

REPORT:

INTRODUCTION:

During the whole Summer Student program I worked as a member of Sasa Bajt group. This group deals with coating of multilayer for, strong X ray sources. Multilayer is high reflective device made from several thin layers of different materials, coated on polished wafer. It fulfill the Braggs law for X ray radiation. Power of new sources of X-ray make impossible to use other kind of optic, multilayer's (like for example SiC/W), are very good solution for that problems. Polished silicon wafers, coated with different materials can be used as a mirrors, zone plates, pulse compressors etc... The main equation which describe behavior of light in those sources is of course Bragg equation: $2d \sin \theta = n\lambda$, where d is distance between two layers in a crystal (period in the multilayer), " n " order of the next maximum, θ angle of incidence, and λ wave length. This simple equation plus a little bit of trigonometry seems to be enough, but there of course exist a "dynamical theory" of diffraction based on the quantum electro dynamic (light pulse is described by a Hamiltonian and one can observe how it is evolutes during the time- that is after going trough the different optical devices). Samples are made inside two systems MAG-1 and MAG-2, those are vacuum chambers with 4 electro magnates. On the surface of the magnets there is a thin film of a certain material (for example W), pure silicon wafer (2 or 4 inch) is moved through the created plasma with certain velocity, and in that way wanted material is sprayed on the surface of the wafer. Two factor which were interesting for me during the job were: Γ factor which is simply the proportion of the higher Z number material to the whole sample, and period that is thickness of layers of different material.

TASKS:

During my work I had to solve several problems using AFM measurement and X-ray high angle diffraction:

- How roughness is changing as a function of a period (SiC/W multilayer- AFM measurement)
- How roughness is changing as a function of a gamma (SiC/W multilayer- AFM measurement), and if there is a change of the crystallization in the sample around the gamma 0,5(X-ray diffraction).
- Determine the difference in roughness of an ion beam polish and not polish sample (Mo/Sc/ B_4C /Cr -AFM measurement) :

AFM:

The acronym AFM stands for an: Atomic Force Microscope, this is a type of: "scanning probe microscope", having a small (usually) silicon tip. During the scan tip approaches the surface of the sample and is bended by atomic force. Knowing the Hook Law (it allows us to say what is a value of force which bends the tip), one can evaluate the shape of the surface of the sample. We can use AFM in several modes: contact mode in which tip is touching the surface of the sample, non contact mode (Van der Vaals force is used) and tapping mode in which the tip is oscillating near the surface (according to the "Wikipedia" the typical oscillation range is between 10 and 200 nm), which protects a surface. Atomic Force Microscope allows us to reach the resolution of an angstroms. All the data is proceeded on a "standard" PC, on NanoScope® software. However it is enough for my measurement, there exist a open source software: "Gwyddion", which can work on raw data from NanoScope®. That program allows us to do some other operation on pictures (profile, PSD, roughness, background extraction etc...).

Surface characterization:

A surface of the sample can be characterized by several factors: like roughness or PSD. Roughness is a root square over a sum of height of a singular point divided by numbers of the

points. $Rq = \sqrt{\frac{\sum_i^N Z_i^2}{N}}$ (in fact it is the Root Square Mean Roughness), in that way we can obtain one factor which can characterize a surface. Roughness of the sample is very important factor. Sample has to be very smooth because of the reflectivity and Bragg's law (or Laue law in the different geometry). However, one may observe that this value is not very useful to "map" the surface. PSD is an acronym for "Power Spectral Density" and is usually

presented in: $\frac{W}{Hz} = \frac{Power}{Frequency}$. Power is given as a function of strength of a signal:

$P(t) = f^2(t)$. In our case: $f(t) = Height(x, y)$. The Fourier transformation of a signal is

given by: $F_t(\omega) = \frac{1}{\sqrt{T}} * \int_0^T f(t) \exp(-i\omega t) dt$. The factor R is given by:

$R(\tau) = (f(t) * f(t + \tau))$, and $PSD(f) = \int_{-\infty}^{\infty} R(\tau) * e^{-i\omega\tau} d\tau$. There is a correlation between the

roughness and the PSD that is given by: $Rq^2 = 2\pi \int_{f1}^{f2} PSD(f) df$. In our case it is possible to check the value of Roughness that way, but it is unfortunately very hard to achieve value of PSD, from Roughness due to lack of data.

Si, SiC, W

It is good to mention those measurement at first (although there were made in meantime between the set of different periods and different gammas) because results can give a briefly look into the behavior of different materials in multilayer:

NAME	Rq 1 ,nm	Rq 10 ,nm	MATERIAL
W-120806B	0,181	0,185	Sic
Si	0,218	0,183	Si
W-120806C	0,399	0,326	W

SiC wafer Has the lowest value of roughness, but the difference in roughness between Si wafer and SiC is much too low to deduce something from that (third place after the point cannot be taken into account the error is too big). In my opinion value of Rq is similar in both case. However PSD plot (**appendix 1**) shows us that SiC has much lower, higher spatial frequency roughness. The roughest one is W sample because of crystal structure which is building up in this material. Also PSD of W show us that it is true (**appendix 1**). Next measurement was comparison of two silicon wafers, one made earlier and next made later on, the aim of it was to see if the quality of the samples is equal during the time. PSD Plots (**appendix 2**) and Rq values:

NAME	Rq, 1 nm	Rq, 10 nm
Si old	0,218	0,183
Si new	0,206	0,166

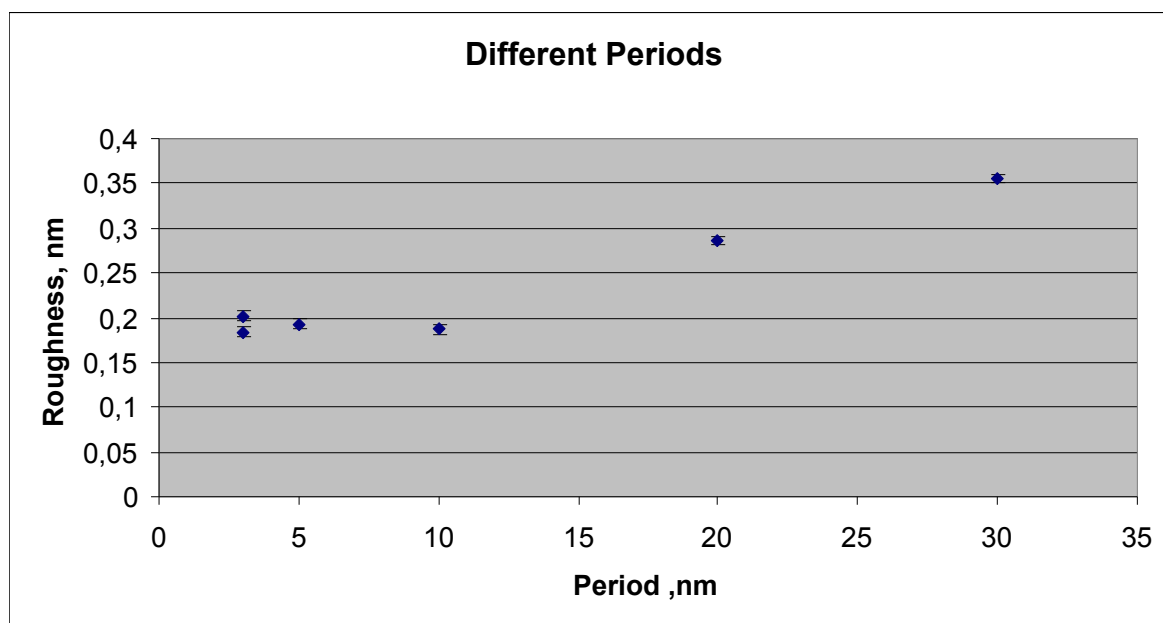
Shows us that there is no big difference in value of sample with the time.

Different Periods:

The first set consisted of 6 SiC/W multilayer, made with different periods of the layer - between 3 and 30 nm (two first samples have same period but were made with different pressure 1.5 and 2 mTor):

NAME	GAMMA	PERIOD, nm	PRESSURE, mTor	Rq 1, nm	Rq 10,nm
W-120801A	0,5	3	1,5	0,184	0,169
W-120801B	0,5	3	2	0,202	0,199
W-120802A	0,5	5	1,5	0,193	0,174
W-120802C	0,5	10	1,5	0,187	0,172
W-120803A	0,5	20	1,5	0,286	0,274
W-120803B	0,5	30	1,5	0,355	0,319

Pictures were taken in the two range $1\mu m$ and $10\mu m$. Unfortunately due to my mistake and strange behavior of software the $10\mu m$ data are not always reliable (see appendix 3), data on some of measurements are not full, that is why it is better to look at a $1\mu m$ range measurement results. The roughness of the sample is increasing with the increasing of the period which is quite normal behavior as within the bigger period crystallization process in samples are more developed (crystallization occurs mainly in W). This tendency is also proofed by PSD plots, the only strange thing is quite high PSD value for 20 nm period sample, for lower frequency (appendix4-5).



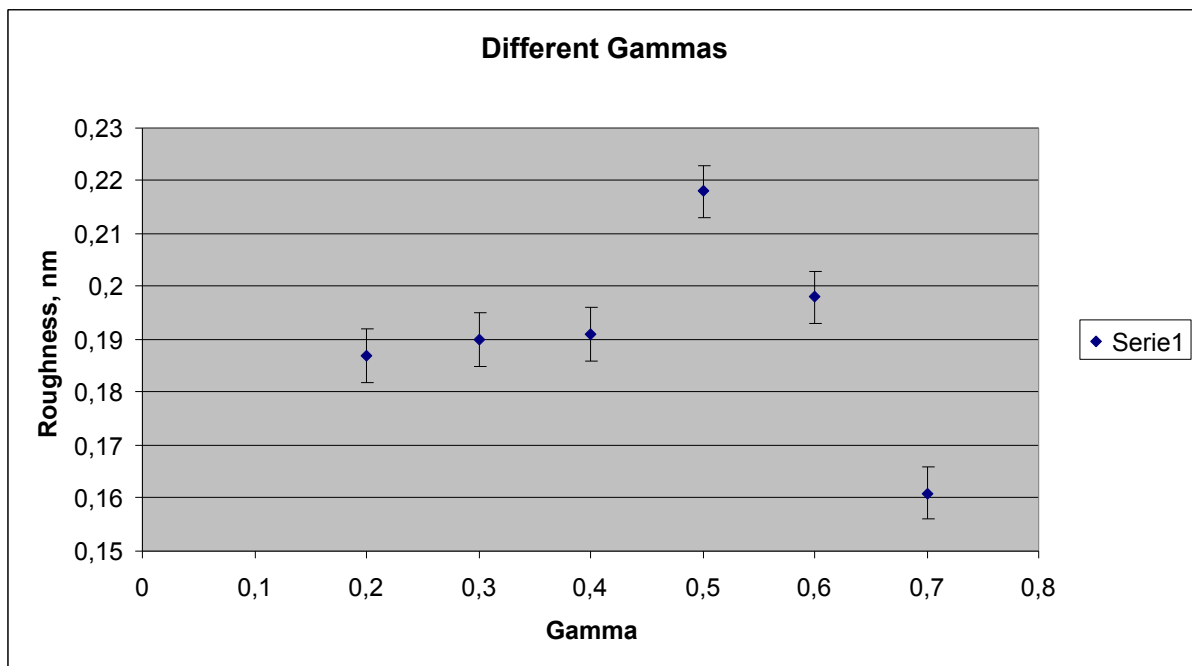
Plot is almost flat up to 10 nm and then the grows (lineary?). Probably before the 10 nm there is no place for building crystal in W and Roughness is close to its lowest possible value.

Different Gamma:

Second set of measurement consisted of samples made of SiC/W in the same conditions, but having a different gammas factor. The reason of such a measurement was a strange behavior of the stress in samples around sample with $\Gamma = 0.5$. According to the research stress should change linearly with different gammas. Thanks to that it comes through the optimal value, at some point, but the stress in SiC/W samples behave differently because, it grows up and then decrease (similarly to parabola). The question is if this strange behavior has any connection with, a changing of the crystallization in samples. It can be evaluated for example from the changing of the roughness of the sample (crystal should make samples rougher).

NAME	GAMMA	PERIOD	PRESSURE	Rq 1,nm	Rq 10, nm
W-120730A	0,2	3	1	0,187	0,162
W-120730B	0,3	3	1	0,19	0,149
W-120727C	0,4	3	1	0,191	0,118
W-120726B	0,5	3	1	0,201/0,139/0,218/	0,160/0,119/0,159
W-120727B	0,6	3	1	0,198	0,168
W-120731B	0,7	3	1	0,161	0,153

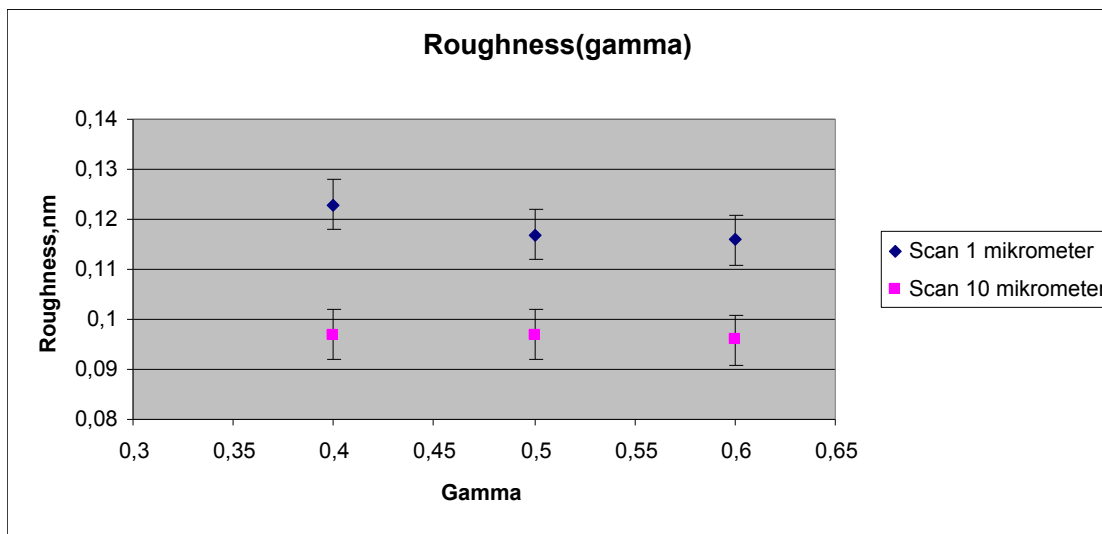
Sample with gamma 0.5 had to be measured three times because first two measurement were not reliable enough (due to unwanted vibration-construction outside the building). The plot of the roughness (again only the results from 1 mikrometer scan were taken into account as more reliable-the same case as with different periods):



Roughness is changing with 0,5 gamma factor. Difference between gamma 0,4 and gamma 0,5 is $\Delta Rq = 0,03nm$, and between 0,5 and 0,6 is: $\Delta Rq = 0,02nm$. Difference itself is not big, the tendency to increase, up to the gamma 0,5 and then decrease is really something unexpected. From the plot we can see that decrease of the roughness is much faster than the increase but it does not necessarily have to be true as the value for sample with $\Gamma = 0,7$ is a little bit too low (we expect that value should not be smaller than roughness for SiC wafer 0,18). Moreover from PSD plot (**appendix 6**) we can observe too that differences in roughness values of samples are not very big, so probably there is no change in the crystallization.

During the measurement we noticed a large usage of AFM tips. SiC/W is very “hard ” material and, due to that 1 tip lasts only for (average) 3 scans. To save the tips we decided to switch to tapping mode. It indeed helped us to save tips, moreover we obtained another set of measurement for comparison with Nanoscope ® mode. In Tpping mode set of measurement consisted of 3 samples with gamma factor around 0,5,(due to strange behavior of the stress the 3 most important sample are those with: $\Gamma = 0,4 - 0,6$.) and bare silicon wafer (for comparison):

NAME	GAMMA	Rq 1, nm	Rq 10, nm
W-120727C	0,4	0,123	0,097
W-120726B	0,5	0,117	0,097
W-120727B	0,6	0,116	0,096
Silikon Wafer_new		0,18	0,123



It can be observed that value of roughness is lower that this obtained with Nanoscope® mode, and sample with $\Gamma = 0,5$ is not the roughest one. PSD of those set of samples shows us the same thing (no extreme change in the roughness value-that is in crystal structure too- occurs) (appendix 7).

X-RAY MEASUREMENT:

To evaluate the period of the layers in samples, laboratory is provided with the X-ray source. This is standard copper source producing, 8 keV, energy wave. Data are collected by a CCD matrices, and evaluated by ” X-pert viewer” software. The material in which we expect the crystallization is Tungsten, according to the literature we should see a Bragg peak around angle of 40° (and at others angles too but this one was chosen because is the strongest one), coming from that material. Due to that we do not have to analyze whole spectrum of Bragg peaks, but only the high angle. That is why usually scans were made between 20 up to the 50 degrees. Scan of samples, with $\Gamma = 0,4$, was made with 60 seconds steps, $\Gamma = 0,5$ with 100 second steps and $\Gamma = 0,6$, with 50 steps.

The length of the steps was motivated by the limited time for measurement (X-ray source was during also used for others task in laboratory). As we expected one can observe Tungsten

peak on spectrum. To see the change of the crystallization in the samples (the size of a crystals) we use the Scherrer formula:

$\tau = \frac{K * \lambda}{\beta * \cos \theta}$, where, “K” is a shape factor (between 0-1 I could not find its value for Tungsten, that is why is taken as a parameter) dependent of the material, λ is wavelength of X-ray wave, β is width of the is the line broadening at half the maximum intensity (FWHM) in radians, θ is angle on which peak was observed.

GAMMA	CRYSTALLIZATION, nm
0,4	$2,083 * K$
0,5	$1,835 * K$
0,6	$2,035 * K$

As we can see with gamma 0,5 there is no sudden increase in the crystallization, results are supported by roughness measurements. Crystal of the sample with $\Gamma = 0,4$ are even slightly bigger that crystal of the sample with $\Gamma = 0.5$ (It maybe due to error of measurement). If we divide value of the crystallization of sample with $\Gamma = 0,4$ by crystal size of $\Gamma = 0,5$ we

obtain: $\frac{\tau(\Gamma = 0,4)}{\tau(\Gamma = 0,5)} = 1,11$, or $\frac{\tau(\Gamma = 0.5)}{\tau(\Gamma = 0.6)} = 0,9$.

Polished samples vs non polished samples (more of AFM):

Third set of samples consisted, as I mentioned before of ion-beam polished and non polished samples. The question was which set of samples has biggest roughness. During measurement I found out that measurement of ion beam polished samples changes value of roughness after measurement with range $1 \times 1 \mu m$, next PSD plot (with a range of $10 \times 10 \mu m$), do not mach to the first one (they should meet at the same spatial frequency, for both scans) (**appendix 8**). First I assumed that it is due to different mode of setting Z-range (Z range it is maximum distance between highest and lowest point in the image- for moving tip), but after changing this mode to the standard one use during AFM measurement I could not see any difference. The best matching of PSD plots was achieved with: M-120612A1 sample (**appendix 9**).

The most important comparison is that of samples with name: M-12023A1 and M-120620A1 because they both have the same period and number of layers (300 layers). From PSD (**appendix 8**) of that samples we see that, non ion beam polished is smoother. It can also be seen in the Rq values (red one). There was also made a comparison for other samples (**appendix 8-11**). At the end I compared all Ion polished beam samples(**appendix 12**).

NAME	Rq 1,nm	Rq 10,nm
M-120223A1	0,195	0,171
M-120620A1	0,277	0,302
M-120614A1	0,381	0,327
M-120228A1	0,157	0,128
M-120227A1	0,156	0,142
M-120612A1	0,221	0,231
4 inch Silicon wafer	0,156	0,126

Next comparison of the samples was made for one cut sample M-120223A1. Two scans were made: one in the center of cut part and another near to the edge (that scan is also used for previous comparison). From PSD (**appendix 13**) and Rq values one can see that there is no big difference in roughness of the samples:

NAME	Rq 1,nm	Rq 10,nm
M-120223A1-center	207	173
M-120223A1-edge	195	171

This comparison was made because, on different piece of M-120223A1 sample we found dirt, very uniformly spattered on the sample. The question was if was it due to oxidation which occurs with time (that would mean that samples are not very stable, which is of course not good),due to cut or due to some other factor. But we could not find any sing of such a pattern on the rest parts of the samples.

OTHER TASKS:

My additional task was to, write the software which could calculate the velocities of the coating of multilayer with certain period, and gamma factor. Not knowing any “higher level ” computing language like “Matlab”, or “Mathematica”, I tried to do write software in C++, using the Cramer method to solve equations, but that program was not very useful from because every value has some error which, has to be taken into account. After that I am trying to implicate the Jacobi method (first we assume that solution is a zero vector and then we make iterations in which iteration we increase the solution vector), this method seems to be more accurate because one do not have to have accurate solution but every solution has its precision (and the input has its on precision too). I probably will not manage to end useful software by the end of the Summer School, but it is important feedback for me, that my knowledge of computation is not good enough and in next semester I do have to concentrate on computer science more.

SUM UP:

AFM measurement shows clearly a few things first of all roughness is increasing with the period of SiC samples, then it was shown that there does not occur any sudden change of roughness value, around gamma 0.5. This results combined with X-ray diffraction give us an clear answer, that there is no change of W structure around gamma 0.5. However only perpendicular to the borders between two layers change in crystallization was checked, it is possible that crystals are building up along the line of layers and this changes stress in sample. To check it one would have to use:” HRTEM “ that is: High-resolution transmission electron microscopy- a mode of Transmission Electron Microscope. With that device you can make so called dark field imagining, the image is made by a diffracted electron beam. Maybe that kind of research would give an answer to changing stress behavior.

AFM measurement of ion beam polished samples and not polish one shows that those not polished are smoother, and its surface is more resistant for measurements, (it does not change during measurements).

Measurement of cut sample and comparison to other sample made at the same period shows us that sample are stable and do not oxidize during the time.

High usage of the tip, on standard NanoScope mode, give us a clue to use tapping mode during measurement of SiC samples. However the question remains why the value of

roughness in this mode is lower, so far the AFM manual or other sources do not give us an answer.

During the summer program 2012, gained really usefull knowledge of photon science, that include not only classic and electromagnetic optic but also broad knowledge about evaluating features of different materials. The most important part of my job was AFM measurement. Never before had I chance to work on that device, and thanks to program now I know the specifics of working with AFM. Experience, gained during those 87 measurement is very precious, now I do have “feeling” when measurement is reliable, how to change parameters of measurement to get “truth” value, change the tip etc...

At the end I would like to thanks very much to my supervisor:, Sasa Bajt, and all member of the group: Mauro Prasciolu, Miriam Barthelmess, Andrej Berg, for help and taking care of me during whole summer program.