NCPRE TEXTURE CLEAN BENCH

STANDARD OPERATING PROCEDURES (SOPs) 2020 (v.1)

NCPRE

NCPRE FABRICATION LAB

SO - Sreejith K. P. Co SO-Sandeep Kumbhar

CONTACT: sreejithanni@ee.iitb.ac.in, sandeeo.kumbhar@gmail.com

9930837262, 9969347860

TABLE OF CONTENTS

Equipment Information:

- NCPRE Texture clean bench is customized bench having the heating bath facilities for saw damage removal (SDR), alkaline pyramid texturing Process, hydrochloric acid (HCl) cleaning provided with respective DI water rinsing baths as shown in figure.
- Allowed Chemicals: NaOCl, KOH, IPA, HCl, H_2O_2, HF.

Technical Details:

i. Frame Heaters:

Multiple layers of PFA jacketed heating coils affixed in PTFE frame.

Electrical specifications:

Watts- 3Kwatt,
volts- 230 vac,
Amps- 13.06Amps,
Ohms: 17.61ohm,
Thermocouple type: RTD

Original manufacturer

HEATEFLEX CORPORATION
405 E Santa Clara St
Arcadia, CA 91006-7227
Fax: 626-599-9567
info@heateflex.com
www.heateflex.com

ii. Chemical Circulation Pump:

Model: T025-NHT-PTTT-000

Original manufacturer:
All-Flo Pump Co.
7750 Tyler Blvd. Mentor, Ohio
44060 USA
Phone +440.354.1700
Fax: +440.354.9466
sales@all-flo.com
www.all-flo.com

Local Provider:
Nano Clean Contamination Control Solutions
#126, 9th cross, 2nd Main, 1st Block, BEL Layout,
Vidyaranyapura, Bengaluru: 560 097
P : +91-(0)-80-4890 5897
F : +91-(0)-80-2364 0428
W : www.nanocleanindia.com
E: sales@nanocleanindia.com

TRAINING PROCEDURE:

1) File a request in the NCPRE slot booking module and get it approved from guide.
2) Contact the System Owners for training sessions.
3) Minimum three training sessions and three hands-on sessions are mandatory for authorization on the tool.

CHEMICAL SAFETY MANUAL

Please go through the generic chemical safety manuals before starting any wet chemistry process. Click on following link

For IITB

https://www.iitb.ac.in/safety/sites/www.safety.iitb.ac.in/files/Chemical%20Safety_0.pdf

For IITB-Nanofabrication facility

http://iitbnf.iitb.ac.in/iitbnf/images/headers/manuals/iitbnf_safety_manual.pdf

General rules to be followed in chemical area

- Users MUST have received the wet bench training by an authorized user or system owner.
- Users MUST go through the material safety data sheet (MSDS) for each chemical that is being used prior to work and appropriate precautions must be taken.
- Before mixing any chemical, user must be aware of the chemical reactions that may occur and the damages it may cause.
- Users are NOT ALLOWED to bring new chemicals inside the cleanroom without approval of concerned faculty in-charge of the lab.
- ALWAYS wear the Personal Protective Equipment (PPE) at all the times while working in chemical area: chemical apron, then full-face mask and lastly the tri-polymer chemical resistant gloves.
- Identify the chemical carefully when taking the bottle(s) from the cabinet.

- User MUST label any lab ware with the name of the chemical/process. If you leave the wet bench unattended, indicate name of the chemical along with the expected time of disposal (as applicable), your name contact number, date of process, expected time of disposal etc. Use chemical form for the same.
- DO NOT leave solutions unattended when there are chances for strong reactions to occur (hot RCA, Piranha mixtures, etc.).
- User MUST properly rinse the wafers/samples in DI water in between when using several incompatible chemicals during a process (e.g., solvents and acids, RCA-1 and hydro-fluoric acid (HF)).
- In order to avoid cross-contamination all lab wares will be kept as a set-in designated boxes for the process (do not use lab ware from one bench at another bench).

SAFETY PRECAUTIONS

➢ Safety Symbols:

Warning:

NEVER touch clothes, goggles, face shield or any surface outside the acid/base wet benches while wearing the chemical gloves because abrasive chemicals might be left on the gloves and cause injuries.
Remove the complete PPE if you leave the wet bench area and use other device like computer, microscope, etc.
Any hot chemical or mixture (e.g. KOH, Texturing Solution, RCA 2) must be cooled down (< 40°C) before disposing of it.

➢ Chemical storage

Use separate cabinet to store the fresh chemicals to avoid cross reactions.

➢ Chemical spill kit-

Spill kits contain neutralizers for all chemicals (acids, bases and organics) and PH indicators with other required accessories as shown in fig.

1. While handling the chemicals in case there is a chemical spill on the wet bench or on the floor, NCPRE fabrication staff needs to be contacted.
2. If any fabrication lab user found the spill of some unknown liquid on the wet bench or on the floor, please inform to the available NCPRE fabrication staff and leave the area.
Note: The authorized NCPRE staff member will use spill kit to take care of the chemical spill.

CLEANING METHOD

- Wet bench should be clean with DI water and lint-free cloth.
- The lab wares should be clean multiple times with DI water and should wipe with lint free cloth or dry with PN2 after completing the process.
- All baths should be clean with DI water only.

THEORY

SAW DAMAGE REMOVAL

The sawing process to cut ingots into wafers induces surface damage and introduces residual contaminants on the wafer that will lower the solar cell performance.

Figure1. Shows a monocrystalline silicon wafer which was sawn using diamond wire sawing. The sawing (horizontal) marks from the process are clearly visible on the wafer.

Figure 1.

Therefore, it is necessary to etch 10 µm (slurry-based sawing) or 5 µm (diamond wire sawing) of each side of the wafer before further solar cell processing. The wet alkaline etch process is commonly used for this purpose. The most common solutions used are as sodium hydroxide (NaOH), potassium hydroxide (KOH), or Tetramethyl Ammonium Hydroxide (TMAH) diluted in de-ionised water as the etch solution. The reaction process is essentially similar for all solutions, where OH^- and water (H_2O) plays a key role in the reaction:

$$Si + 2OH^- + 2H_2O \rightarrow SiO_2(OH)_2^{2-} + 2H_2$$

The etch rate depends on the $[OH^-]$ and $[H_2O]$ concentrations. The etch rate increases with increasing [OH-] concentration until some maximum point is reached. With further [OH-] increases, the etch rate decreases due to decreasing water content (H_2O). In Figure 2 we see that the etch rate after a point decreases with concentration, which is due to the lack of water in the process. The etch rate of KOH may vary from as low as 1 Å/s in dilute KOH at room temperature to as high as 2000 Å/s in 40 % KOH at high temperatures. The etching is anisotropic due to different energy levels of the back-bond states with the different crystal planes. However, depending on the etching condition, the level of anisotropy of the process can change. For KOH the anisotropy ratio is 30:1 (100) : (111) at 100 °C (high etch rates) and >>100:1 at room temperature (low etch rates). In the saw damage etch process, the solution is 30-40 % KOH and the temperature is held at 70-80 °C. Such conditions result in high etch rates (2-4 µm/min) decreasing the anisotropy of the process, i.e., the saw damage etching is quasi-isotropic.

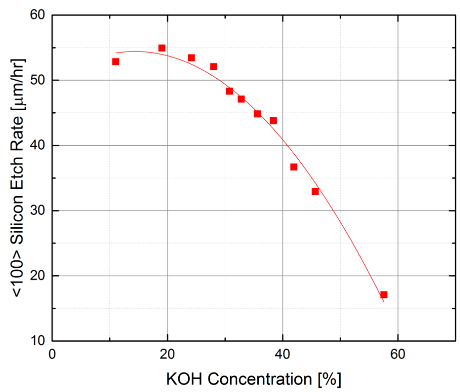


Figure 2: Etch rate of the [100] silicon crystal plane as a function of KOH

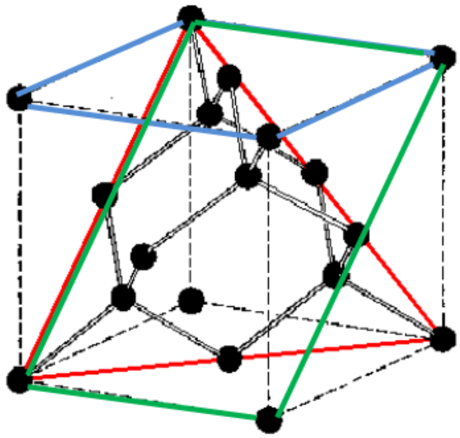
concentration at 72 °C (graph adapted from [1]).

It is important to control the etch rate of the process. Over-etching leads to thinning of wafers that could lower production yield due to breakage. Under-etching can lead to shunting and degradation of the minority carrier lifetimes because of residual surface micro-damage.

ALKALINE TEXTURING

Monocrystalline silicon wafers with [100] orientation are the most common type of monocrystalline wafer in the industry because it can be easily textured using an alkaline etchant, for example KOH. Silicon crystallizes in a diamond cubic lattice (two inter-penetrating face-centered cubic lattices).
Figure 1. Shows the diamond cubic lattice of a silicon crystal and the representation of the different planes as indicated by colored lines.

Blue (100), **Green (110)** and **Red (111)**



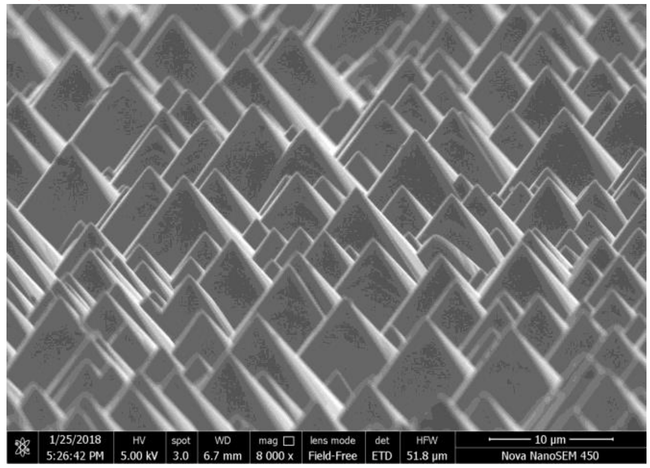


Figure: Pyramid formation after Texturization

Alkaline etchants etch [100] silicon surfaces much quicker than [111] silicon surfaces, which is the basis for the anisotropic etching process used to make pyramid texture. The main difference between saw damage etching and texturing is the etch rate. To increase the anisotropy of the process the etch rate needs to be low, i.e. 2 µm/min or lower. To accomplish lower etch rates, either the process temperature can be lowered and/or the etchant concentration decreased. For instance, a typical texturing recipe which uses a KOH concentration of 1-2% (in comparison to a 30-40 % concentration in saw damage removal) at 70-80°C. The result is a surface populated with randomized square-based pyramids where the sides are formed by [111] planes and the base is the [100] plane. This is depicted in Fig 2. In reality, the etched pyramids are not perfect square-based tetrahedrons with a base angle, a, of 54.74°. For most industrial texturing processes is between 49° and 53°. This is because the tip of the pyramid is etched for the longest duration.

The texturing solution also includes isopropanol which acts as a surfactant which enhances surface wetting and ensures that H_2 gas (released by the etching) does not stick to the surface. If isopropanol is not used, round “hillocks” can form due to H_2 bubbles blocking etch rate at the surface. Isopropyl reduces surface tension and allows H_2 bubbles to release from the surface more easily.

There are many factors that contribute to the quality of texturing, as follows:

- The texturing result depends on the initial surface.

- The process is sensitive to the presence of residual silicates from saw damage etching.

➢ The balance between pyramid nucleation and pyramid destruction.

- Over etching can lead to the destruction of pyramids.

➢ The evaporation of Isopropanol occurs after the bath temperature reaches 90 °C.

- Isopropanol has a wetting function – stops H_2 bubbles from sticking to the surface.
- Ventilation is important, but can affect isopropanol evaporation rate
- Typical process durations are 15-20 mins, therefore the evaporation rate must be monitored.

➢ To get very good texture surface Chemical circulation is important as it keeps bath components well mixed. Good texturing is important because the surface texture is directly related to the ability of the solar cell to harvest light and to generate current.

➢ Texturing the surfaces improves the cell current via three distinct mechanisms as follows:

1. Reflection of light rays from one angled surface onto another improves the probability of absorption.
2. Photons refracted into the silicon will propagate at an angle, increasing their effective path length within the cell, which in turn increases the chance of electron-hole pair generation.
3. Long-wavelength photons reflected from the rear surface encounter an angled silicon surface, improving the chance of being internally reflected (light trapping)

A good texturing should lead to lower reflectivity for the whole visible wavelength range.

WET CHEMICAL CLEANING

Wet chemical cleaning is essential in solar cell fabrication to ensure silicon quality is maintained and to prevent contamination of equipment, which could contaminate following samples. Surface contamination arising from “dirty wafers” can have disastrous effects on the bulk minority carrier lifetime of silicon wafer solar cells, especially if impurities are allowed to diffuse into the wafers through high-temperature thermal processes such as diffusion. These impurities can also out-diffuse and contaminate production line components such as diffusion furnaces, firing furnaces and plasma enhanced chemical vapor deposition (PECVD) tools. Silicon wafers are cleaned through a series of wet chemical steps. To clean the wafer surface we follow Ionic Cleaning process as recommended.

RCA II: Ionic Clean

This treatment is used to remove metallic contaminants. The solution consists of a mixture of aqueous hydrochloric acid (HCl), H_2O_2 and DI water in a 1:1:5 (2.5L: 0.250L: 12.5L) ratio heated to ~80 °. Wafers are immersed in the solution for 10 minutes. The resulting wafers have a thin, chemically grown oxide, which according to the next processing steps, is either removed in a HF acid dip, or left on the surface to act as a passivating oxide.

HF – oxide removal

A hydrofluoric (HF) acid dip is typically performed to remove any native oxides that may have grown on the silicon surface. Wafers are immersed in an aqueous HF solution until a hydrophobic surface condition is observed. This process is usually performed with high purity materials as the bare silicon surface is highly reactive and may easily become re-contaminated.

STANARD OPERATIONAL PROCEDURE

1. Before entering and starting the process in the chemistry room, wear complete Personal protective Equipment (hair net, face mask, apron, safety glasses and shoes.) as shown in fig.
2. Before planning any process, always make sure that all the required chemicals are available in stock.
3. Switch On the mains of the fume hood (back side).
4. Switch On the wet bench blower.
5. Turn ON the DI water and fill all rinse baths. Each bath is provided with separate DI connection to avoid cross contamination.
6. Before starting the process, clean all the process baths with DI water and drain it through drainage facility provided at the back side of the wet bench. As each bath is provided with separate drain tap as shown in fig.

7. After cleaning baths, always make sure that the all the drain taps are closed before pouring the chemicals into the respective baths.
8. Make sure the level sensor and temperature sensor are completely dip into the solution. Failing to this, your process will not start. We have to connect the level sensors separately, as shown in fig.

Fig: level sensors

Fig: low liquid level

9. Once ready with filled baths, Turn ON the MCBs for heating control units (front side).

10. Now set the required temperature on the Digital Temperature controller of each bath as follows:

- Press 'SET' once.
- Press UP arrow ↑ and down arrow ↓ to set the required temperature. The maximum temperature is 95°C.
- Press 'SET' to start the HEATING UP program.
- Turn 'Heater Power' switch ON.
- SV: indicates set value & PV: indicates Present temperature value of the solution.

11. In case of texturisation bath, turn on the CDA based chemical circulation pump for proper mixing of the solution. Use assembled CDA flow meter for control of pump speed. The pump should be on throughout the texturisation process for proper circulation of chemical.
12. In case of SDR bath, turn on the PN2 bubbling for proper mixing of the solution. Turn OFF the PN2 once temperature is reached.
13. Now set up the wafer jigs, and make necessary measurements before starting the process if required, till the temperature of the solution reaches to set value.
14. The standard process flow is as follows:
    SDR> DI Rinse> **Texturisation**> DI Rinse> **HCl clean**> DI Rinse> 2% HF dip/Oxide removal.
15. After completion of cleaning step/ oxide removal step, wafer cassettes are dried using a spin rinse dryer or prior to the next process.
16. Once done with process, let the solution to cool down to room temperature, keeping the exhaust blower ON. Inform this to NCPRE facility person.
17. Once the process is over set the required shut down temperature on the Digital Temperature controller as follows:
    - Press 'SET' once
    - Use DOWN arrow ↓ to reduce the oven temperature to 20^{o}C.
    - Press 'SET' to start ramp down program.

- Turn 'HEATER POWER' switch OFF.

18. Rinse the chemical resistant gloves with DI water and dry them with a wipe. Remove & place in the appropriate location.
19. Clean up the wet bench after use. Dispose of all wipes used and do not leave any wipes on the deck of the bench.
20. Used chemicals to be transferred into their **respective** 'used chemical' bottles carefully. Label them appropriately.
21. After disposal of used chemical, clean all the process baths with DI water and drain it through drainage facility provided at the back side of the wet bench.
22. Circulate the fresh water through Chemical Circulation pump of Texturisation bath to avoid salt formation/ clogging in the pump line.
23. After completing the slot, SWITCH OFF the blower.
24. Make a Log entry of the usage.
25. Hang the aprons at their proper place.

Note:

1. As the NCPRE Texture clean bench is dedicated to NCPRE Baseline process, we have not discussed any process details here. The process details such as chemical composition, process time etc. will be available in process details book in NCPRE fab lab.