterization of the structures, following the experimental steps to be reported in upcoming chapters, discussion about the results and conclusion will be presented at the final section beside to the different prospect candidate for structure observation and outlook of the upcoming aspects of the project already done using diffractive x-ray optics.

To clarify the framework we have to subdivide the work in terms of different aspects:

- **Problem scheme**:

  Working on Focusing in the nanoscale regime for better imaging and focal length optimization in the module work of resolution performance.

- **Methodology:**

  Approaching the problem, the methods that we have to use is to identify the structure, here is a zone plate, and specifically define the step evolving the targeting optimization. The case we are dealing with is substrate thinning.

  Why ► thinning the substrate will help enhancing the transmission through the zone plates, giving rise for higher performance in terms of diffraction, increasing the focal length for higher aspect ratio of the patterned zone plates based on thinner substrate.

- **Experimental approach and outcomes:**

  Based on many works performed for thinning substrate, and the results achieved, the one that we are interested in is the use of chemical etching for structure realization by means of sequential fabrication figure 6 steps exploited for that end, especially the chemical we are encountering with is KOH as etchant for thinning Silicon backside as substrate, decorated by metallic etching mask to selectively remove

silicon from the depth and resulting in super deep trenches in depth of the substrate, to be used for the next step as electroplating sample.

Precisely the work devoted for the thinning resulted in 2 order of magnitude of micrometer trenching inside the silicon substrate, using different amount of alcohols to study the mechanism of etching rate and surface roughness of the realized structure, the methods used for that were around surface tension and adsorption density measurement beside to Raman spectroscopy[10] , the transmission will be around red-infrared spectrum, the preferred crystal plane will be <100> for the bottom, and <111> for the sidewalls; in order to explain the fabrication steps clearly we can end up with the schematic below:

Figure 6: Process flow for the nanofabrication steps must be followed in order, also the repeatability is the golden factor when fabrication come into account, and people use SOP to manifest their work as reference sheet in the lab.

Chapter 2

Theory

The physics behind the work that already done for nanostructure fabrication and characterization will be categorize in steps as follows:

- Physical concept of the optics case study
- Nanofabrication Process
- Characterization and related metrologies

For the **optics** we are dealing with the part of zone plate definition and its related parameters such as resolution and diffraction efficiency, x-ray characteristics when dealing with Nano imaging and Nano focusing.

For the realization process we are going to explain immediately different aspect in terms of nanofabrication and better to say microfabrication for surface micromachining such as optical or photolithography for patterning, electron beam evaporation for material deposition, and chemical or wet etching for thinning or etching process for trench realization inside the substrate.

For the characterization or metrology process the idea that we reached for that was to use optical and contact profiler for thickness and roughness study, meanwhile plenty of optical microscopy records gathered during each micromachining process before substrate thinning.

2.1 Optics and nanostructure justification

The idea coming out when dealing with Nano imaging is the best instrumentation to be used when the goal is to optimize the focusing efficiency and spatial resolution. Focusing to very small spot size ranging between 10-100 nm, and respectively the energy range from 6-10 Kev, eventually we come with an idea of x-ray radiation [11].

Considering especially the x-ray radiation, the more precise subrange of that spectrum is regarded as hard x-ray radiation which in some cases has overlap with gamma radiation. Regarded to each case study such as the instrumentation and physical condition required for the experimental work we can exploit hard or soft x-ray, in case of energies for higher than 10kev the range that we are dealing with is hard x-ray radiation. The appropriate technology to be used in order to orientate the x-ray diffracted radiation into the path to the detector is the Diffractive optical lens called **ZONE PLATE** which for focusing uses diffraction method instead of reflection or refraction. Of course for any type of technology the optical characteristics must be taking into account, here for the diffraction concept we are dealing with diffraction efficiency which is a fraction of incident light focusing in a specific order of diffraction, and related issues such as diffraction limit, x-ray properties of the composed elements, attenuation length, absorption (ABSORPTANCE) and Transmittance are considered [12]

The zone plate itself as a diffraction grating in circular pattern has radially decreasing width which is addressed for the resolution in a sense that the width of the zone restrict the achievable resolution. The distance between each zone is calculated directly to the optical path in a sense that from each zone to the zone plate focus is an integer multiple of the wavelength, which results in constructive interference [13], this is the essence of the interface and constructive pattern, They are forming a periodical structure of opaque and transparent zone figure 7, for rotationally symmetric grating, decreasing the period whence the radius increase.

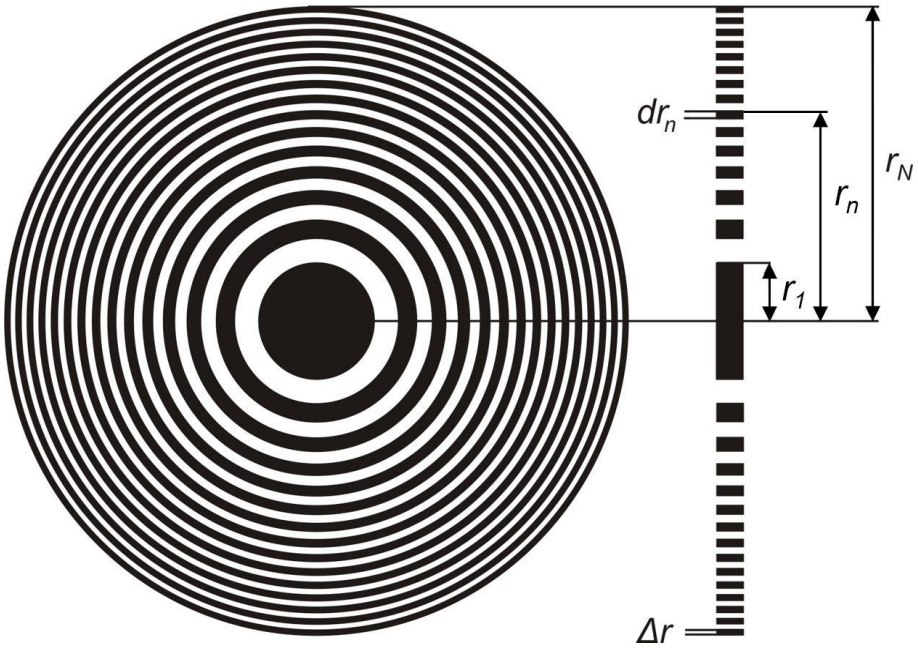


Figure7: the zone plate lens. r_n Is the innermost radius, Δr is the outermost zone width, dr_n is an arbitrary zone width and $r_N$is the radius of the zone plate lens [14]

The distinctive focal spot along the axis is constructed when the constructive interference are produced in higher diffraction order. Related to the light microscopy one can make a relation between resolution and the diffraction limit due to the resolution angle as

$$W = 1.22\frac{\lambda}{d} \qquad (2.2.1)$$

And $\Delta r'$ spatial resolution to the wavelength due to the formula

$$\Delta r' = \frac{0.61\lambda}{NA} \qquad (2.2.2)$$

The NA here is the numerical aperture, the link for connecting the spatial resolution and the outermost zone width is as follows [15]:

$$\Delta r' = 1.22\,\Delta r \qquad (2.2.3)$$

In a sense that the achieved resolution is in turn proportional to the smallest zone width, this is the key point for the spatial resolution factor that in turn is related to the width of the smallest or the last period of the circular zone plate. While the incident wave can been approximated as a plane wave. Clearly the explained zone plate that is already composed of alternative patterns of opaque and transparent zone, will absorb half of the photonic energy, and about 10% for the first order focus, considering that the higher diffraction order brings about lower efficiency, meanwhile the focal distance is determined by the diffraction order used for focusing, and to achieve for example 10 nm resolution for imaging it could be reasonable that the structure must be arranged for nanoscale features.

To make a clear perspective for the zone plate design and application three parameters are coming into consideration as: the wavelength (λ), the outermost of finest zone width (Δr) and the zone plate diameter (D).

Regarded to the diffraction efficiency the predominant factor is the zone plate thickness and material of choice, ranging in the first order of microscale; for the desired energy say about 8.2 Kev the efficiency will show different pattern depending on the material property figure 8.

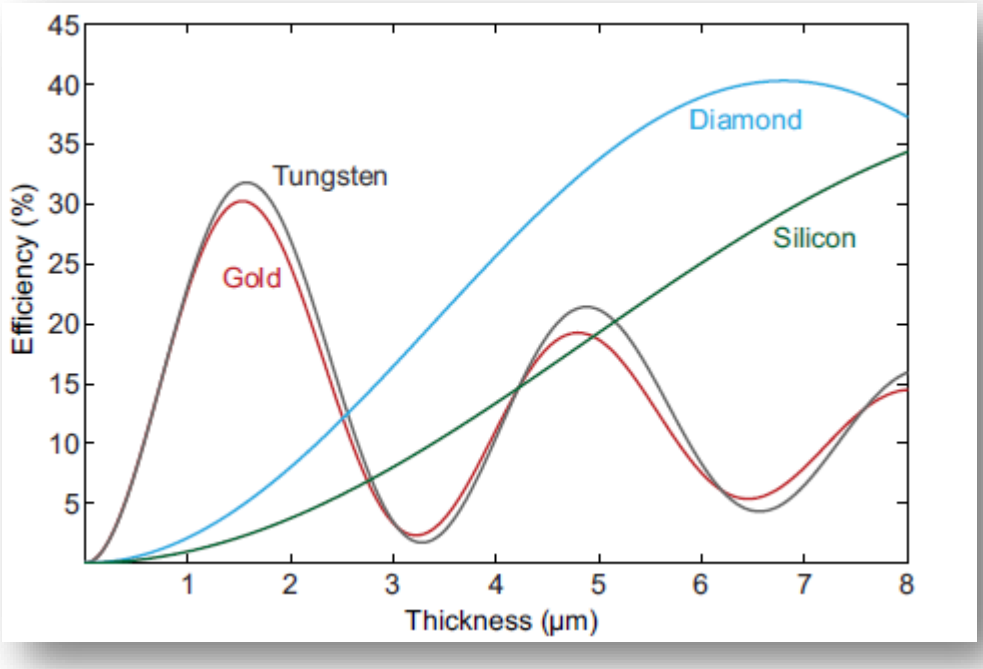


Figure 8: the efficiency versus thickness for different materials for the given energy of 8kev, using Silicon manifested that for the lower range energies the efficiency has monotonically increased comparing to tungsten with damping in efficiency [1]

The tradeoff between diffraction efficiency and resolution which it turn are affected by the thickness and width of the zone plate play a challenging factor in prospective technologies. The other important factor that come into the rule is the wafer thickness that the goal for thinning is going to be based on a fact that for 10-20 micrometer, we have a rough absorption of 10% for silicon thickness of 15 micrometer by the photonic energy strength of about 10 Kev, this the so called silicon back-etch or substrate thinning, and confirmed by x-ray absorption measurements it has been pointed out that about 10 micron of silicon substrate will show red spectrum or near infrared range. To make an end to the discussion above it could worth to denote that Fresnel zone plate are exploited for its features regraded to easiness in alignment and tuning of energy and the most important factor in transmission x-ray microscope which is to say large field imaging capability.
Also to give an idea about the measurement the analysis elucidated that silicon substrate thickness for the about 10 micron thickness will correspond to about 8 Kev for the maximum range of the first order diffraction efficiency, which can be translated into absorption of 12% of the incoming photonic energy penetrating into

the material when passing 9.8 micron out of 10.2 micron silicon substrate, and the noble idea is coming into consideration when the modern high resolution zone plates and high aspect ratio is coming into the rule since it is responsible for higher increase in device efficiency to overcome this amount of absorption and attenuation.

2.2 Nanofabrication Process

Now we are ready to explain the whole experimental process behind the realization of the structure to be defined for zone plate fabrication, which is specifically dealing with substrate thickness and roughness working section. For that end we have almost four sequential steps for micro and nanofabrication of the trenched patterned etched silicon substrate figure 9.

1. Optical or photolithography for contact patterning
2. Physical vapor deposition or thin film coating for material deposition
3. Lift off for resist removing from the coated silicon substrate
4. Chemical or wet etching so as to define etched region in substrate depth

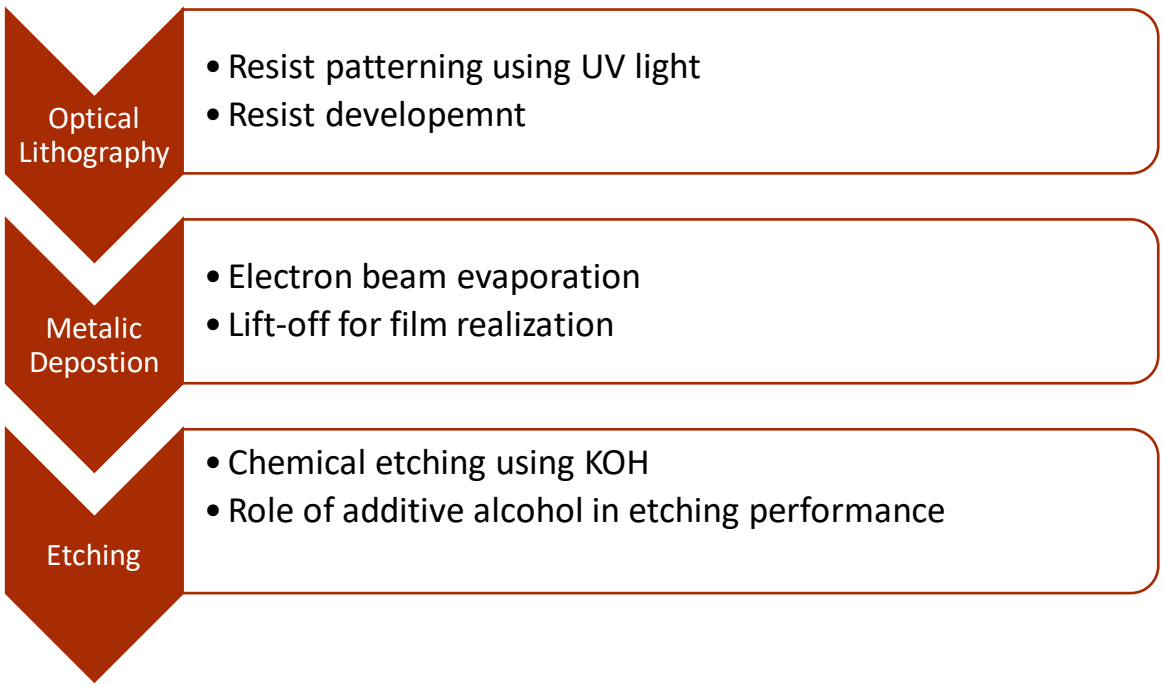


Figure 9: Process flow for the nanofabrication process, step by step procedures must be done in way that repeatability be obeyed and get the same result.

For each of the steps identified above we have to introduce some parameters dealing with the physical and chemical reaction as well as parameters that are determining the realized structure dimensions and shape geometry. All the project viewpoint is to make parameter optimization so as to facilitate better structure, it means that by the method of micromachining we will realize the micro and nanostructure suitable for the nanoscale imaging when hard x-ray microscopy is considered. In some cases high aspect ratio micromachining is defined when the thickness in order of micron to centimeter, like using some instrumentations such as LEGA or DEEP RECTIVE ION ETCHING [16],[17], however here the specialized case is to exploit the concept of chemical or wet etching for achieving the trench inside the depth of the silicon substrate already deposited by materials and then using chemical etchant for thinning the microstructure down to the depth of the substrate, ANISOTROPICAL etching will carried on by means of chemical etchant to dissolve selective locations inside the silicon wafer based on the crystal orientation, based on the nature of the silicon to be bare and considering the amount of dopant for the silicon the etching rate and so called etch stop will be analyzed, the physics Behind the etching mechanism in some ways is corresponding to the crystal structure and the binding which in turn make a competition for more susceptibility and higher etching comparing to other crystal orientation in silicon substrate.

2.2.1 Optical Lithography

Contact lithography is a method for transferring a mask pattern by exposing light through the mask into the photoresist, UV radiation is the incident light that coming through the mask and pattern the resist in a contact mode, using the exposing light, the chemistry of the resist will be changed that eventually will be patterned by using development procedure, talking about the photoresist it is well chosen based on its photosensitivity and stability while exposure is carried on, in a sense that alteration is obtained in resin chemistry when exposed to light, absolutely we can make use of positive or negative resist for lithography based on the application to see inverse behavior when the pattern module will be changed.

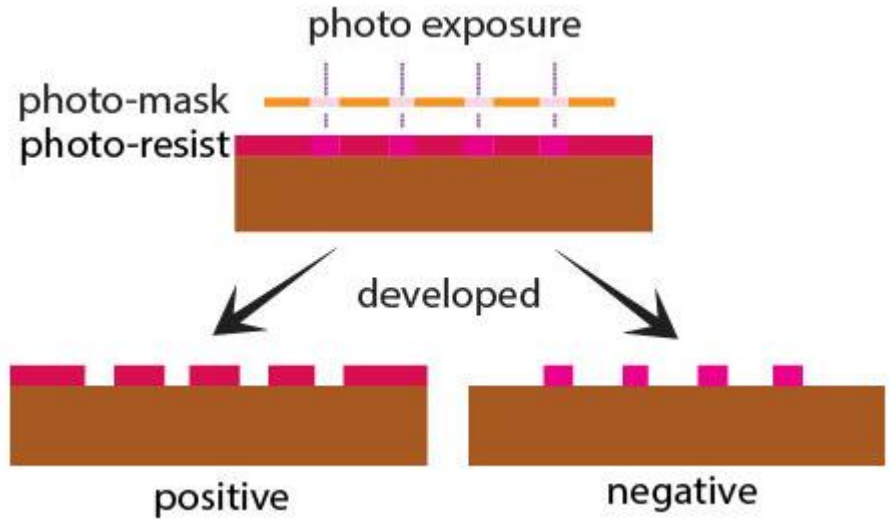


Figure 10: Schematic processing steps of forming patterns for window realizing on the substrate for metallic deposition [18]

In order to implant resist on top of the substrate surface we have to use a machine for coating in a homogeneous and gentle way so as to have isotropic and equally dense resist coat on the surface, for that reason SPINCOATING will be provided for uniform thin film deposition figure 10, the liquid resist will be doped on the substrate and then the spin coating procedure will start to coat the chip in a uniform style, the program for adjusting the speed and rotation module must be identified during that, centrifugal and viscosity forces are playing the roles for thickness determination.

The next step for solvent diffusion is regarded by baking, whether in an oven or on top of a hot plate, here also parameters affecting the result and the residue of the solvent form the resist already coated, such as time and temperature for baking.

This is soft baking after spin coating that plays an important factor in photolithography, in a way that if the remaining content of the solvent will be high after baking, the dissolution in a high degree will take place; after baking the hot and sensitive patterned chip is ready to be transferred for the contact lithography machine or mask aligner, as well as the already mentioned procedures up to now, here for optimizing the pattern during exposure different parameters take into account such as the time of exposure and the pattern geometry needed for Patterning, all of that have to be considered beside to the standard focusing module for exposing, this means that since we are working with focusing, the fringes have to

be recognized primer to the exposing procedure, the most delicate issue regarded to the POSTPATTERNNIG step is handling of the sensitive light emitted photoresist chip, which has to be delivered for the next step, DEVELOPING.

Developing is the last step for lithography, a chemical liquid developer is needed for patterning immerse inside and that means that the developer is the responsible agent for bringing about a clear picture of the already done resist coating on the chip, the resist pattern is a perfect image of the mask, however some lack points during the process such as slopped sidewalls could take places, this is due to the difference in the size of the mask and the resist pattern, the physics behind this issue is due to the distribution of the exposing dose affected by diffraction, absorption.

2.2.2 Material Deposition

Having patterned the silicon chip by photoresist, now the pattern is playing the role of guiding structure for sequential material deposition, depending on the structure and the application variety of deposition methods can be used, whether to be assisted on the chemical reaction method or physical reactions. Electroplating and physical vapor deposition are among the best candidates for that end, of course CVD is also exploited for other applications. The PVD technique to be explained here is considered as the first step for material deposition in the production line of zone plate fabrication. We use the method of film growth by means of vapor condensation on cool substrate using for metal layers. Low surface damage must be considered, and the thickness achieved by the incoming source is depending on the angle between surface and the beamline bombarding that. Here especially we are interested in ELECTRON BEAM EVAPORATION figure 11, in a sense that the method for material deposition is aided by electronic stimulation and orienting the target material for coating. In this method electronic beam is heated by a filament to bombard a target material say a metallic target as an anode to be transferred into gaseous phase, eventually the gaseous form will make transition into solid form so as to ultimately coat the substrate, this is name **thin film growth**.

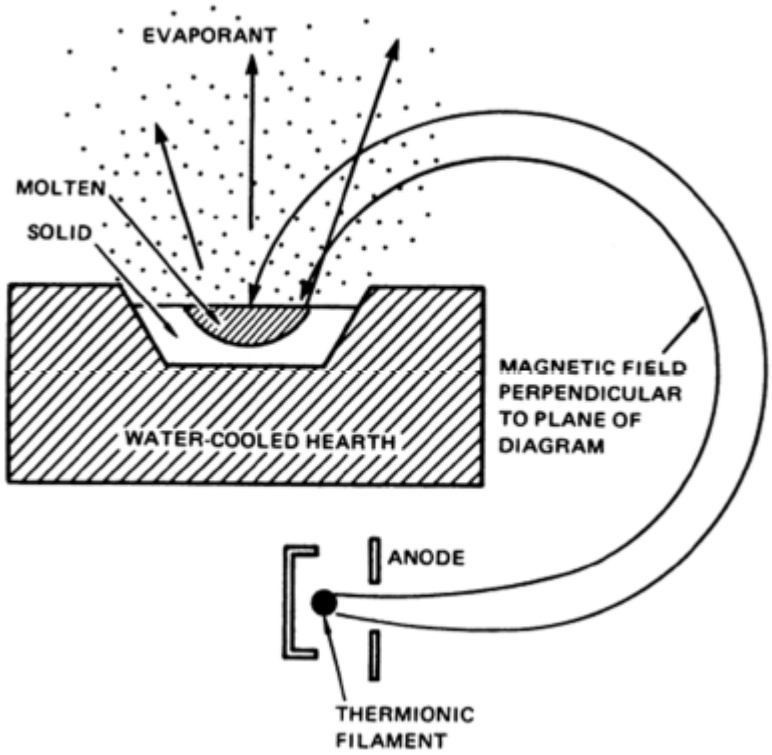


Figure 11: Electron beam trajectory toward the target material using thermionic emission so as to melt the sample and bombard it on the substrate [19]

We are dealing with physical process in sense that the particle extraction from solid source is addressed for different methodologies such as evaporation or sputtering, and the case for our mission is the extract method using thermal heating. The application may serve for electronic or optical means like $Ohmic$ contacts and also waveguides in optical communications. The physics behind the process is in essence dealing with melting or sublimation point of the source when temperature increasing takes place, in turn are affected by the pressure of the chamber.

Vapor deposition is the method for thin film growth on substrate, the condition is that the sample positioned in the vacuum chamber must be tight enough for material deposition process, the actual thickness for the material deposition as coating film is ranging from nanometer to micrometer scales, and the vacuum scale for the chamber is 10^{-5} mbar. This high vacuum is required for the reason of boosting the mean free path of the target particles and also decrement in the reaction taking place among particles and impurities. Also in thermal evaporation the shutter is addressed to be located as an Intermediate agent between the substrate and crucible so as to make fast interruption for particle flowing. The location inside the chamber dedicated for

the source is named Crucible that will be heated by electric current by means of some coils. Typically the chip attached to the holder is located in a higher level with respect to the crucible for as much as possible vapor phase condensation.

There are many parameters determining the ultimate state of the process such as:

- Chamber pressure
- Temperature
- Electric current and related rate
- Deposition time
- Film structure
- Growth speed

The physics behind the rate itself is controlled by the HETZ-Knudsen LAW, determining the number of particles evaporated in unit are and time from the source as the target, managed by high energy electrons ranging between $4 - 40\ Kev$ for bombardment, the body structure of the election gun is working like a heating elements for electron beam emission, focusing toward the target source is controlled by electrostatic or magnetic lenses.

2.2.3 Lift-Off

In order to make the already patterned coated substrate ready for structure realization, it is required to make the structure in some points activated for the etching, this is means that knowing the chemical reaction during the process we have to identify unpattern dimension on structure surface, this could be done by means of a chemical solvent to make dissolving for the coated surface and remain the other parts unchanged. The solvent is responsible to wash out the unwanted amount of thin film in the active regions needed for etching. We call the process Lift-off. The solvent will dissolve the resist coated by thin film and leave other parts of the surface unchanged coated by the metal film. Now active and passive gates for etching are ready to be processed. In a sense that based on the chemical reaction the etchant solution will make chemical interaction with the active or bare regions of the substrate and leave the already metallic coated part UNETCHED. Good lift-off

process ensures that only the metallic film is adhered to the substrate and the remaining parts are assumed as bare silicon with tiny amount of contamination or dust remained from previous processes. If the metallic layer is too thick, then lift-off will give rise in unwanted opening on the substrate surface due to the unwanted washing out of the metals in vicinity of the resisted pattern regions, so breaking off will bring about unwanted etching and trench inside the silicon depth. Lift off could be performed whether gently in a manual handy way such as shaking the chip inside solutions of ACETONE and IPA in order way each for couple of minutes, or using sonication method that by means of ultrasonic machine the stimulation will boost the reaction in the lift off process so as to increase the reaction rate, the scenario is more obvious when the metal thickness is going to be increase further than 100 nm.

The most prominent problem facing the lift-off is that there is a risk of *REDEPOSITION* of already washed out metal on the substrate, beside to the fact that the metal-resist bonding could affect the removing procedure, this means that the sacrificial layer that was supposed to be the resist will be conjugated to the metal to wash that in its accompany. In all aspects the lift-off priority to the etching is based on the fact that the risk of metal-substrate unwanted etched will be eliminated, the other advantages is due to the cheap procedure comparing to etching that takes expenses for processing.

2.2.4 Etching

Looking for a selective and directional removing of the developed structure, we have to choose a method well-defining that target. Of course physical and chemical etching mechanism are coming into idea, however, the one that we are interested in is chemical etching or wet etching, which is based on choosing a chemical etchant in liquid phase so as to make chemical reaction with the patterned structure. In contrary with the physical etching such as deep reactive ion etching (DRIE), chemical etching is based on no isotropic or ANISOTROPIC etching, in a sense that the final

pattern processed by etching will be like V-shape Grooves Trench. So different feature are considered when etching is coming into event such as:

- Selectivity
- Anisotropy
- Uniformity
- Etching rate

Where the last one is responsible for the speed of etching with the scale of (**nm/minutes**), and the direction of etching. For instance when the silicon substrate is considered, the crystal plane itself must be analyzed in terms of the miller index and its crystallography, for Si we already know that etching rate in the <110> Direction is much faster than <111> direction, show different rates. Of course Anisotropic etching stands for only perpendicular material removing, without equally directional removing, this is related to accurate transfer of the mask without undercutting that. The ratio between lateral and vertical etching rate will determine the anisotropy, the less be the ratio the higher will be the value. Considering for the chemical etching the most important weak point with respect to the isotropic or physical etching is the resolution obtained after etching. Also the corrosive and harmful side effects of the chemical etchant make the investment in that are somewhat narrowing.

The best example for chemical etching in the modern era is MEMS, using KOH as the etchant, will bring about in variety of applications such as MICROMIRRORS, pressure sensors and actuators.

The important case in chemical etching is to use a method for thickness determination controlling, there are many different methods for that case as [20]:

- Timing technique
- Boron etch stop
- Electrochemical etch stop

In the experiment coming out two different experience has been done for the boron etch stop and the data manifested that the chemical etching rate has been decreased tremendously due to the very low etching rate of high doped substrate. The

advantages of this method is due to its high selectivity and reliability, however when the case is related to the tensile stress and CMOS it will be recommended.

According to the theory of chemical etching, the reaction is based on **Arrhenius equation** [21] that make a reasonable relationship among etching rate and temperature dependency during the reaction, it should be noted that the reaction is more controlled by temperature rather than diffusion, so the agitation coming into issue for the reaction is overcome by temperature factor, using the formula below we can see that the etching rate has exponential dependency on the temperature:

$$\boldsymbol{R = R_{\circ} exp(-\frac{Ea}{KT})} \quad \textbf{2.2.4.1}$$

Where the factors $\boldsymbol{K}$, $\boldsymbol{T}$ and $\boldsymbol{Ea}$ are standing for Boltzmann factor and temperature and activation energy respectively, the factor $\boldsymbol{R_{\circ}}$ is rate constant and some factors such as diffusivity and density of the liquid substances determine that, the problem encountering here is more issued by the bubbles attacking the selected surface for etching that are coming into the idea by diffusion limited and agitation dependency in etching process.

It worth to denote that there many factors controlling the etching rate, among those the most prominent ones are: Temperature and agitation which have to be trade off when reaction-limited and diffusion limited etching process are assumed, considering that for example the reaction-limited etching process is more controlled by temperature named as **ARRHENIUS EQUATION**,

$R_{\circ}$ is a constant and the diffusivity of the reactants will determine that

$\boldsymbol{Ea}$ is called activation energy, for high degree (higher than 20 $Kj\ /mole$) of the etching process it is called to be reaction-limited process rather than diffusion-limited etching process. Since the product of the etching process in most cases is gaseous, the risk of bubble formation will affect the etching rate which has to be overcome by etching parameters to change the condition such as stimulating by mechanical rotation or imposing agitation so as to orientate the etchant toward that.

The case study here is addressed for silicon substrate thinning and the best etchant for abovementioned procedure is POTASSIUM HYDROXIDE (KOH), as wet

chemical etchant for cavity realization inside the silicon depth, consisting of corrosive alkaline chemical etchant, conjugated by DI WATER (DI) and thermal regulation, also the temperature, etchant concentration and any type of defects like impurities attached to the surface must be considered to affect the etching rate. Of course the benefits of KOH are addressed here to be:

- Easiness in handling
- Repeatability and fast etching process in lab equipment
- Economical and beneficial for batch fabrication

The other chemical and competent etchant that is also useful for the wet etching is TMAH or TETRAMETHYLAMMONIUM, however, the beneficial factors of KOH in terms of the etching rate and morphology (less hillocks formation) will make that in priority for experience.

The case for using KOH as the liquid etchant will bring about some feature as the consequences of the etching which can be counted as morphology and dimension alteration due to silicon removal, especially for the case of MOEMS or optical MEMS the mission is to create a structure to be well defined enough in terms of dimensions (geometry) and morphology, when the roughness of the etched substrate is considered for the diffraction optics. For that end there is a suggestion to introduce an auxiliary agent in addition to the alkaline etchant KOH so as optimize the reaction based on the etching rate and surface morphology due to the adsorption conceptual meaning in surface studies; the most well-known candidate is called IPA or ISOPROPYL ALCOHOL, which affect the efficiency of the chemical etching in terms of etching rate and nature of interaction

Affecting the chemical solution. The crystallographic orientation of the etched surface and saturation of the solution will determine the surface coverage by the alcohol. For instance the performance for the (100) is different from (110) in a way that for the first one the alcohol concentration required for surface coverage is above the saturation while for the last one is below the saturation. The best characterization acquired after the experimental issues where used by means of **surface tension measurement** and **Raman spectroscopy** for etching silicon in aqueous KOH.

2.3 Characterization and related metrologies

The idea for the structure characterization is based on two modules:

1. Material geometry and surface morphology
2. Spectroscopy measuring for imaging analysis

For the first module the subject addressed to thickness and morphology evaluation based on different physical concepts such as optical and electronic phenomena.

- Atomic force microscopy (AFM)
- Scanning electron microscopy(SEM)
- Contact profiler
- Optical profiler or Interferometer machine

Based on the geometry and nature of the structures and surface materials, each of abovementioned method could be exploited, especially for the thickness and roughness measurement and analysis we are interested in taking into account contact and optical profiler evaluation to achieve an idea about the precision obtained during etching and material deposition. The contact profiler is based on mechanical motion probing the surface, for the case of optical profiler the idea is based on interferometer and the imaging analysis for thickness and roughness Evaluation. Worth to say that optical profiler is more precise in terms of geometry and roughness results, considering that the advantage is in trade off when economic issues coming into account for instrument establishing and software programming. The next module is dedicated to imaging achieved during the spectroscopy and photometry. The physics is based on the material structure and optical properties when light beam incident is attacking the surface, followed by the fact behind the physics of absorption and transmission in the object to be imaged. In a way that for the application in x-ray imaging the absorption is determined by the transmission characteristic of the material object. Penetration into the object give rise to divisions dedicated to absorption to be graded in different parts of the structure, such as the idea of thickness dependency in zone-plate lenses when the substrate thinning is the

issue. The difference in the thickness at different part of the structure bring about projection of image in x-ray absorption spectroscopy and imaging. The factor that ruling the x-ray absorption dependency is the mass absorption coefficient, which in turn is a function of x-ray energy and the atomic number of the crystal, the more the atomic number give rise in higher absorption, thus we can terminate with a formula relating the transmission intensity to that coefficient by:

$$I(x) = I_{\circ} e^{-x\rho\mu} \quad 2.3.1$$

In the structure that we are dealing with the sequential thin films coated on the substrate will play nothing but a little bit error in transmittance spectroscopy due to the great ratio between the thickness of the substrate already etched an thin films, the order of magnitude for the difference in ratio is about 1000:1 talking about 20 micrometer for the trench residue thickness and the thin film coated thickness to be about 20-100 nanometer. The other factor that also is important is the refractive index, which can in turn finalize the idea for the transmittance when the thickness is about 20 micrometer for the silicon, the transmission will give a rise for imaging spectrum around red or **infrared** IR range as red light.

Different kind of instrumentation are exploited for the transmittance measurements following by reflectance analysis such as photoluminescence measurements or spectrophotometer named as **UV-VIS spectroscopy** for finding the refractive index And absorption measurements, or typically using FTIR or Fourier transform infrared spectroscopy for absorption-emission measurements in different phases. [22]

For inspection and discovering the chip after deposition and evaporation we can use OPTICAL microscope. The instrument is modified for two different modules named:

- EPI (light incident from the above toward the sample)
- DIA (light is orientating from below into the chip for illumination)

The EPI is based on reflection of the light from the sample after illuminating that from the same direction, it could be adjusted for **DARK FIELD** or **BRIGHT FIELD.** Ultimately we can calibrate the microscope for the desired module and image background and then by adjusting the magnification, the best images depending on the nature of the structure and its impurity could be received,

however, the magnification alteration is coming into the same concept for resolution, since the magnification is just focusing for smaller region when the magnification is going to be increased.

Beside to imaging and optical microscopy of the structure in each step for fabrication, the target has to been analyzed in terms of thickness and roughness.

We use contact profiler for the rough measurement, however, for the range around the one desired for the trench thickness we have to use more precise method so as to define the thickness of the structure and its roughness index in each region, it has to be precise more than mechanical error of the contact profiler, for that end optical profile is used, based on interferometer concept.

The contact profiler concept is similar to atomic force microscopy, in a way that a tip is probing from up down on the surface to scale the sample for the desired range then it make analog signal to be converted in digital signal, probing the surface eventually a pattern of surface elevation in each desired section is acquired. Resolution, length and elevation from the surface for the contact stylus could be adjusted, the more gentile be the surfing on the surface the more will be the precision of the result on the plot. Data signal as a rate and the speed for scanning are determining the horizontal resolution.

For the case of non-contact profiler the optical profiler is not based on a pen or better to say non-stylus interferometry, the records are assured in terms of reliable results for height data in each step for achieving thickness around nanometers for the film or substrate, the topography will be imaged into 3 dimension for the scale adjusted for probing, in contrary with the contact profiler it is well defined for very fine structures.

The optical profiler is based on white **light interferometry (WLI)** figure 12, back to the interferometer concept in physics it works based on fringes ordering, and the refractive index of the sample is a prominent factor for 3D image providing, The dark point of the optical profiler is issued when transparent structure is analyzed. Since the optical path is based on the reflection of light in fringes zones and the equally ordered fringes define equal height in each step.

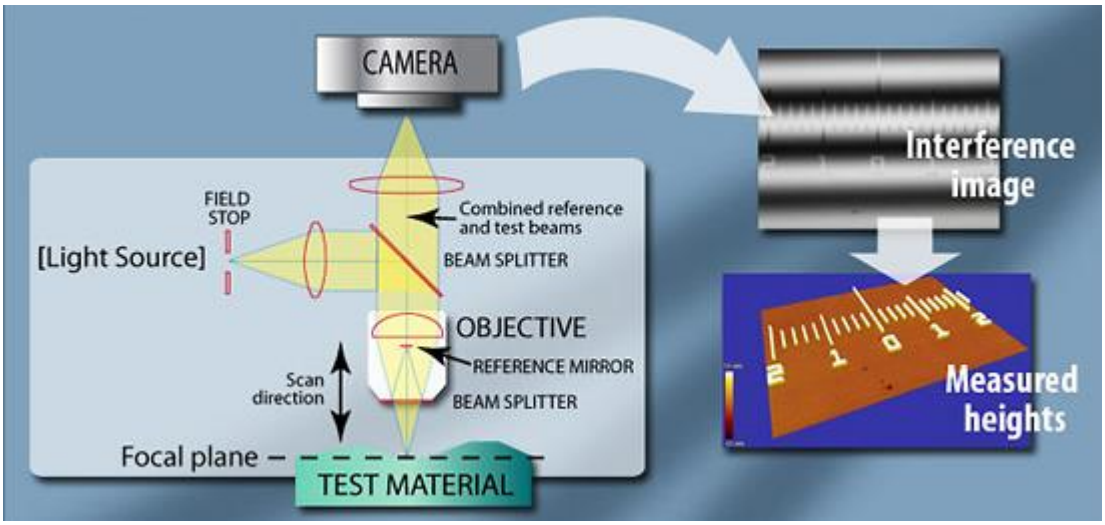


Figure12: Optical profiler principle of operation based on interferometry and fringes counting for height analysis [23]

When we are dealing with etching resulted in deep etching, say more than 200 micrometer, the challenging point is that the resolution of the device has to be arranged separately for each level and then focused so as to find and analyze the dedicated reflected fringes, this could be done by adjusting and leveling the base and plate of the stand that the chip is coordinated on that, then rotating the angle for the best degree, in this way the focusing fringes will be visible, and the interferometer is in stability for focusing and recording data fringes. Some standard data in terms of thickness and roughness standards will be acquired that would be separated in each step and be analyzed independently to study the morphology more precise, this is in competition with the AFM, however, the characterization by means of optical profiler will be more precise and economic for large scale with respect to AFM for smaller portion of the structure.

Chapter 3

Experimental work

3.1 Sample preparation

As the first step for fabrication procedure we have to choose the best sample (wafer) needed as the substrate for the beginning. In the Alba nova Nanofabrication facility wafer sizes around 50.8 millimeter for the diameter equally 2-inch in diameter and thickness about 270 micrometer has been opted, bringing the wafer it has to be clean enough for the starting step, for that end using ACETONE and IPA separately each by immersing the wafer inside them for 5 minutes will help in cleaning the wafer out of dust in the laboratory, it can be done by shaking the wafer inside beaker of each liquid or putting the wafer inside glassware using ultrasonic machine for shaking efficiently, the **ultrasonic** bath has to be adjusted for the desired power on the scale of 5 and heating calibration for **25-45 Celsius degree** .

As the experience concept is discussed in a way that the etching process will be performed in batch etching procedure, we have to scribe the wafer into small piece of squared chips scaling on the order of 1cm*1cm, for this reason we have to use the appropriate machine for cutting or scribing the wafer precisely into small equally sized chips, the machine ***PRECISION − DIAMOND − SCRIBER MR* 200**,

Figure 13.

Is equipped:

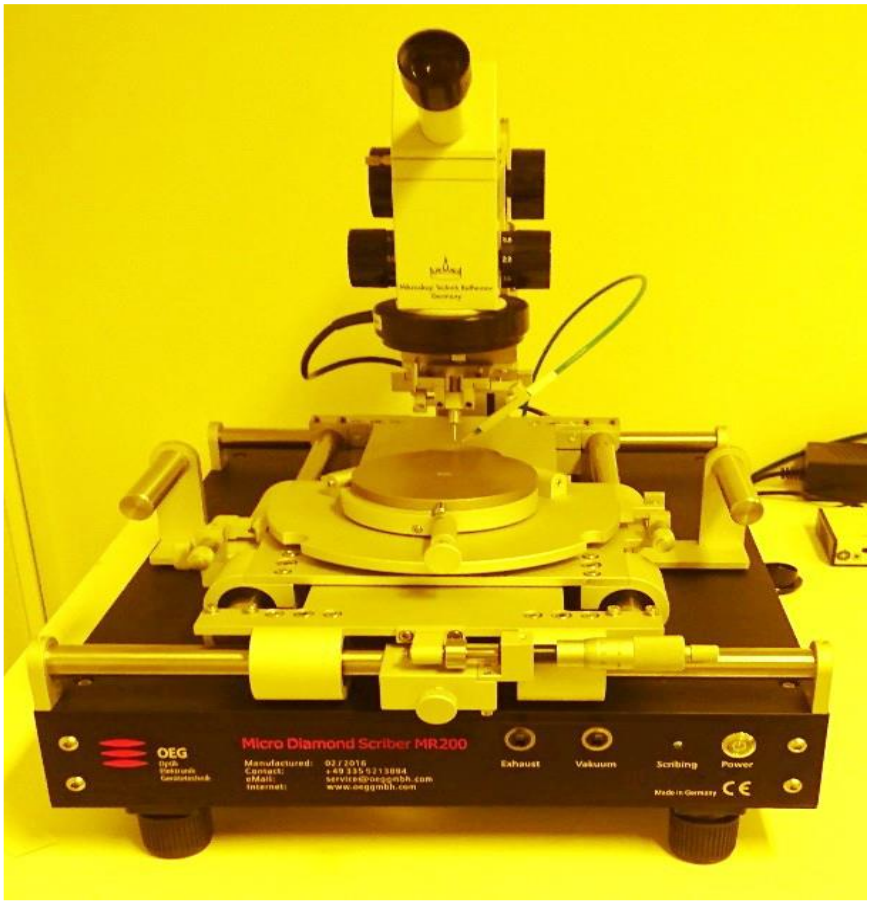


Figure 13: the diamond-scriber MR200 apparatus for chips preparation, the chalk in the middle is the central part used for cutting the wafers into desired chips [24]

It worth to say that during the scribing it will be the risk of contamination attaching on the boundaries or surface of the resulted small chips arising from splash came out during scribing by the tip on the wafer surface, this effect is inevitable, however could be managed by different tasks, such as redo the rinsing for the small chips separately, or using resist as the sacrificial layer to protect the chips from splashing during scribing, for that end we use a polymer resist named PMMA for coating the wafer thoroughly and then spin coat that so as to uniformly cover all the surface wafer, the machine used for spin coating is named RESIST SPINNER modelled **as *EMS SPINCOATER MODEL*** 4000 figure 14.

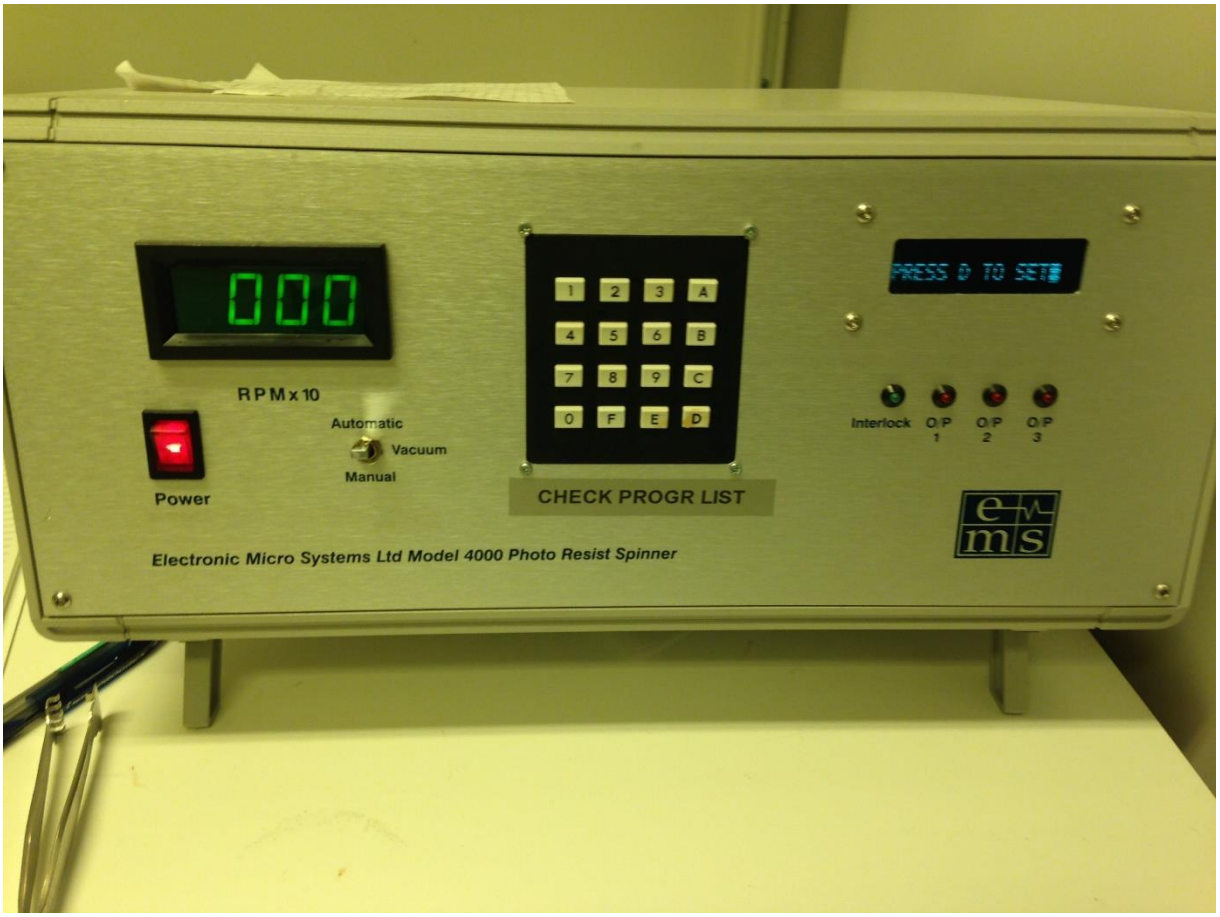


Figure 14: spin coater programing system for adjusting the speed and modules of rotation

The controlling unit has to be programmed for the desired rotation speed and steps. Having resisted the wafer now it is ready to be scribed into small chips, obviously the chips after scribing have to be rinsed again so as to eliminate all the resist coated on their surface, in that way eventually we have to different types of resulted small chips scribed from:

- Rinsed wafer and scribed normally
- Thin film wafer already spin coated and the scribed

Of course both types will be undergone for the whole procedure for nanofabrication and we will report the difference in terms of etching rate and result after etching.

3.2 lithography

In order to allocate the regions dedicated for etching as trench zones, we have to define a method for that realization part, the idea is that we have to introduce some method so as introduce that region on the chip surface, the way for that is to using a light beam to incident the surface and selectively alters the surface morphology, we use optical lithography so as to pattern the surface layer by already sacrificial layer, for that end we need to coat the surface with a resist and then pattern that for the desired application, and finally develop that so as to clearly define the selected regions as clear as possible. Figure 15 The steps are:

1. Resist dropping and spin coating
2. Baking
3. Patterning with light exposure
4. Development
5. Rinsing and drying

Figure15: spin coating tool and the plastic gun are placed inside the LAF-HOOD

The resist is that we chose here as the sacrificial layer is PMMA or (Polymethyl methacrylate) is a polymeric material for imaging application in

microtechnologies.it is a high resolution positive resist that suits for Ultraviolet (UV) exposure in microlithographic patterning. We use that a coating layer to protect the chip during wafer thinning and better adhesion issues. That is coated on the chip surface by means of spin coater machine. Of course in order to achieve a well-defined thickness for the resist coating there have to be a corresponding relationship between the resist liquid parameters and the speed of the spin coater, it means that due to the experience and the nature of the polymer for stacking on the surface the polymer has to be diluted for a certain concentration that is **spin-curve** characteristic figure 16 for each polymeric type resist.

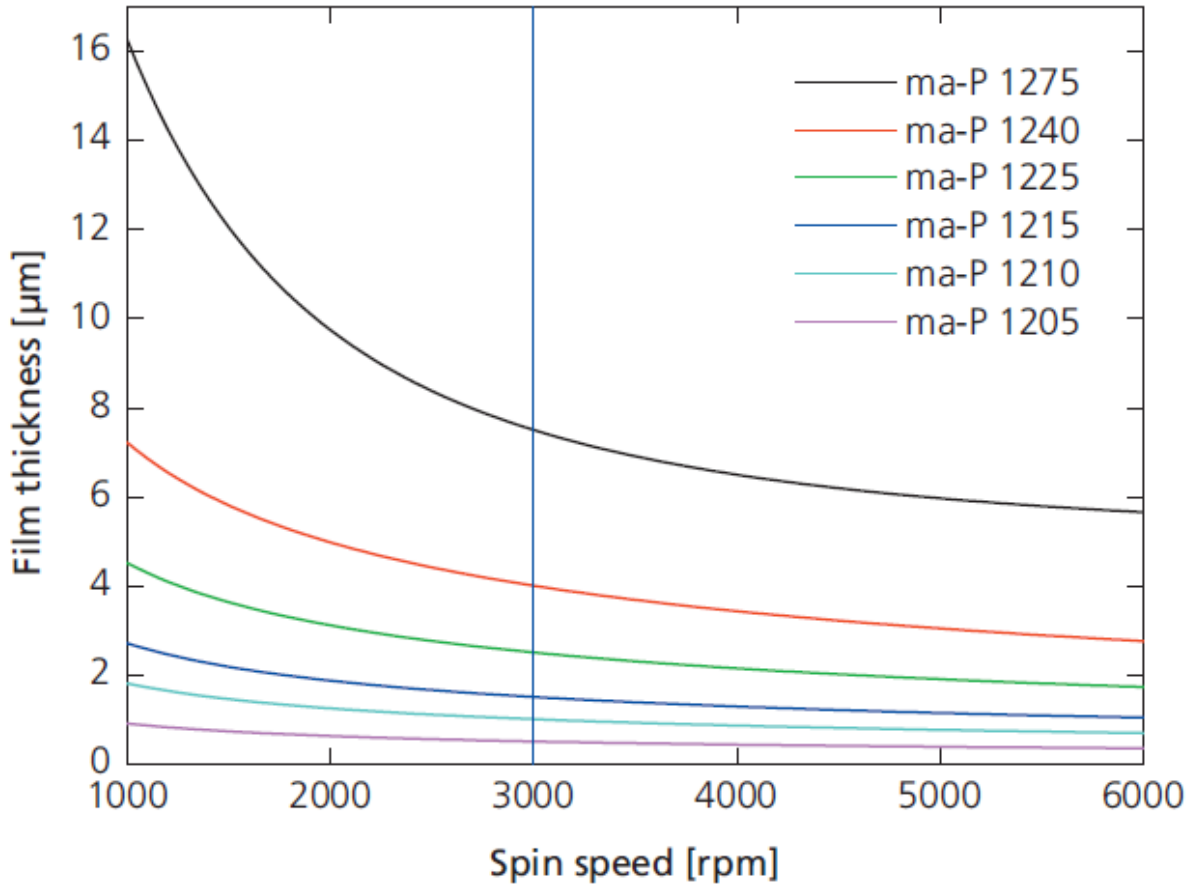


Figure16: The required spinning speed for the desired thickness depends on the material characteristic, especially the one related to PMMA used here is corresponds to the green line for ma-P 1225, thickness was adjusted for 5 µm [15]

The experience result that we acquired during the process is as follows:

PMMA thickness µm	First ramping speed	Second ramping speed
4.8-5.2	400 for 10s	1660 for 60s

Table 1: the resist characteristic well defined for spin coating program

Having resist the chip without any intermediate step we bake the chip at 100 °C for 60s table 1, in this step the resist will be thoroughly stacked to the surface and for removing extra amount of the resist solvent, hot plate named ***EMS HOTPLATE*** is exploited.

Immediately after baking the resisted sample we have to make the exposure by means of UV light, it is well done by an instrumentation named MASK ALIGNER, the mask aligner used in the lab is named: ***Karl süss model mj23***

The mask aligner used in the lab is operated by means of a mercury lamp that is power supplied to orientate through sequential lenses and expose the surface structure using UV light, operated by 350 watt in high pressure regime. Of course for the area of experience with different applications we need to hide and bare some selective zones on the chip surface so as to define the pattern, for that reason we use an auxiliary intermediate object to transfer the pattern into the resist thin film coated to the surface, **MASK** is a piece of glass decorated by almost chromium on its surface using comer data printing or three dimension printing to define the pattern needed for the lithography on that. The mask that we are interested in is composed of 3 **separated rectangular structures** as follows to be patterned on the surface:

The most important factor in exposing step is time of exposure, the threshold for achieving the best and clear pattern on the surface is related to the time needed for the light intensity to illuminate the mask and transfer the pattern onto the surface; due to the experience we repeated the experience for time ranging from 4 to 9 seconds, for different attempts.

After exposing and releasing the material out of the mask aligner, immediately the surface must be developed, in this case all the unwanted remained from exposing will be eliminated to give rise a well-defined clear pattern on the resisted structure.

The developer is a type of chemical solution that we used by the commercial named: **MF219** to dissolve the resist in exposed regions as the positive photoresist. There is a trade-off between the exposing time and developing time table 2 that has to be considered in terms of dust or contamination remaining on the surface or harshly washing out all the resist patterned when developing time is taking longer than a threshold. For that reason we came with an idea for the exposing and developing as follows:

Exposing time	Developing time
4-9	15-25

Table 2: The corresponding developing time for exposure to be well introduced

After development the sample must be rinsed into DI water and be dried by means of nitrogen plastic gun.

3.3 Deposition and Lift-off

The next step toward Si backside-etching is metallic thin film coating. The idea behind this step is back to the concept that during the etching the temperature may affect the resist melting, to overcome this issue, the state of art is that to use gold as the etching mask when KOH is applied as an etchant solution for selectively degrade the silicon depth from the standard thickness of the substrate. Considering this factor we have to overcome another problem, since the silicon is already oxide in the normal condition as an inevitable factor, the substrate will not tolerate to preserve adhesion to the gold thin film, for that reason an intermediate auxiliary metal has to be deposited as middle thin film before gold deposition, say titanium. The reason for using TITANIUM as the sticking layer for gold deposition is related to the weak chemical bonding of gold to the silicon or oxide silicon layer, that could be detrimentally affected by temperature increasing during the deposition process and related reactions, for that reason Titanium is used as a sticking so as to optimize the surface for better nucleation of the gold to the surface.

The desired thickness for gold evaporation onto the substrate is related to the experience, actually we are interested in 25 and 100 nanometer thickness for the

gold, the corresponding thickness for the titanium as the sticking layer will be 5 nanometer.

The experience for both gold thickness after etching manifested that the one that we used for etching with lower gold thickness (25nm) will be easily flake out during etching, according to the stiffness and weak chemical bonding of gold onto the substrate after lift-off. The morphology of the substrate for both of the thicknesses after etching can be tremendously different. Mechanical stability for better and uniform etch rate desired for the etching has been proved.[26]

For the deposition we use electron beam evaporation instrumentation for thermionic emission, named **EUROVAC.** Using high pressure evacuation and two sequential deposition process we come out with a two level thin film deposition on top of the surface patterned by photoresist layer. The table shows different aspects related to the experience, in terms of the instrumentation parameters table 3 and desired thickness.

Material	Thickness (nm)	Rate (Å/s)	Current
Ti	5	1	0.8
Au	25	2	0.9

Table3: for different material the adjustments of electrical properties are different in terms of the power supply for heating and crucible performance

Operation procedure for the deposition tool is based on the thermionic emission using electron beam bombarding the target source for melting. The mechanism for emission is related to filament heating by means of power supply, and then increase the related current and rate for the desired illuminance for bombarding, the directions for orientation must be well positioned in the middle of the hole during bombardment, and the frequency for X-Y directions must be adjusted so as not to exceed or waste the energy achieve for bombarding. For the thermionic emission we are encountered by gently controlling the proportion between rate and current,

initially by increasing the current there is no sign for rate increment, since the current is directly going to heat the filament, having received a temperature approaching the threshold figure 17 the rate will increasing sharply in a shape of semi-exponential pattern as follows:

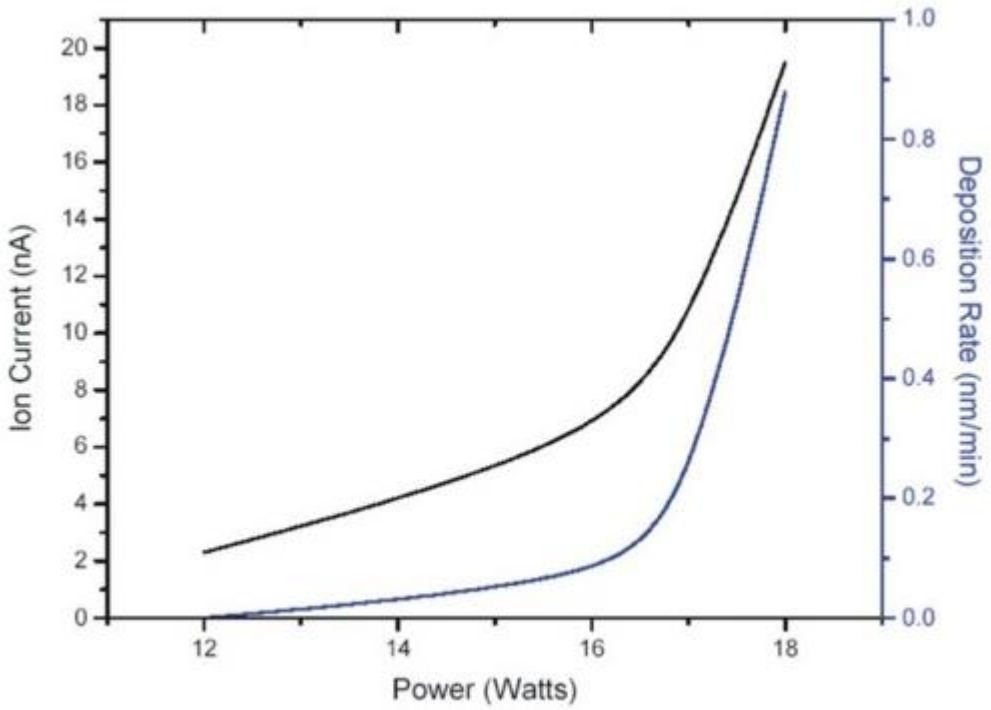


Figure 17: the deposition rate versus the pumping energy required for filament heating the electron beam, the cut-off shows semi-exponential behavior [13]

Having finished the deposition process, the sample is decorated by titanium and gold thin films, however the resist is already attached to the surface beneath the coated films, the next step is to take a method for photoresist elimination, for that reason we have to use solvent to wash out the sacrificial layer form the structure, and remain the coated films in the selected desired locations to be ready for etching. The process is called Lift-Off.

Using ultrasonic tool adjusting the temperature on 45 °C we will sequential immerse the samples in ACETONE and then IPA respectively for 5-15 minutes depending on the gold thickness, gently the sonication will wash out the resist layer and the well-defines silicon substrate already deposited by thin films of titanium and gold be achieved. After Lift-off the sample is ready for etching, in order to get a certain idea

about the thickness of the coated films one way is to use contact profiler figure 18 to mechanically probe the surface and determine the thin film thickness as follows

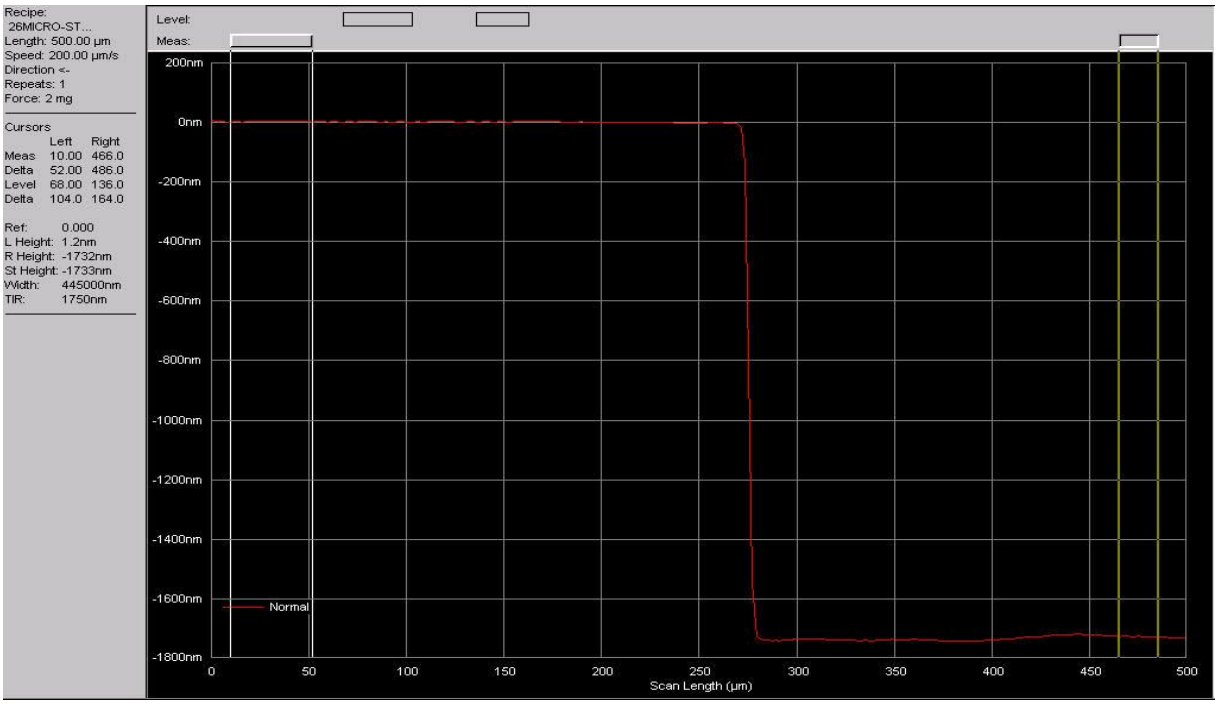


Figure 18: the contact profiler record acquired from the elevated surface probed

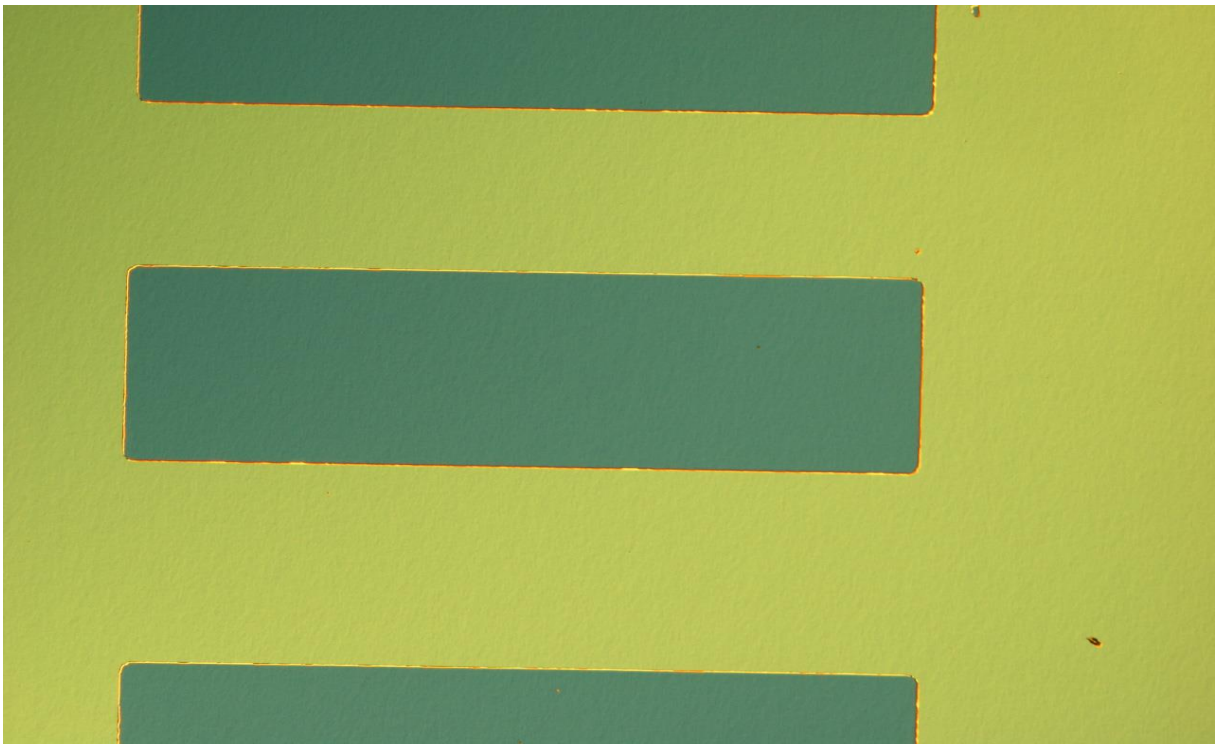


Figure 19: Optical microscopy from the lifted-off sample deposited by titanium and gold, the windows for etching selection are colored in dark green, the background

thoroughly is in grassy color, while the lift-off figure 19 remains the residue of the sample on the gold part declared in brown dots, could be detrimental after etching.

3.4 chemical etching

Till now we have prepared the substrate to be introduced for etching, the target of the process here as the last fabrication step is aim to define some trenches inside the silicon substrate, i.e. thinning the backside of the substrate well-enough for light transmission optimization. The type of etching method to be used here is based on anisotropic etching in selective locations defined on top of the silicon surface, the addressing point is the trenches realization alternatively between sequential orders of the metallic deposited regions, called wet or chemical etching. For that reason we use KOH as the chemical etchant in liquid phase for different concentration diluted in DI water, the etching mechanism that we are dealing with is based on chemical reaction and diffusion happening on the surface region of the etched material.

We are dealing with 3 case studies:

1. Resisted substrate before scribing and same fabrication process
2. Bare substrate scribing and typical fabrication process
3. Boron doped substrate and typical fabrication process

All the above mentioned cases will be introduce for etching and the results will be reported, in terms of the etching parameters. We will soon realize that using boron as a doping contamination in silicon substrate will drastically reduce the etching rate that could be treated as etch stop factor, in selective etching procedures, especially for superficial etching in the silicon depth.

Starting the procedure KOH must be prepared, for that reason we need to dilute KOH powder in DI water to achieve the required solution percentage, however, in Nano lab facility in Alba nova we make use of the defined percentage of diluted KOH for 47% percentage brought from markets. Then the corresponding initial molarity of the solution regarded to the percentage and related chemical parameters such as molecular weight and density of the dissolved solution must be taken into account for calculating. Then after the final concentration will be defined and the

required volume will be determined using equilibrium functions in chemical reaction, as follows:

1. 470 gram of KOH powder diluted in 1000 ml DI water to result in KOH with percentage of 47% as dissolved solution
2. In order to find the appropriate density related to the solution

$$d^1 = \frac{m(KOH+DI\ water)}{v\ (DI\ water)} \quad 3.4.1$$

So: $d^1 = \frac{470\ gr+1000gr}{1000\ ml} = 1.47\ gr/ml$

Considering that the density of DI water is: $1\ gr/ml$.

3. The initial molarity $\underline{\boldsymbol{Cm}}$ can be derived knowing some chemical parameters

- $\underline{\boldsymbol{Cp}}$: Weight percentage 47%
- $\underline{\boldsymbol{M}}$: Molecular weight $56.11\ gr\ /\ mol$

Using the formula as follows:

$$Cm = \frac{Cp*d}{100\%*M} \qquad 3.4.2$$

So the value for the molarity of the initial solution is: $\underline{12.3\ gr/mol}$

4. Defining the required molarity we have to put an initial volume for the KOH solution and then find the related amount of DI water needed so as to achieve that molarity for the final solution of KOH + DI water, using the formula :

$$C^1V^1 = C^2V^2 \qquad 3.4.3$$

C^1 : The initial molarity of the KOH solution identified as Cp

C^2 : The final molarity of KOH dissolved in DI water

V^1 : The initial volume of KOH before solving

V^2 : The final total volume of KOH diluted in DI water

In order to find the related parameters we are willing to dedicate to final molarity for the experience to be done:

- $Cm^1 = 5\ gr/mol\ \ \&\ V^1 = 100\ ml$

- $Cm^2 = 8.15\ gr/mol\ \ \&\ V^2 = 200\ ml$

The related volume for each final molarity adopted for the DI water to be added will be as follows:

- $Volume\ of\ (DI\ water)\ for\ Cp^1\ is\ 146\ ml$
- $Volume\ of\ \ (DI\ water)\ for\ Cp^2\ is\ 102\ ml$

Theses valued found from the final volume of the solution extracting the initial volume of KOH from that, i.e. $V^2 = V^1 + V\ (DI\ water)$

5. Having the final molarity we need the related percentage of that, since the density will be different for the final solution.

$$Cm^2 = \frac{Cp^2 * d^2}{100\% * M} \qquad 3.4.4$$

In turn the related percentage will be achieved knowing the density d^2

$$d^2 = \frac{m\ (KOH\ +\ DI\ water)}{v\ (KOH + DI\ water)}$$

Considering that:

$$m(KOH) = d^1 * v(KOH)$$

Using the equation for final molarity we ultimate with the percentage for both cases to be as follows:

A. Initial molarity and concentration : $12.3\ mol/lit\ \ \&\ 47\ \%$
B. Final case for $v^1 = 200\ ml$: $8.14\ mol/lit\ \ \&\ 34.9\ \%$
C. Final case for $v^1 = 100\ ml$: $5.00\ mol/lit\ \ \&\ 23.6\ \%$

So for two different diluted solution we can proceed for etching.

Beside to the solution concentration and related composition, we have to determine the effect of other included parameters in both cases.

Temperature is the most dominant factor controlling the reaction process, previously we have seen that etching rate has exponential dependency with the temperature due to **Arrhenius** formula.

Also it must be considered the mechanical factors such the rotation factor stimulating the process, in a sense that the solution thoroughly is placed on a hot plate that stirring with a fixed rotation index, **RPM,** the duration of time regarded to

etching process is also taken into account, the more gentle be the process, the more ideality will achieved in the reaction, similar to the etching time, the gentle process is granted when the temperature stability will be considered, i.e. less temperature gradient, and more maintenance, also the other factor is the positioning of the structure inside the liquid, in what follows we will report the effects of the structure and geometry that how will alter the etching rate duly for different positioning for chemical etching.

Nearly we have mentioned all the included factors in a way affecting the etching rate, however, there is another phenomenon that in turn can be impressive in terms of etching rate and surface morphology; many reports has been done using different types of alcohol to analyze the rule of adding alcohol and its effect on the surface morphology and etching rate, among all for the KOH etching we will proceed by using different amount of alcohol concentration, named ***IPA***, to report the etching rate and surface roughness achieved for each case, in terms of saturation and surface chemical factors.

According to has been mentioned the factors encountered are:

- Concentration of entities (KOH + IPA)
- Temperature
- Stirring speed and etching time (more ideality in reaction)
- Structure positioning inside the liquid for etching and crystal plane
- Adding IPA to the solution
- UNDOPED vs DOPED silicon substrate
- Gold thickness as the mask layer

Considering all the methods mentioned above figure 20, we start etching along preferred crystal plane <100> [11], considering that etching along <100> and <110> is two order of magnitude faster that along <111> plane, trenched structure will be etched inside the Si structure. The challenging point is to stop the etching before complete etch the substrate, but make it as thin as enough, for that reason observation via IR or visible light transmitted through the holder hanging the structure inside the silicon will be a proposed solution, the threshold for the reaction due to the thickness of the substrate is the order of microns that in turn equalized to the infrared or red visible light [12].

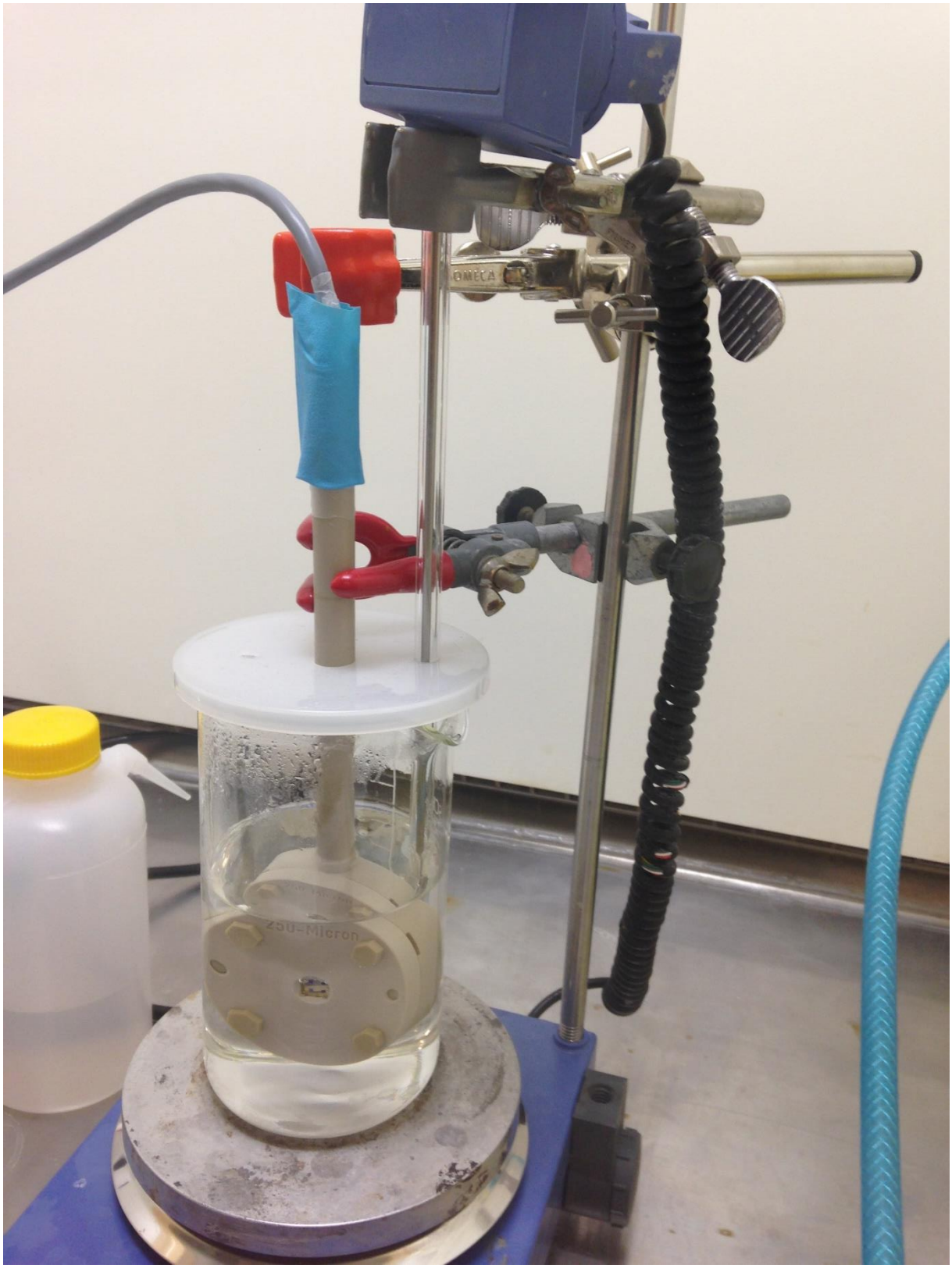


Figure 20: assembly of the chemical etching process, the hotplate and thermometer all well-equipped for the temperature and rotation adjustment, also the sample is fitted inside the holder which in turn is hanged into the solution elevated to a height above the magnetic pill responsible for solution stirring, also IPA is used

continuously so as to control during the experience overcoming the evaporation slightly.

The admitted depth that we are interested in is to reach for further than 240 micrometer out 270 micrometer silicon depth for the substrate; as thin as the substrate will be, the light transmittance will approach its optimized value.

Starting the procedure, the temperature adjusted for two values separately on the thermometer for maintenance: 75°C and 80°C, the stirring speed of the hotplate will be adjusted for three value separately as: 4500, 6500, 700 RPM, the sample hanged inside the appropriated facilitated holed with IR connection will be immersed inside the solution to be put on the hotplate. We will etching for more than 3 hours to be ensured about that less turbulence and higher ideality will be achieved for the gentle etching reaction process for decreasing the roughness, between 230-328 min.

Due to the evaporation geometry the sample will be positioned inside the holder and in turn for the etching reaction in horizontal orientation figure 21, of course the vertical orientation will be studied for the same condition, the doped silicon and already scribed resisted wafer will be evaluated and reported too.

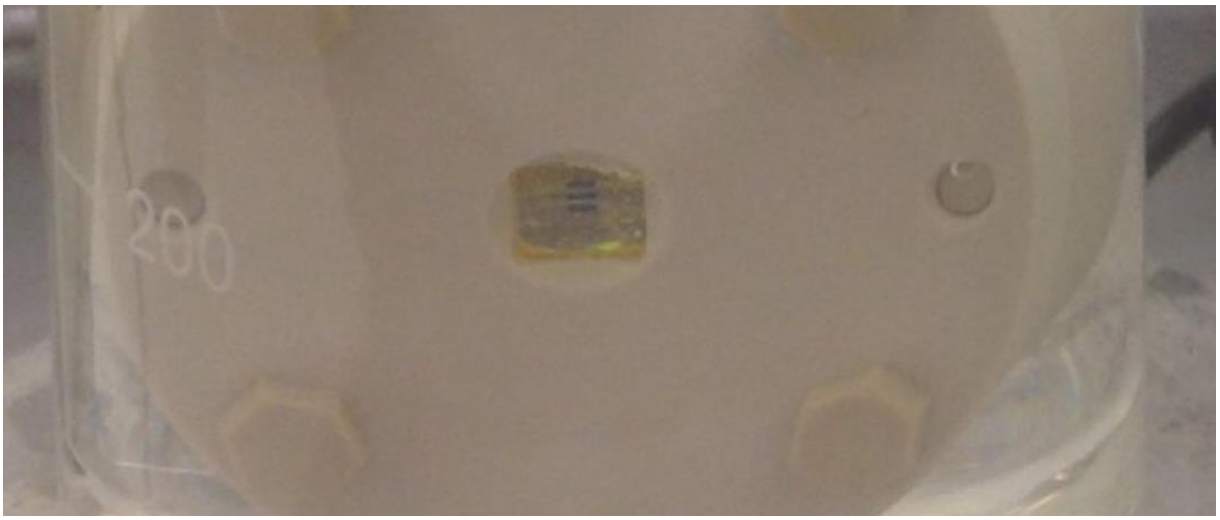


Figure 21: the well-positioned fitted configure of the chip inside the holder to be etched by the KOH solution, the horizontal windows are etched uniformly, however, always a gradient of reaction happens due to the upward diffusivity of etchant.

IPA for different amount and concentration will be added and the effect will be reported, the idea for adding IPA is backed up by two reasons:

- Crystal plane <111> suppression
- Decreasing surface roughness

The idea for the IPA adding also is impressive when the report is justified due to the surface tension and surface adsorption density for the saturation case.

Different uncontrolled factors **like bubbles** will affect the etching performance in terms of etching rate and surface roughness, for that reason the positioning of the structure and perfection of the sample is required, especially when there is a defect or tiny corrosion in the edges to open up a gate for the solution to penetrate back into the sample and etch it reversely. The other factor degrading the etching rate is due to the inevitable exposing to oxidation, always there is an amount **of oxide attacking** the surface and making that as a silicon oxide surface, giving rise for insulating the surface from the KOH etchant to penetrate into the depth of the substrate. For that reason one method to decline is factor is to put the sample in plasma etching process for deoxidizing the surface as much as possible, this is called KOH etching pretreatment that could be used in future for optimizing the etching rate.

Proceeding for the etching with all the different wafer natures (bare, doped, resisted scribed wafer) and different parameters bring about to different consequences in terms of surface roughness and substrate thickness which measured using optical profiler as an interferometry tool for metrology.

The idea for adding solvent like IPA can be analyzed for the surface energy of silicon and the adsorption density, in a sense that adding alcohol to the solution give rise to gradient in adsorption density of the surface to be maximum well fitted for the minimum etching rate (complete formation of monolayer on Si surface) also adding more alcohol and approaching the saturation the chemical bound monolayer attached to the surface will gradually bread down and disappear to consequently increase the etching rate. So the etching rate will behave like a convex to start for a value that declines by increasing the alcohol concentration, more increasing the alcohol for saturation level, the monolayer breakage, will give rise in increasing the etching rate to higher value, corresponding to higher adsorption density and less

surface tension for the case of additive alcohol procedure that in turn surface energy of Si is considered for adding solvent.

The structure already etched figure 22 decorated by Gold mask is ready to be positioned for the front side electroplating or Metal assisted chemical etching (MACE). The state of art in during the etching from backside was to preserve the front side polished and undamaged as much as possible to be a clean working field for MACE.

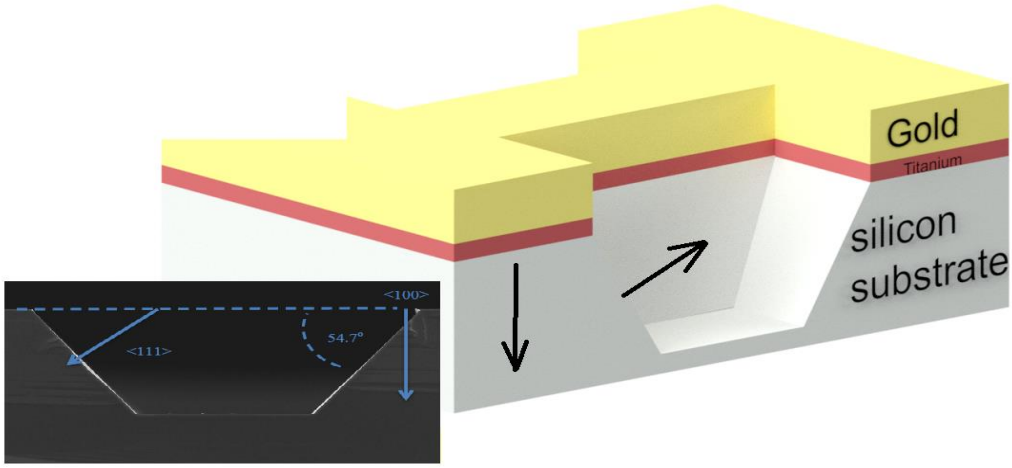


Figure 22: the simple finalized target structure realized for silicon backside thinning for light transmission optimization, the resist patterning of the surface followed by electron beam evaporation of titanium and gold for 5nm and 100nm respectively performed. The structure is thinned along the <100> crystal plane (vertical arrows) to have a vertical angle equal to 54.7 ° with the sidewall plane <111> (oblique arrows). The substrate thickness in its ultimate value trenched for 245µm with respect to the total depth of 270µm, gave rise for the light transmission in the range of red-IR spectra, used for the electroplating of the front side zone plate. The idea for the suppression of the <111> plane with respect to the <100> is extremely depends on the additive IPA used for stimulating the etching mechanism.

3.5 Results using Interferometry

Having etched the sample for different parameter changing and wafer used we are about to come with an idea about the thickness and index of roughness for the structure etched, for that reason after optical microscopy for observing the sample for different magnitudes, we have to use optical profiler using interferometry tool to measure the parameters needed. For this case we run the software WLI and place the sample already etched on the plate, then we have to focus the beam on each level required to be exposed for optical beam measurement, having etched the sample we have two tremendously different levels elevated from each other in the side wall of the cutting edge between the Gold mask and the already etched surface at the bottom, say the boundary or the ramp region (vicinity to the <111> crystal plane, which is transparent due to the total light reflection), then the beam must be focused separately for each level and the fringes adjustment to be equalized by one per each screen focusing must be achieved. Then after the fringes will measure the thickness and regional roughness counting the optical path difference for sequential reflections they are processing in each step, in a sense that equally high fringes give rise to equal level or thickness in each trench region been processed by the reflected ones. Eventually the measurement for each sample with specific etching parameters during etching gave rise to the results as follows:

- $\underline{75\ °C}$: the etching rate for KOH 8.15 mol/lit ,without adding IPA was $0.731\ \mu/min$, adding IPA at the same condition degrades the etching rate to the amount of $0.662\ \mu/min$, then again adding IPA more to the saturation level will finalize the experience for increasing the etching rate to the ultimate amount of $0.737\ \mu/min$. Also the same happening for less concentrated KOH equally $5\ mol/lit$, that for the saturation case of IPA adding the rate is $0.674\ \mu/min$, while for less IPA concentration the rate is reduced to $0.598\ \mu/min$.

  Adding IPA to the solution at low concentration, give rise in single molecules of the alcohol to be adsorbed, higher concentration of alcohol bring about alcohol molecules aggregation and surface monolayer creation, to decrease the etching rate, then after adding more alcohol will take place in saturation level for the

concentration and chemical bonds breakage to disappear the monolayer from the surface, it will resume the etching rate for higher amount.

- Less stirring speed by the amount of 500 RPM, give rise in etching rate declined to $0.728\ \mu/min$, at same condition and IPA concentration.

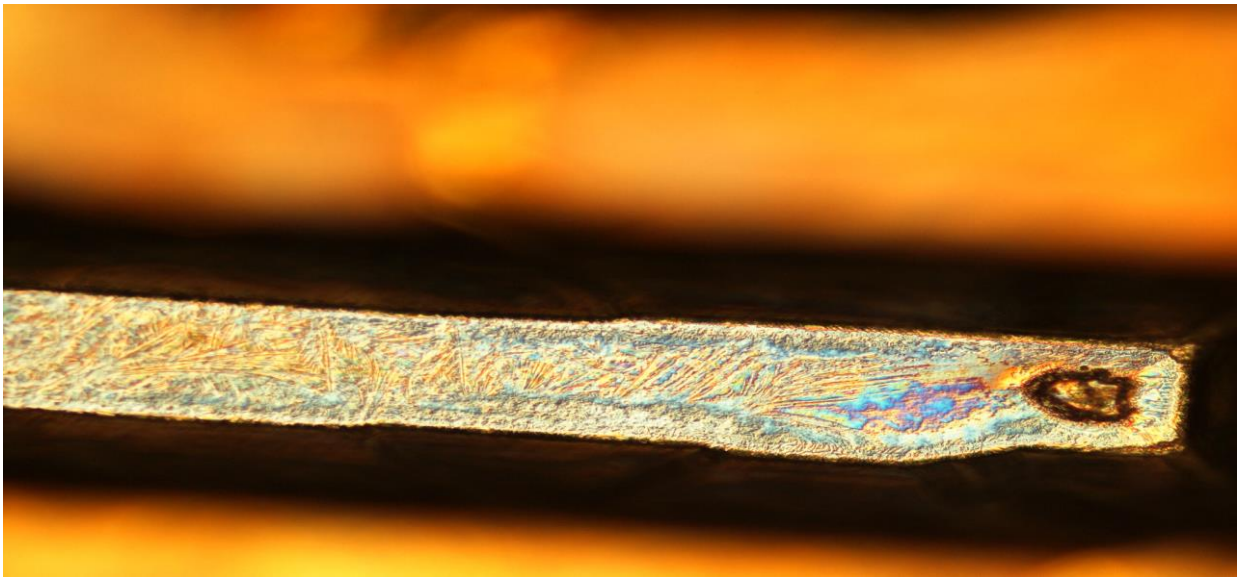
  Decreasing the etching rate for the less stirring speed is due the influence of mechanical stimulation that in some cases is responsible for eliminating the bubbles from the etched surface so as to resume the declined etching rate, the surface coverage by bubbles has very important rule in hindering, slowing down the chemical reaction for etching and accordingly the effective surface.

- Higher etching rate achieved at the same condition when the substrate used for etching was completely bare, without any etching mask, to the amount of $0.747\ \mu/min$, less diffusion.

  The etching mask will reduce the etching rate, due to less degree of freedom for etchant mobility attacking the surface to penetrate into the sample, the higher will be the aspect ratio for the selected etching mask regions, the less will be the etching rate, and the higher will be the restriction (less diffusion).

- Positioning the sample in vertical direction perpendicular to the hot plate, give rise in higher etching rate mounted to $0.766\ \mu/min$.

  For the etchant solution, the chemical reaction is approaching always the surface, putting the sample in vertical orientation give rise in higher etching rate, however due to the anisotropic diffusion and chemical reaction, the etching will be non-homogeneous.



The upper level is the right sidewall

Figure 26: non-uniform etching pattern for the silicon substrate due to the vertical positioning of the structure inside the solution, due to the diffusion and upward flow of etchant, the etching rate is more gradient to the upper half of the structure to the extent of removing almost the upper sidewall of the windows declared by the ultimate right-sided circular trench near the sidewall

- Increasing the temperature to $80\ °C$ and considering that the boiling point of IPA is about $82\ °C$ (faster evaporation and breakage of hydrogen molecule bonds), the etching rate has three phases when IPA is introduced: for low IPA concentration the etching rate will be higher for less IPA equal to $1.022\ \mu/min$, for higher IPA concentration below saturation, etching rate is $1.008\ \mu/min$, for the highest amount of IPA about the saturation point condition, $R = \ 0.996\ \mu/min$.

  The Idea behind this changing is due to the problem of IPA boiling point, which is in vicinity to the etching temperature. So there is a tradeoff between etching rate dependency to the temperature as a reinforcing factor for etching rate, in turn be temperature will be a detrimental factor when boiling point is approaching, so controlling the reaction rate is challenging. So for the highest concentration of IPA around the saturation point the monolayer breakage is controlled due to the assured thickness of the monolayer that make the whole monolayer evaporation as slow as possible, however for the less amount of IPA below saturation, the less be the IPA amount the faster will be the monolayer breakage due to evaporation point, so the

phase for thick enough layer of alcohol and less evaporation give rise for the etching rate to be the less around saturation point.

- At 75°C and saturated IPA, for 35% KOH, rate is $0.750\ \mu/min$, the 26.8 % KOH is corresponding to $R = 0.689\ \mu/min$ and for less concentration of 23% KOH, the rate is degraded to $0.607\ \mu/min$

  This pattern is due to the fact that for the saturation condition, the less being the concentration of KOH, figure 25 the higher will be the amount of IPA for saturation, also at the same condition for the temperature and alcohol saturation, the higher will be the KOH concentration, the etching rate will be higher.

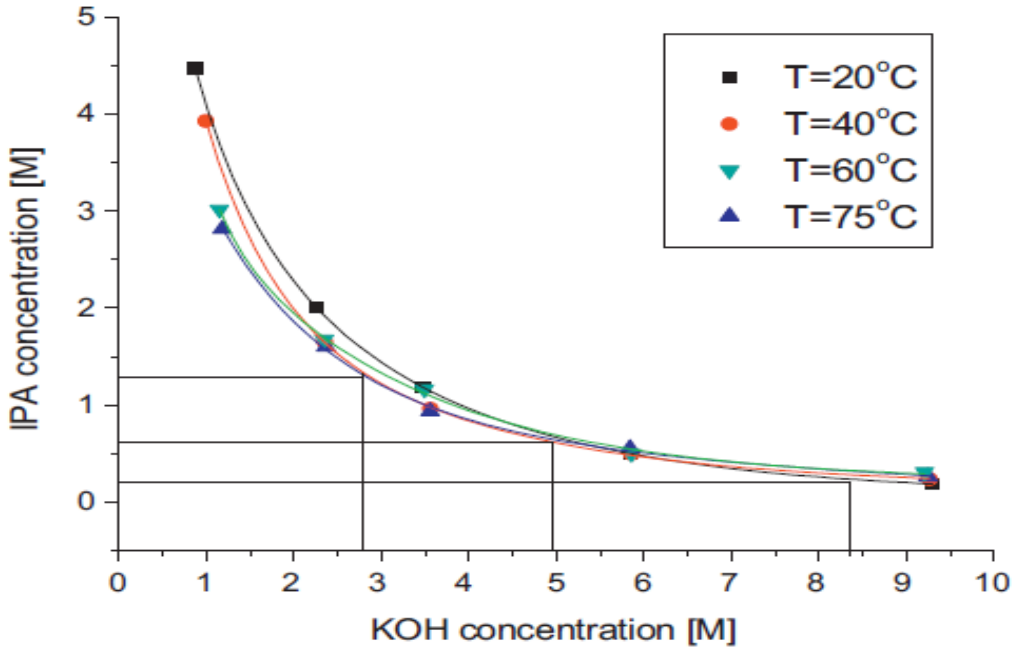


Figure 25: Saturation of alcohol concentration versus amount of chemical etchant (KOH) for different temperatures are shown, the higher being the chemical etchant concentration, the lower IPA concentration required for saturation [9]

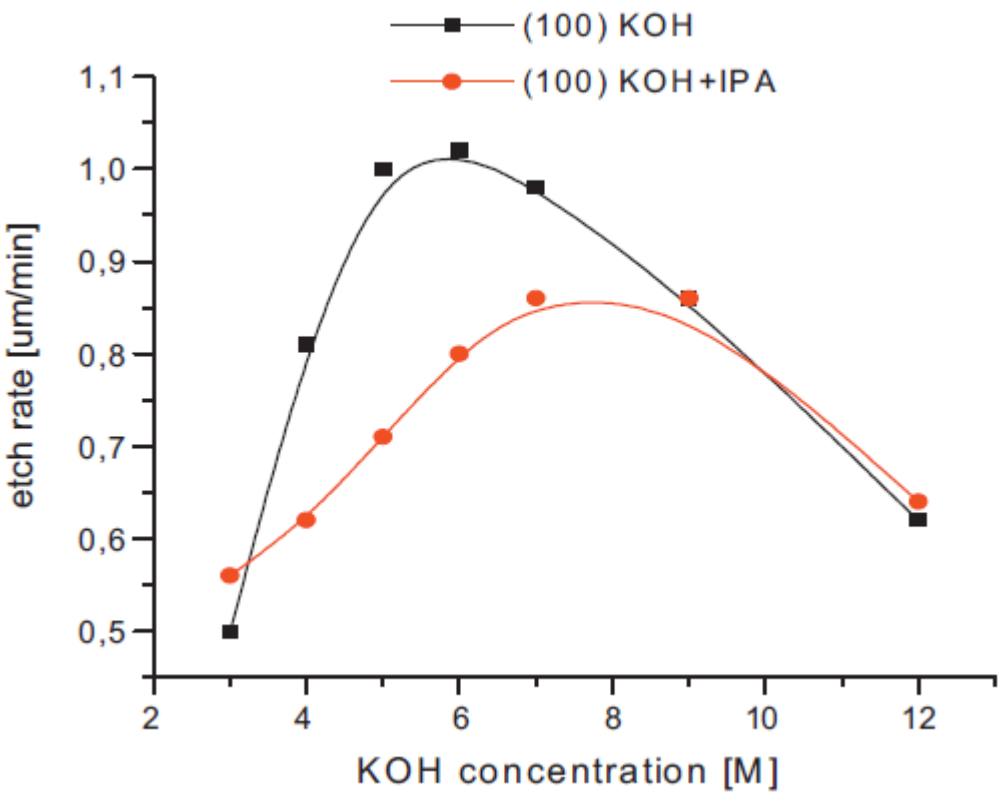


Figure26 : additive alcohol concentration for chemical etching give rise for higher etching rate, when the KOH concentration is increased, comparing 8 M to 5 M, the etching rate declined to 100 nm/min for more diluted solution [7-9]

Also more alcohol concentration is required to decline the surface tension and approaching saturation level is more demanding when the KOH concentration is going to be decreased figure 26.

- Increasing the temperature for $\Delta\theta = +5°C$, give rise to higher etching rate from $R = 0.662\ \mu/min$ to $R = 1.022\mu/min$ for same condition, however, the roughness index $Rq\ (RMS)$ is increased for the higher temperature from $12\mu m$ to $38.3\mu m$.

  Increasing the temperature the higher will be the mobility for the etchant to participate in the reaction for etching; theoretically due to the Arrhenius formula the etching rate in turn has direct exponential dependency to the temperature. Also regarded to the roughness, the higher will be the temperature, the lower will be the contact angle, elastic modulus, and high hillock with rough surface will give rise for higher surface roughness [10]

- Using resisted wafer before scribing from the beginning, will leave the sample with high degree of passivation against the chemical reaction, hindering the etchants from

targeting the surface and less selected bare regions will be exposed for chemical reaction, exactly same as the scenario for high degree of oxidation on top of the substrate surface, depriving the chemical reaction from proceeding, giving rise to etching rate valued about $0.311\ \mu/min$ exactly for the same conditions.

- Using doped silicon for the etching (lower wafer resistivity around 3 order of magnitudes in $\Omega.cm$), the carrier concentration will be different, in turn the hole injection will change, oxidants and etchant concentration reactions bring about for lowest etching rate. The etching rate will be translated as the etch stop issue, in a sense that for the different etching time, the doped substrate will control the etching depth for the thickness of $6.5\ \mu m$, say $R = 0.02\ \mu/min$, increasing the doping concentration figure 27 results in resistivity decline and in turn etching rate will approach to a cease point as etch-stop characteristic of the related concentration [8]

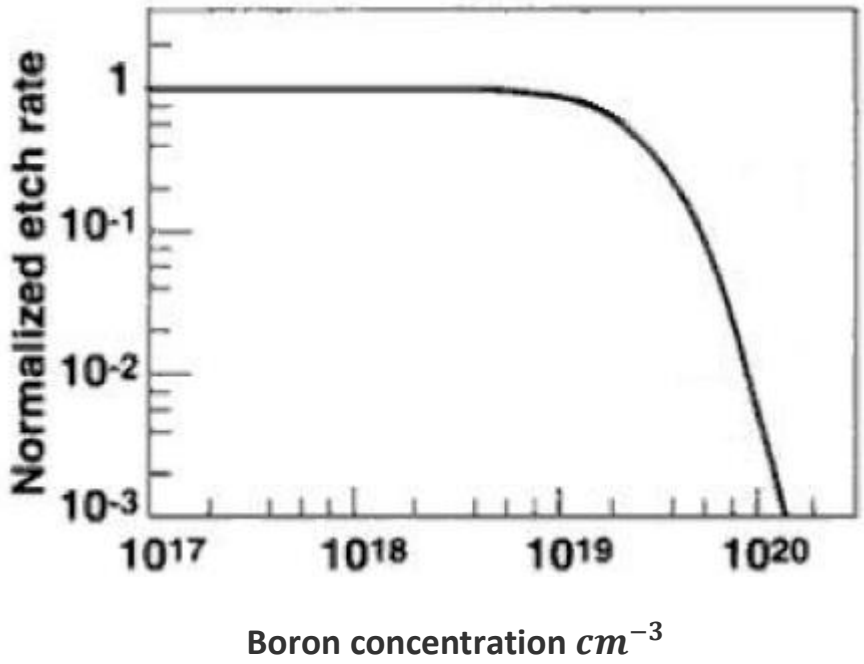


Figure 27: Adding boron as doping concentration issues for degrading in etching rate and ultimately cease the process when it approaches the corresponding value around 10^{20} or resistivity equalized to $0.001\ \Omega.\text{cm}$ [7]

This technique is called etch-stop by the method of heavily doped substrate, used for anisotropic etching using KOH, the higher doping concentration of boron will block the surface (etching suppression along <100> direction) from chemical reaction with etchant, giving rise to very low degree for etching rate.

- Among all arrangements for positioning and etching parameter adjustment, two cases gave rise for the best receipt due to the restriction imposed during etching to optimize that in its category for fixed amount of KOH molarity table 4, for the vertical case the analyzed record is manifested figure 28.

$Temperature\ (°C)$	75	80
$C.KOH\ (mol\ /lit)$	8.14	8.14
$IPA\ volume\ (ml)$	50	30
$Etching\ time(min)$	320	240
$Etching\ depth(\mu m)$	**245**	**242**
$Position\ (V-H)$	***Vertical***	***Horizontal***
$Rate\ (\mu m/min)$	1.008	0.766

Table 4: the optimized values for the depth etched are listed due to their characteristics

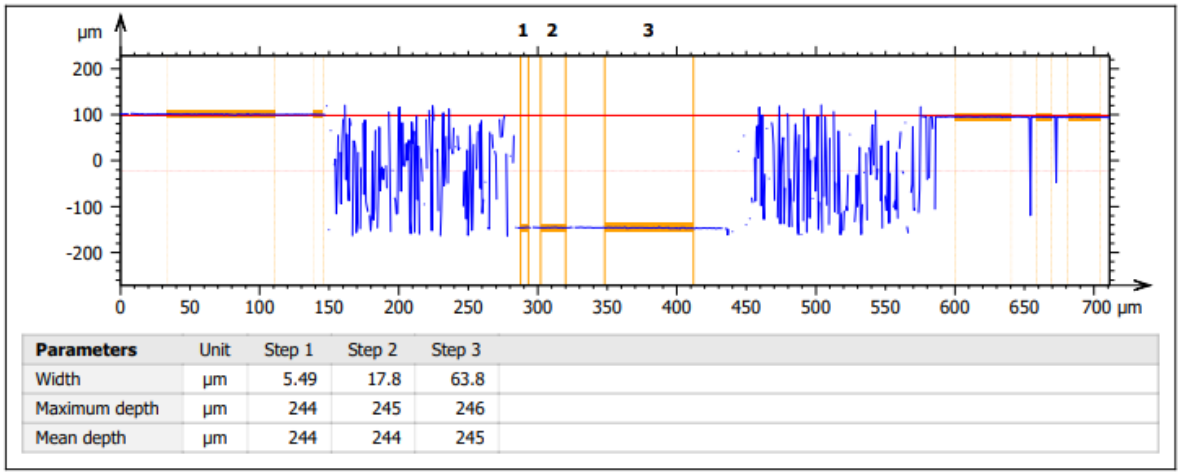


Figure 28: the optical profiler analysis of the structure by three sequential height measurement depicted for the vertical positioning of the structure.

the Spectrum-plot manifests the morphology and topography of the surface been analyzed by a sequence of fringes reflected from the surface, the right side blue rectangular shape in the figure 29 is depicted for the trench inside the substrate, <100> crystal plane, which is etched with respect to the elevated remained surface to the left as the gold background surface, the middle boundary which is declared in white is the region where the light incidence is getting out of focusing point bringing about for the transparency of the sidewall crystal plane <111>.

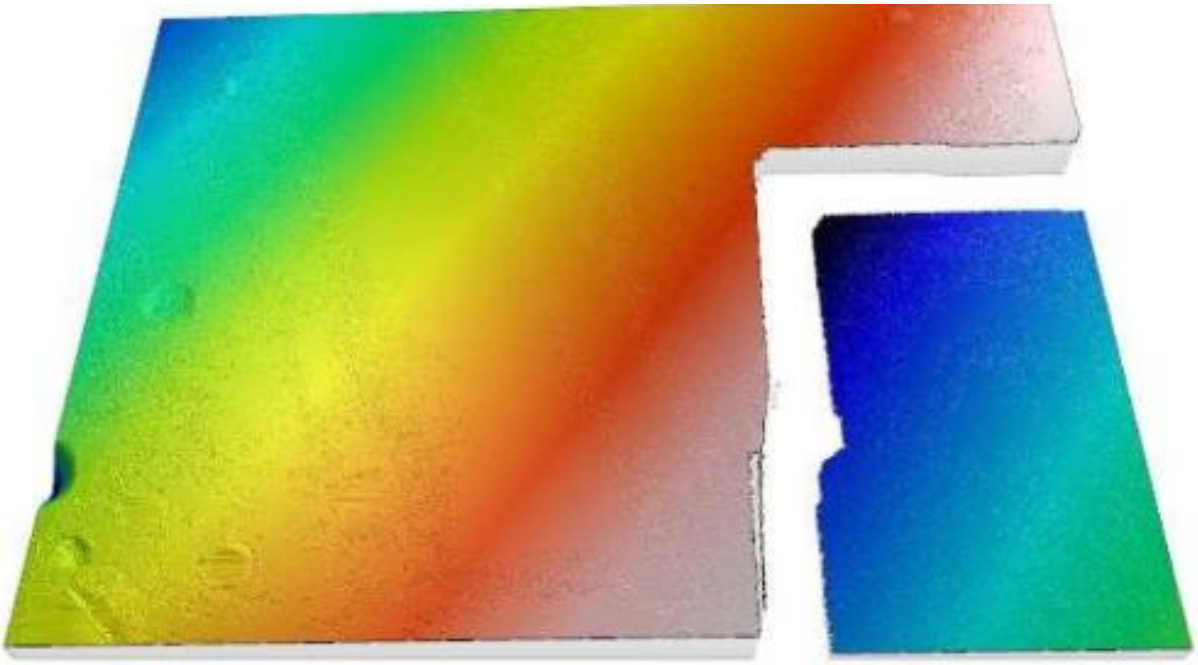


Figure 29: the analyzed sample for trench measurement and the spectrum manifest the elevation with respect to the fringes achieved by reflection from the sample

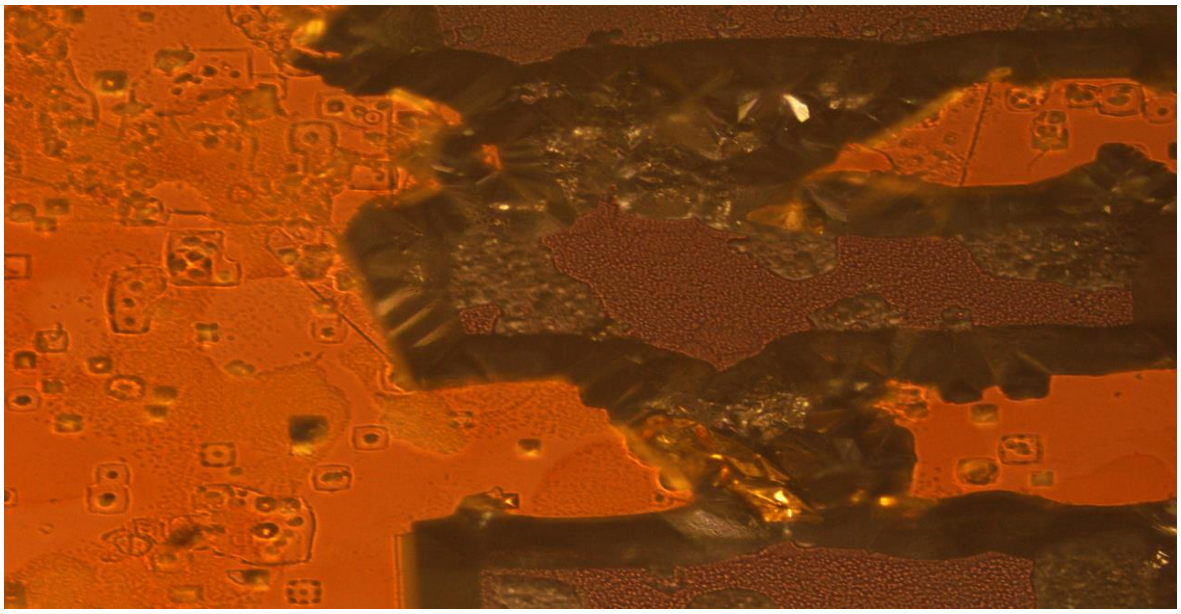
The data collected above are dedicated for the surface roughness of the trenched silicon substrate, the one that we are more interested in is the Rq or RMS value for the criteria assumed for the surface roughness factor, the attempt was to reduce that for 1 order of magnitude, that eventually gave rise to 3.72 µm table 5.

ISO 4287			
Amplitude parameters - Roughness profile			
Rp	10.7	µm	Gaussian filter, 0.25 mm
Rv	0.863	µm	Gaussian filter, 0.25 mm
Rz	11.5	µm	Gaussian filter, 0.25 mm
Rc	*****	µm	Gaussian filter, 0.25 mm, ISO 4287 w/o amend...
Rt	53.5	µm	Gaussian filter, 0.25 mm
Ra	1.95	µm	Gaussian filter, 0.25 mm
Rq	3.72	µm	Gaussian filter, 0.25 mm
Rsk	0.110		Gaussian filter, 0.25 mm
Rku	2.75		Gaussian filter, 0.25 mm
Material Ratio parameters - Roughness profile			
Rmr	0.222	%	c = 1 µm under the highest peak, Gaussian filte...
Rdc	0.213	µm	p = 20%, q = 80%, Gaussian filter, 0.25 mm

Table 5: The surface roughness factors are depicted for the trenched substrate

- Comparing the result for the same condition but different metal thickness as etching mask, figure 30 it is obvious that the detrimental factor for etching all the surface of the substrate is the weakness in the chemical adhesion of mask to the surface to bring about failure in etching.

(A) 25 nm Gold plus 5 nm titanium mask etching metals



(B) 100 nm plus 5 nm titanium, shows better and nice selective etched pattern

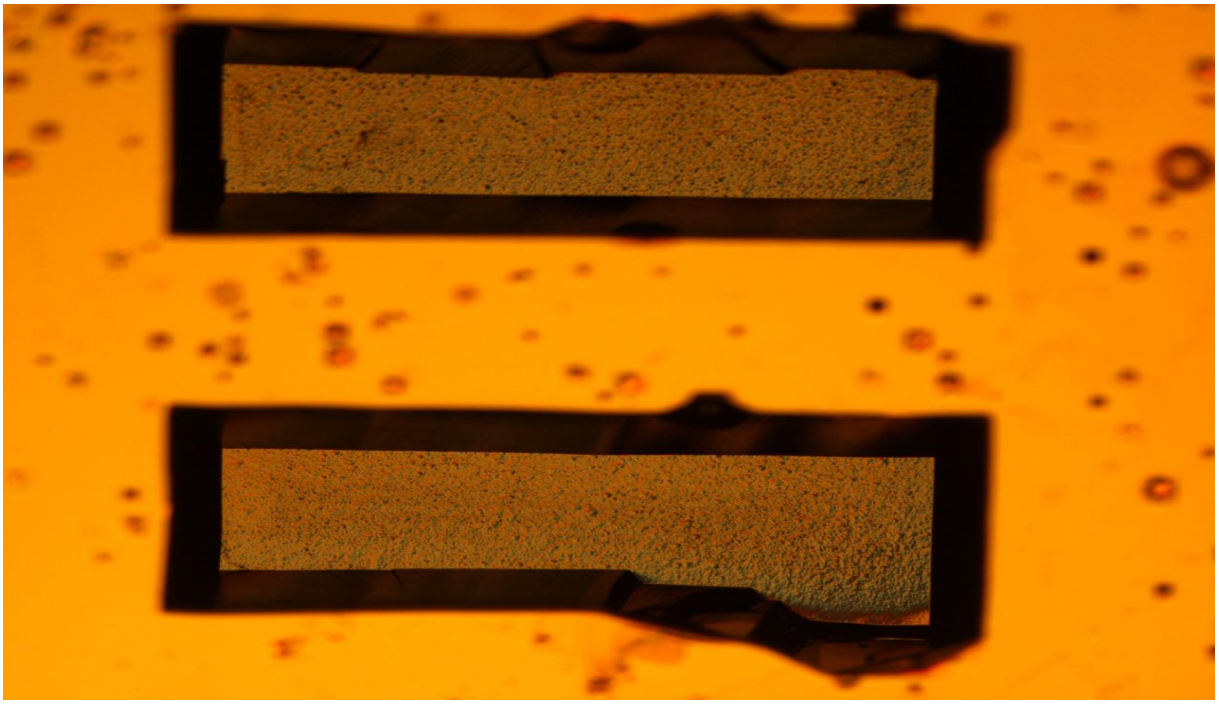


Figure 30: etched process using 25 nm gold deposited film (A), comparing to the (B) 100 nm Gold thickness shows breakage in the connection of sequential trenches.

For all the measurements done above the priority of optical profiler to atomic force microscopy was the able to surface vase region with higher degree of surface with respect to the mechanical movements of the tip attached to the cantilever used to AFM probing (starting from nm upward).

The results of the measurement can be translated in terms of transmittance and wavelength corresponding to incident light for diffraction. The plot shows, figure 31 how the equalized transmission versus wavelength plot can be fitted with the wavelength around 650-750 nm as a cut-off for transmittance achieved.

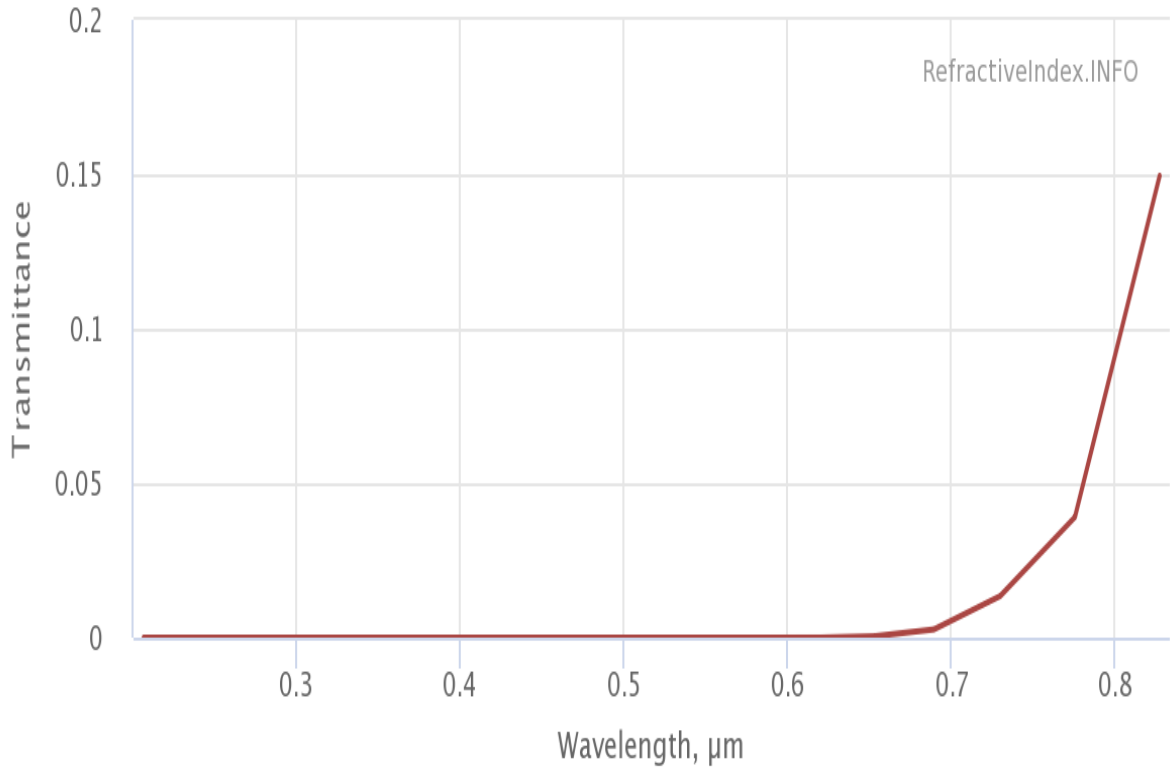


Figure 31: using the reference graph it was proved that the transmittance around the Red Line of light spectrum gave rise to the starting point for transmission cut-off, in a sense that the wavelength for thickness starting from 30 nm down to couple of nanometers corresponds to Orange to IR spectra [32]

Chapter 4

Discussion

4.1 Limitations and Overcoming methods

Having all done step procedures sequentially for the fabrication process, there are always issues to be encountered during the experience, due to the instrumentation calibration and material perfectness.

In terms of the material, the degree of contamination in the lab will always affect the material inevitable deposition of contamination on the surface, the most detrimental candidate to due to so is the surface oxidation and blockage from etching, for that reason one method to degrade the oxidation on the surface is to put the sample in HF (hydrofluoric acid) to decontaminate and deoxidize the surface, which is possible by reaction ion etching (RIE) caring., using plasma assisted process will start thinning the oxide layer from the silicon substrate, considering that 1µm layer of oxidation on the surface will slow down the etching reaction in a way that at 50°C, the KOH etching rate is about 2.8 nm/min for SiO_2 to etch the oxide surface completely and then start etching the substrate surface. This is called for etching reaction as ($\boldsymbol{KOH\ pretreatment - decontamination\ process);}$ the other restriction is issued by using scriber will always give rise for splash depositing on tiny chips due to the cutting and frictions take place for wafer scribing, one method is to use laser beam instead of mechanical method to reduce the effects.

Regarded to the instrumentation always calibration and deformation of the lenses for optical lithography and interferometry affecting the resolution of the tools, these are inevitable factors that could degrade the performance, also the crucible inside the vacuum chamber used for thin film deposition must be orientate well in order to get the best melting energy from the electron beam so as to melt and deposit metals on the surface, also the rate versus current curve has to be considered, increasing the current will suddenly increase the rate in a range named Cut-off rate region, for that

reason the increment for the current must be treated gently and the frequency in all the directions adjusted well to uniformly deposit material on the surface. Regarded to the etching to maintain the processing place as vacuum as possible is important, unless great amount of the solution will participate in diffusion at the interface with the air and evaporation happens. Temperature maintenance is another factor that the error is due to the calibration of the thermometer, homogeneous etching is another case that makes an issue especially at the sidewalls and edges due to the defects of the pattern deposited as mask for etching that in turn is aroused from the non-Ideal UV exposing through the mask, weaken the resolution for exposing cause of the chromium defects on the glass.

4.2 Outlook as a future view

Based upon the work done in the area of diffraction focusing using zone plates, as a future view could be counted for:

- X-ray characterization of the transmission and scattering
- Using conventional light for optical alignment of front side patterned figure.
- Combination of both

4.3 Conclusion

As an intermediate step in Nanofabrication of zone plates, the study of the chemical etching for silicon wafer as a substrate for back-side thinning explained in terms of fabrication steps and the technique to elucidate the realized structure and different parameters. Attempts proceeds so as to achieve the optimized results in terms of thickness, also roughness index and related criteria studied and measured by means of optical profiler. For that end patterning and deposition of material as etching mask has been reviewed and explained for the included parameters affecting the procedures.

For the etching step the use of KOH as a chemical etchant for the chip thinning and methods to optimize that by means of etching parameters also mentioned, considering that due to the previous works the state of art for the etching result to be optimized was the role of IPA as a fostering agent for altering the etching rate,

especially when the temperature was approaching the boiling point for evaporation of alcohol (80-82 °C), the whole work was performed in non-ideal occasion so the rate was affected by different parameters such as oxidation and pressure damping, also the defects and the structure imperfection taken into account for etching non-ideality. Using different type of wafers in terms of doping and method of scribing gave rise for different aspect for etching mechanism that the most familiar one was dedicated for the method BORON-Doped substrate as etch-stop method in etching mechanism to hinder the etching in selective regions. Approaching $245\mu m$ depth out of $270\ \mu m$ each measured by optical profiler, elucidated that the range of transmission for the incident light is around visible light (orange-red) and infrared spectra, when the thickness is around$25-30\ \mu m$, due to the refractive index and Si transmittance plot the cut-off for the transmission will be around $650-750\ nm$ for the wavelength.

The crystal plane priority for etching was on the <100> orientation, thanks to the suppression of <111> when IPA adding is considered. The sidewall transparency also proved by the reflection process using optical profiler that is around 54.5° with respect to the horizontal crystal plane. Also the extremum of the etching rate as a minimum number corresponds to the maximum density of alcohol adsorbed by the substrate surface, explaining the intermediate phase between saturation point for monolayer breakage and also the non-added IPA, which both had higher etching rate with respect to that[33]

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