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Synthesis of nano materials by sputtering

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Abstract: *Nanoscience and nanotechnology primarily deal with the synthesis, characterization, exploration, and exploitation of nanostructures materials. These materials are characterized by at least one dimension in the nanometer (1nm = 10−9 m) range. In this research project nano materials are synthesized or deposited by sputtering process. Prior to this sputtering process, the desired specimen and its pattern is prepared with one of the mask less lithographic techniques such as electron beam lithography (EBL). In this process, EBL machine is used with 220 KV of power and it is used to write the pattern with raster scan method. After co-deposition of Al_2O_3 and SiO_2 with the help of sputtering then finally characterization has taken place. In this characterization, Scanning electron microscope (SEM) images are taken and then finally atomic force microscope (AFM) images are taken in order to know the deflection error, adhesiveness, and DMT modulus*

Keywords: *About five key words in alphabetical order, separated by comma.*

I. INTRODUCTION

A Nanomaterial is a material where some controllable relevant dimension is of the order of 100nm or less. The simple presence of nanoscale structure alone is not sufficient to define a nanomaterial, since most if not all materials have structure in this range. The ability to control the structure at this scale is essential. One could argue, in this sense, that many of the classical alloys and structural materials that contained nanoscale components by design could be called nanomaterials. In modern usage, nanomaterials are newly developed materials where the nanoscale structure that is being controlled has a dominant effect on the desired behavior of the material or device. There are three different classes of nanomaterials: discrete nanomaterials, nanoscale device materials, and bulk nanomaterials. In a broad sense, the approaches used to make materials can be put into two categories: top-down approaches, in which one begins with a bulk material that is then processed to make a nanomaterial, and bottom-up approaches, in which the nanomaterial is built up from finer scales (in the limit, building the nanomaterial up one atom at a time).

It is evident that bottom-up approaches require control of processes at very fine scales, but this is not as difficult as it sounds, since chemical reactions essentially occur molecule by molecule. Depend upon the application one has to select the appropriate approach for the production of nano materials. The electron beam lithography is the one of the best top down approach for the synthesis of nano materials. EBL is well known for its maskless lithography for a better control over line width i.e less than 5 nm. Only one disadvantage of using this technique is it takes too much time for writing the pattern onto the resist material. After transferring the pattern onto the PMMA resist, the desired material can be synthesized with the help of sputtering (electron beam evaporation or thermal evaporation). Sputtering means ejecting material from a target and depositing it on a substrate such as a silicon wafer. The target is the source material.

II. Experimental Procedure

2.1 Materials used

Alumina (Al_2O_3) is very hard material and its hardness is exceeded only by diamond and a few synthetic substances such as carborundum, and silicon carbide. This property of alumina lends itself for use as an abrasive material. Another useful property of the material is its high melting point, i.e., above 2000°C (3632°F), which makes it useful as a refractory and as linings of special furnaces.

Property*	Value
General	
Chemical Formula	Al_2O_3
Mechanical	
Density	3.88 gm/cc
Hardness	2000 Knoop
Tensile Strength	35 kpsi
Modulus of Elasticity	48 - 54 x 10^6 psi
Flexural Strength	57 kpsi
Compressive Strength	368 kpsi
Poisson's Ratio	0.23 - 0.26
Fracture Toughness	4.3 MPa $m^{½}$
Electrical	
Dielectric Strength	210 - 220 ac V/mil
Dielectric Constant	9.7 (@ 1 MHz)
Volume Resistivity	> 10^{14} ohm-cm
Thermal	
Coefficient of Thermal Expansion	5.1 - 7.2 x 10^{-6}/°C
Thermal Conductivity	30.3 - 35.0 W/mK
Specific Heat	0.20 cal/g °C
Maximum Working Temperature	1750 °C
Shock Resistance	200 °C Diff.

The pure form of **Silica (SiO_2)** is Quartz and the impure form is sand. Mainly it is used with aluminium in car manufacturing industries. Used in waterproofing treatments, moulding compounds and mould-release agents, mechanical seals, high temperature greases and waxes, caulking compounds and even in applications as diverse as breast implants and explosives.

Material	Quartz	Fused silica
Density (g/cm^3)	2.65	2.2
Thermal conductivity (Wm^{-1} K)	1.3	1.4
Thermal expansion coeff. (10^{-6} K^{-1})	12.3	0.4
Tensile strength (MPa)	55	110
Compressive strength (MPa)	2070	690-1380
Fracture toughness (MPa)	-	0.79
Melting point (□ C)	1830	1830
Modulus of elasticity (GPa)	70	73
Thermal shock resistance	Excellent	Excellent

2.2 Process flow

The first step in the first stage is sample preparation where SiN wafer is cleaned with Piranha solution. After cleaning of this substrate it is dipped into dil. HF. Then dehydration process will takes place with a prebake temperature of 150 ^{0}C for about 5 minutes. Then Spin the PMMA resist at 6000 rpm in order to get 200 nm thickness for 60 seconds.

Figure 1: process Flow diagram

The spin off stage takes place for approximately 10 seconds after spin up. After obtaining 200 nm thickness of PMMA resist then it is used for soft bake at 95 ^{0}C for 45 seconds.

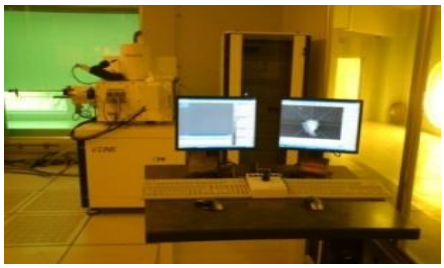

Figure 2: Raith EBL at IISc, Bangalore

In this experiment Raith EBL equipment (shown in above figure) is used to produce the desired pattern and the wafer along with PMMA deposition is kept in the sample loading unit of EBL. Raith uses NPGS (Nano pattern Generation System) software to design the layout of the pattern and the generated pattern is as shown in figure 3. Each cube consists of 30 nm x 30 nm x 30 nm.

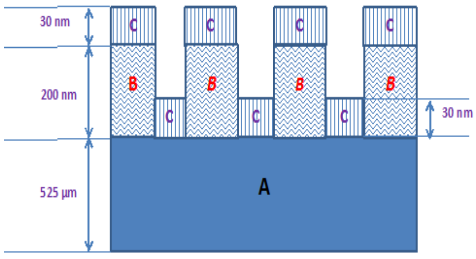

Fig 3: Pattern of an EBL (side view)

In this process, it is proposed to synthesis 50nm cubes of 7*7 matrix. The diameter of the wafer is 4 inch and whereas the thickness is 525 µm. The film of resist are exposed to line dose of 1-2 nC/cm and area dose of 200 mC/cm2 at 30 kV of energy beam depending on the pattern. Then finally allow the electrons from the electron gun from electron source towards PMMA resist in order to deposit the desired pattern which was designed. It took 7 hours of time to deposit the desired pattern. Then finally lift-off process is conducted on the specimen in order to remove the unwanted PMMA resist and to get the final desired pattern which will be ready for next process i.e, sputtering process. In this process the material to be deposited is taken for co-deposition and is kept in sample holder. Then allow high voltage current in order to evaporate from the sample holder and allow it to deposit on to the silicon nitride wafer i.e on to the holes as well as on the PMMA resist. Then allow it for solidification. The lift-off process is used in order to chip-off or remove the unwanted PMMA resist as well as to remove the unwanted deposited material with the help of sputtering process. The final specimen will look like as shown in the figure 4. This specimen is further is used for characterization for SEM and AFM.

III. Results And Discussions

3.1 SEM Analysis

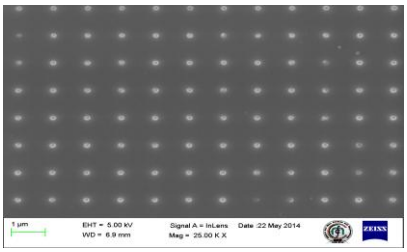

Fig 4: 1 µm level at a magnification of 25 KX

The above figure shows the SEM image of nano sillimanite cubes. It is seen at 1 µm level at a magnification of 25 KX. From this it is clearly visible that the presence of nano sillimanite is available. The white dots indicate the co-deposition of alumina and silica materials. Whereas the remaining dark area indicates the silicon nitride wafer.

3.2 AFM Analysis

3.2.1 3D image analysis

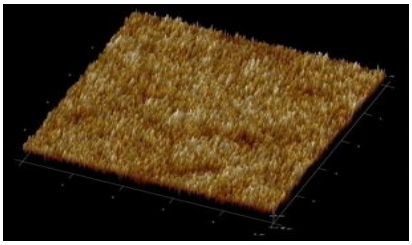

Figure 5: 3D image of nano sillimanite

The figure 7 shows the 3D image of sillimanite cube. The golden yellowish color shows the presence of silicon dioxide and whereas the white color indicates the presence of aluminium dioxide. The valleys present in the image indicate the presence of any voids.

3.2.2 Adhesiveness

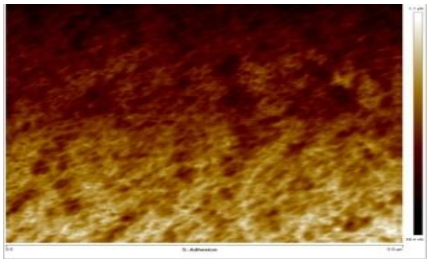

Fig 6: Adhesive property of nano sillimanite cube

The above figure shows the adhesive property and from the figure it is very clear that this image is taken from 0 – 5 µm range. There are various colors on the plot. The yellowish color indicates that the material have more adhesive towards the tip of the cantilever beam. That means there is a frictional force behind this phenomenon. Whereas the dark brown color indicates that there is a less adhesiveness when we move from center of the material towards the edge of the material. And this value again very less when we move towards the edges of the specimen.

3.2.3 Deflection error

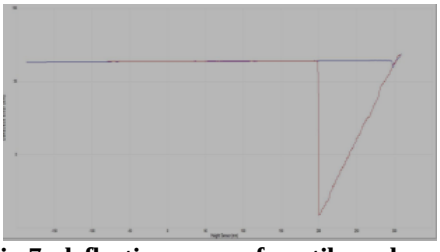

Fig 7: deflection error of cantilever beam

The above figure is plotted between deflection error (nm) and height sensor (nm). When the tip of the cantilever beam is far away from the surface of the material then the deflection error is more and is as shown in figure. When the cantilever beam is started to bring near to the surface of the material then the deflection error is gradually reducing. This zero deflection can also be considered as adhesiveness of the material.

3.2.5 DMT modulus

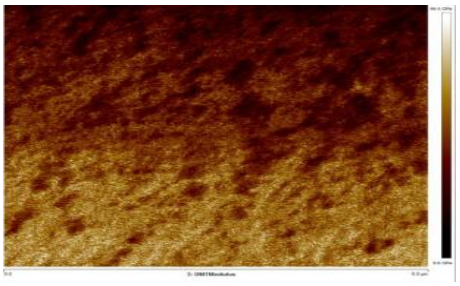

Fig 8: DMT modulus of the scanned area

The above figure shows the **DMT** (Derjaguin, Muller and Toporov) modulus values across the scanned area. Usually the tip of the cantilever beam is of spherical shape. The radius of curvature of spherical tip will be in contact with the surface of the material. The surface energy or adhesive force will have impact on outer side of the radius of the tip. This adhesive force or DMT modulus (dark brown color) at the contact area will be very less because of the presence of porosity at contact area. The yellowish color shows that DMT modulus value is more and this is because of more adhesive force present over there. And it is average at the rest of the scanned area.

IV. CONCLUSION

1) It seen from the SEM images that the distribution of the nano sillimanite tiles of 50 nm is uniform throughout the scanned surface area.
2) It is seen from the image that 50nm cubes are clearly visible.
3) The white spotted areas indicate that the presence of nano sillimanite tiles whereas the dark area indicate silicon nitride wafer.
4) From the AFM images it is seen almost a flat surface except with some valleys.
5) It has very good adhesiveness property with the tip of the cantilever beam of the atomic force microscope and this is because of the surface roughness present between the surface of the material and the tip of the cantilever beam.
6) The combination and presence of two ceramic materials will give almost zero surface roughness i.e 1.21 nm which is almost negligible. The maximum surface roughness at ever be peaks and valleys is 12.4 n

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