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14. ABSTRACT This report results from a contract tasking Charles University as follows: The contractor will investigate the production of CdTe/CdZnTe crystals for use as substrates for epitaxial growth of HgCdTe layers for infrared focal plane array detectors. The contractor will deliver two CdZnTe wafers produced at Charles University with the target specifications identified in the proposal. The wafers should be shipped directly to: NVESD AMSEL-RD-NV/ST-IRT 10221 Burbeck Road Fort Belvoir, VA 22060 U.S.A. Attention: Dr. John Dinan					
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Final report

IR DETECTORS TECHNOLOGY

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1. Introduction

The final report summarizes the results of continuing research and development of CZT substrates for fabrication of IR focal plane arrays. During the contract period we concentrated on further development and characterization of the polishing process and on characterization of the as grown crystals prepared in the new reactor under high Cd pressure.

2. Results

2.1. Polishing

Testing of various polishing conditions and their characterization by Zygo intereferometer and AFM microscope was performed. Two polishing pads – the Hyprocell pellow and the Chemcloth, both supplied by Logitech, were tested. Comparable results of surface roughness were achieved in the preceding period on both polishing pads. These results were presented in the last progress reports. Recently we started testing of a third type of polishing pad supplied by PRESY, France. The first results of the effort on the new type of pad are presented.

In agreement with NVESD we started the round-up testing and polishing experiments on three Nikko samples, where the following sets of experiments is planned:

- 1) Evaluation of wafers by AFM with a scan areas of 10x10um and 30x30um.
2. Evaluation of the long-range flatness using the new Zygo interferometer.
- 3) Repolishing of the three Japanese wafers using optimized process.
- 4) Evaluation of the polishing by AFM using scan areas of 10x10um and 30x30 μm^2 and with new Zygo interferometer.

So far the first two tasks were completed. The results are presented in the following section. Due to the relatively late delivery of the wafers to Prague (end of June) it was agreed, that tasks 3 and 4 will be done in the first half of September based on time availability of the polishing lab and the summary will be sent in a special report.

2.1.1. AFM results of Nikko samples

AFM results of wafer ID42 are shown on Figs. 1 – 3.

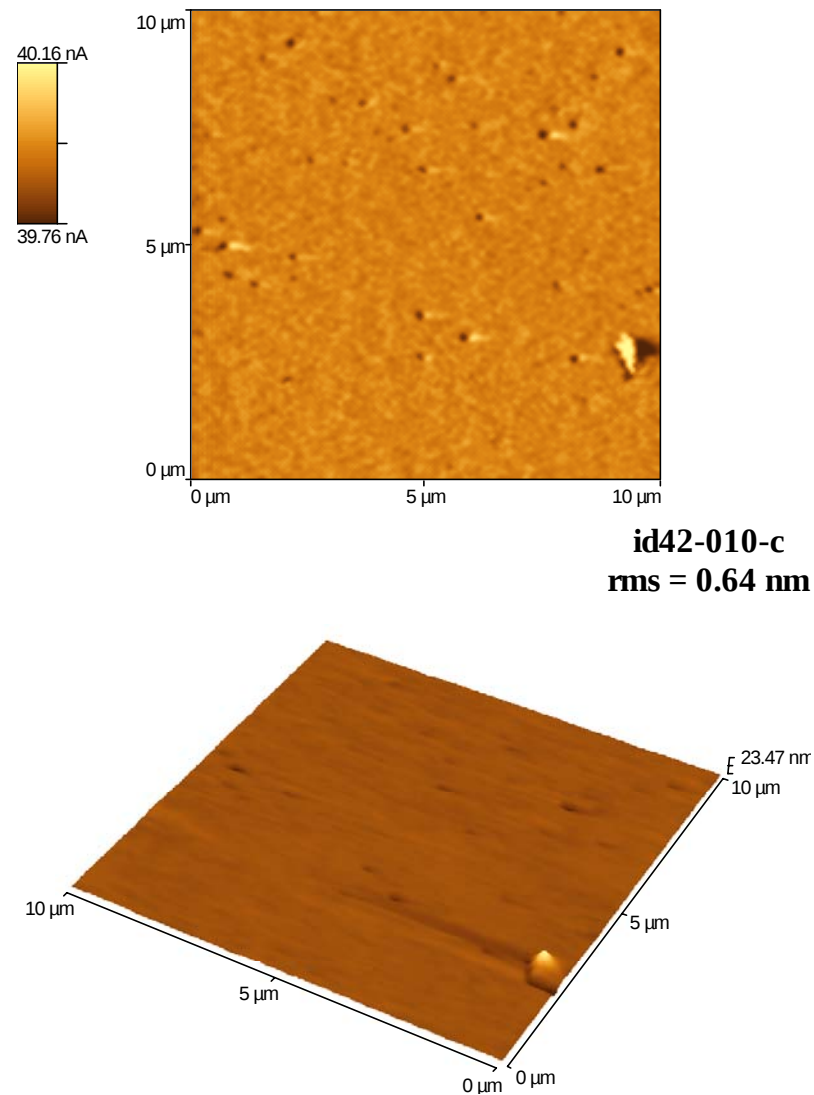


Fig.1 AFM measurement of sample ID 42, measuring area $10 \times 10 \mu\text{m}^2$

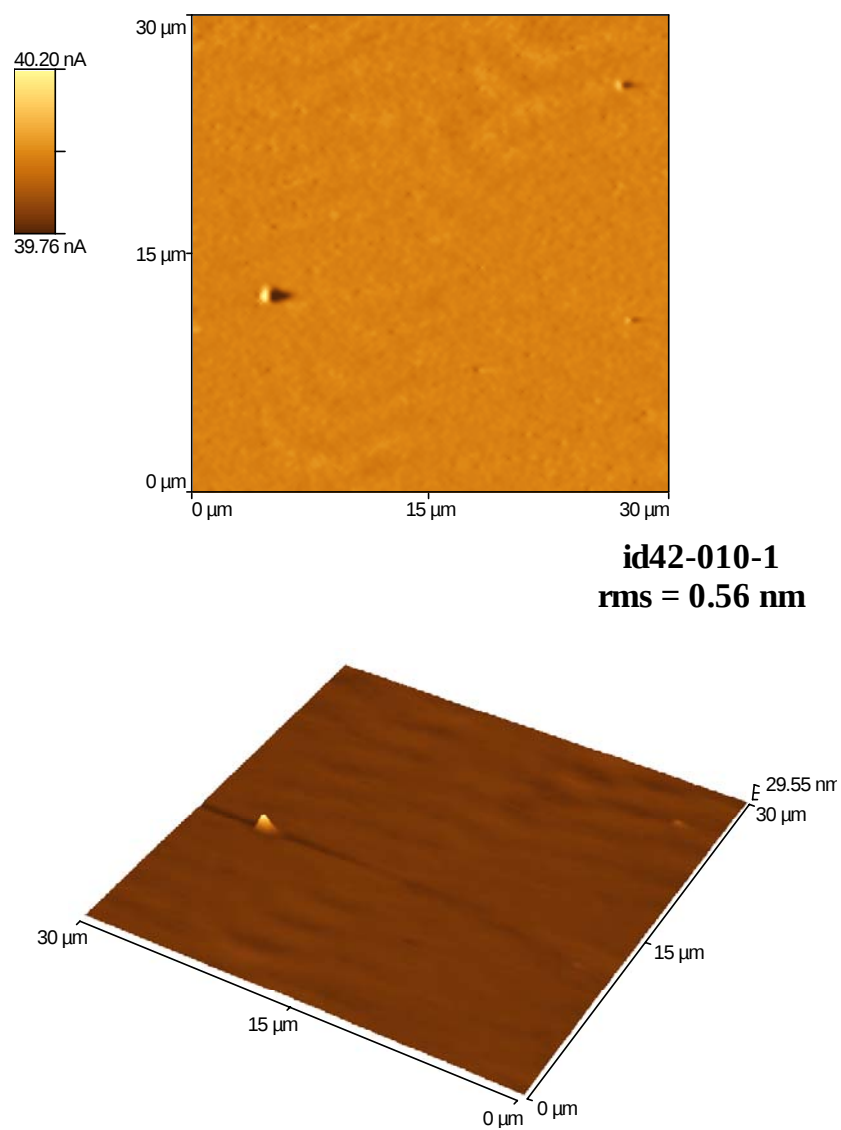
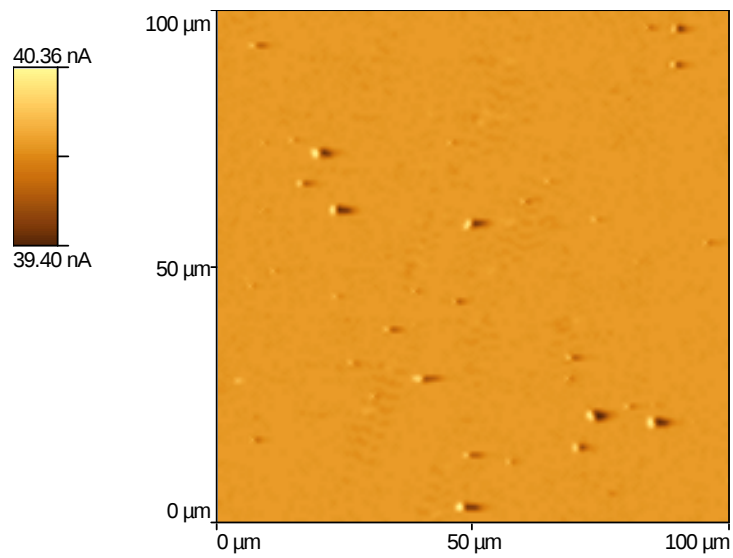


Fig.2 AFM measurement of sample ID 42, measuring area 30x30μm²



id42-100-1
rms = 1.32 nm

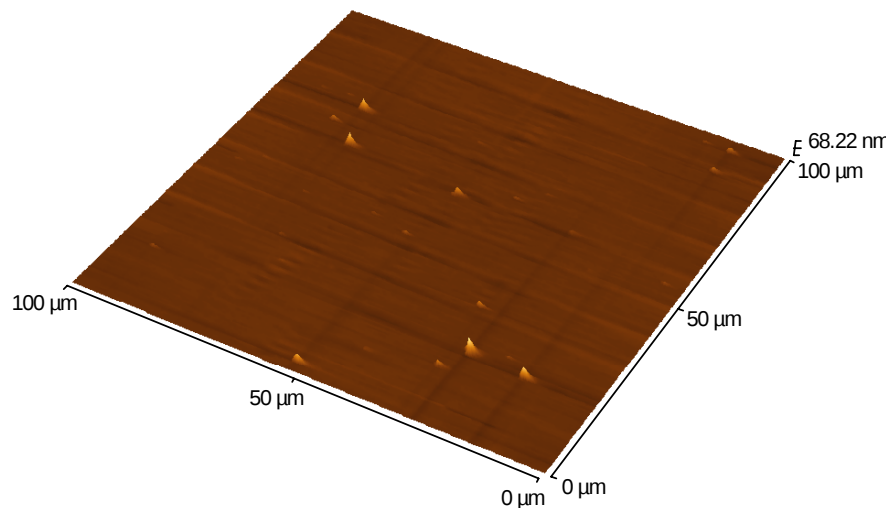


Fig.3 AFM measurement of sample ID 42, measuring area 100x100μm²

AFM results of two other Nikko wafers are similar (ID-43=0.57nm, ID-52=0.73nm, measuring area 10x10 μm^2).

2.1.2. Zygo results of Nikko samples

Interferometric measurements of three Nikko wafers on area 170x130 μm^2 are presented on Figs.4-6 and in Table 2. They show very good flatness (rms 0.2-0.25nm, peak to valley distance P/V 2.7-3.3nm) confirming the results of AFM measurements.

	Rms(nm)	
	Cylinder removed	Plane removed
Id42-z1a	0.373	0.762
Id42-z1b	0.446	0.694
Id42-Z1c	0.293	0.941
Id42-z2a	0.228	0.256
Id42-z2b	0.205	0.329
Id42-z2c	0.226	0.444
Id43-z1a	0.298	0.818
Id43-z1b	0.276	0.622
Id43-z1c	0.286	1.155
Id43-z2a	0.267	0.351
Id43-z2b	0.207	0.253
Id43-z2c	0.232	0.378
Id52-z1a	0.332	1.575
Id52-z1b	0.287	0.486
Id52-z1c	0.437	0.622
Id52-z2a	0.304	0.500
Id52-z2b	0.244	0.279
Id52-z2c	0.271	0.534

Table 2.

Summary of results of AFM measurements on Nikko samples. The symbol z1 means 352x264 μm^2 measuring area, the symbol z2 the 176x132 μm^2 measuring area.

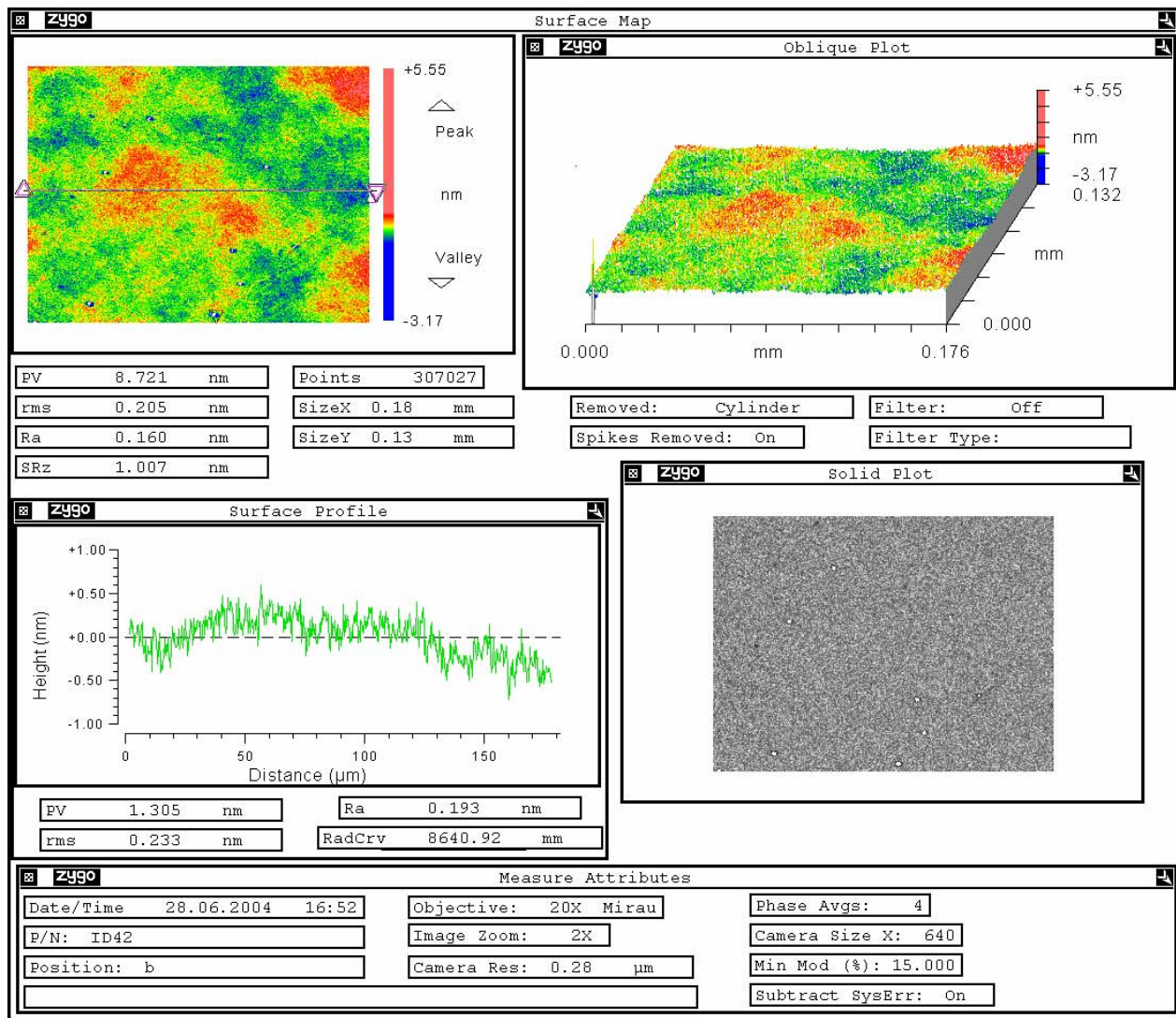


Fig.4 Zygo results of wafer ID-42

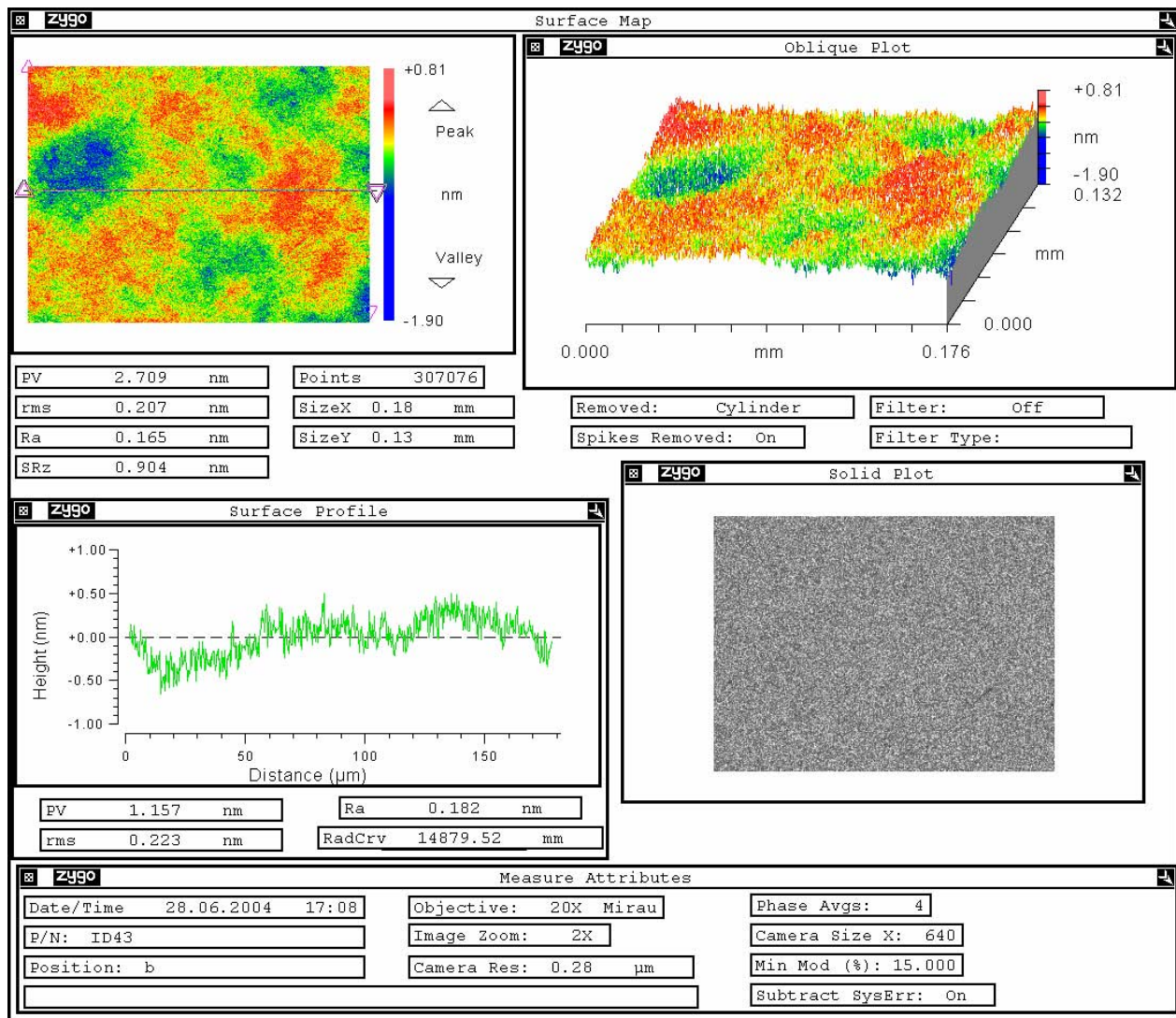


Fig.5 Zygo results of wafer ID-43

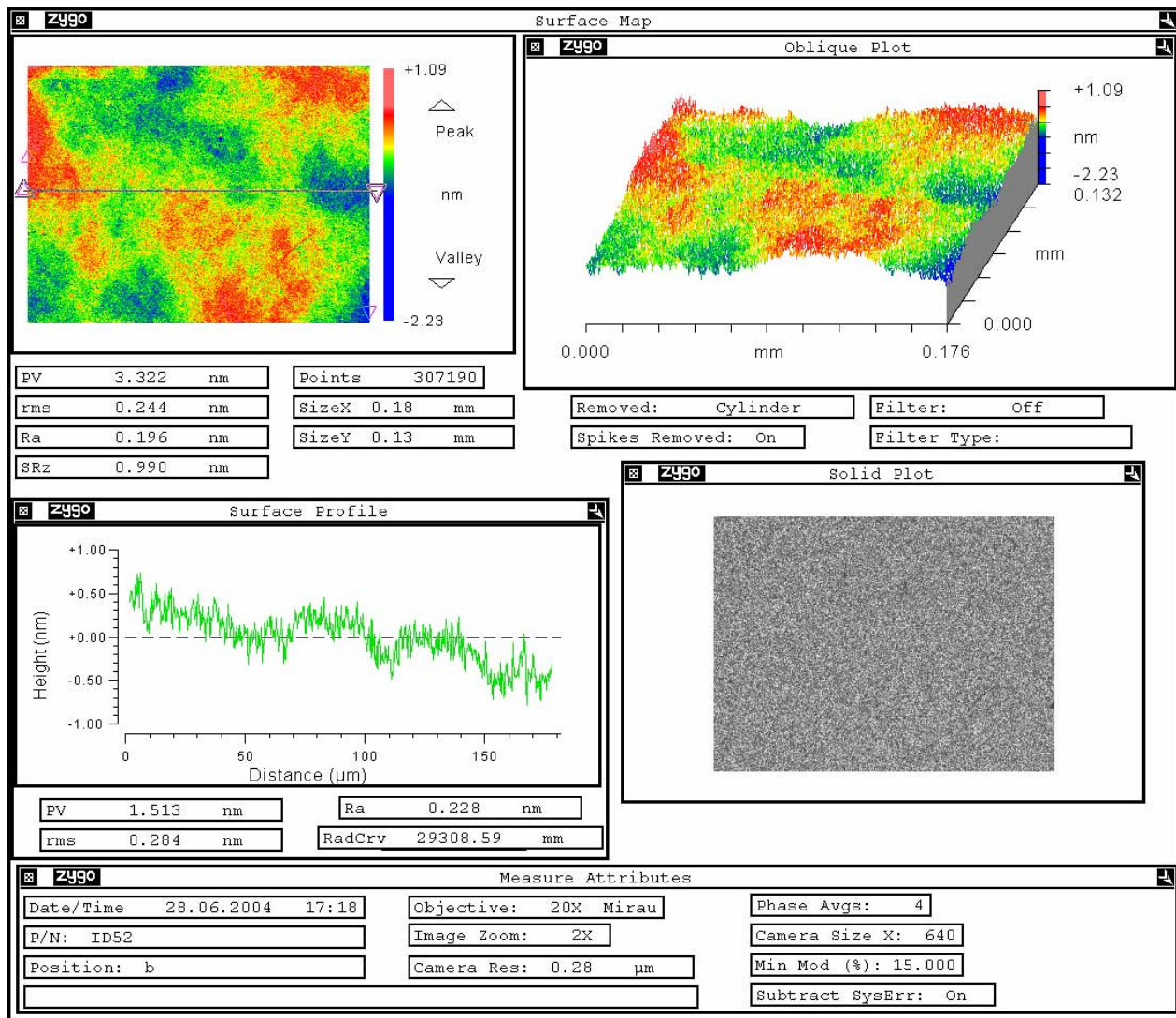


Fig.6 Zygo results of wafer ID-52

The Zygo results show good surface roughness of all studied wafers ($\text{rms} \sim 0.2\text{-}0.45\text{nm}$) when cylindric correction was applied. The rms increased in some cases, when plane correction was applied. We interpret this result by existence of some longer range variation of surface, which probably has no influence on the quality of MBE layers.

2.1.3. Latest polishing results using a new type of polishing pad

The 3x3 cm² wafer delivered together with this report was polished using a new type of polishing pad delivered by PRESY, France. The first results show a better reproducibility of the etching process and certain improvement of the surface roughness, which was achieved around 1nm. Results of Zygo measurement are presented on Fig.7.

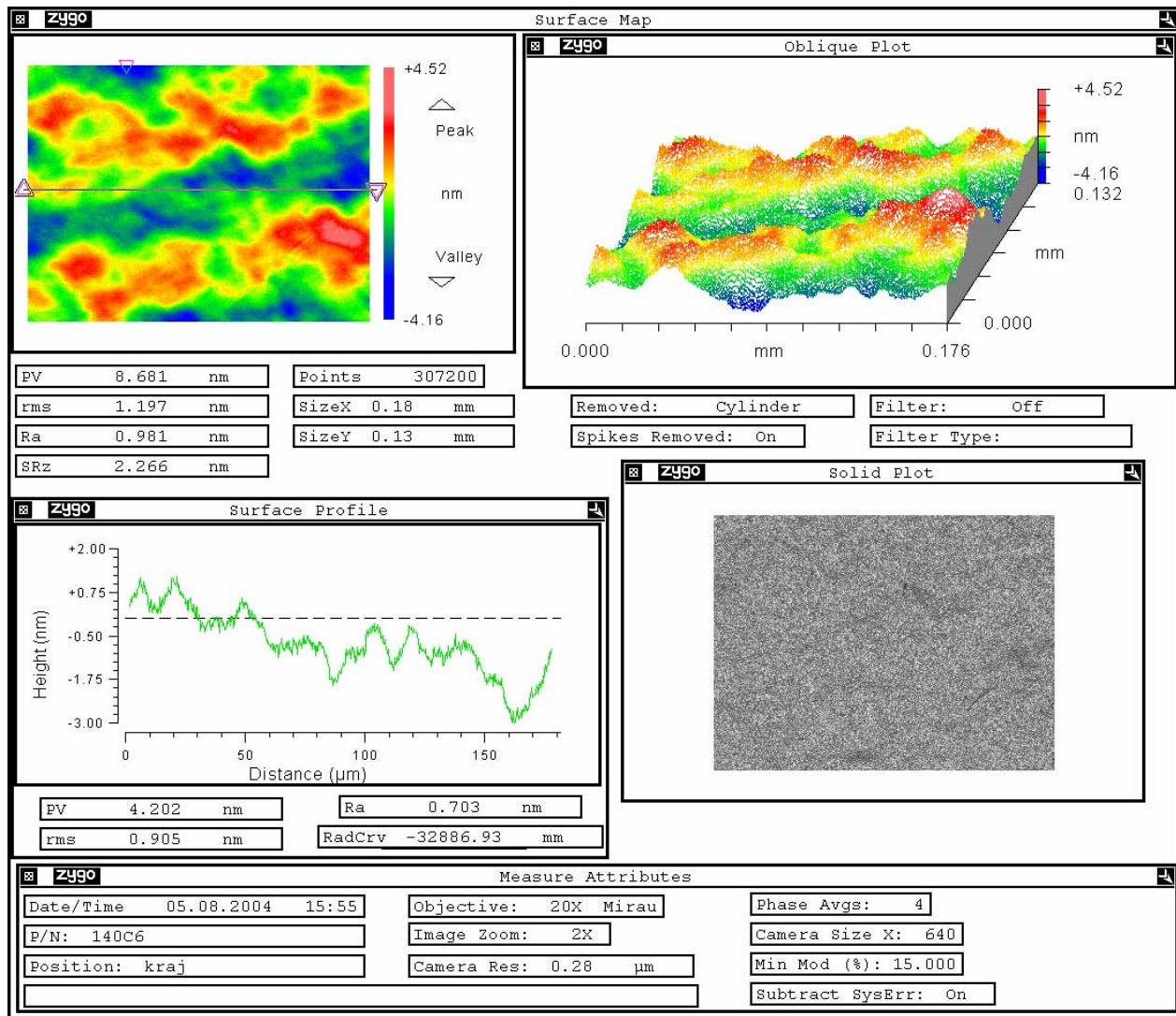


Fig.7 Zygo results of measurements of wafer 140c6

2.2. Crystal growth and characterization

The results of the growth in the new setup were in detail described in the previous three progress reports and will not be repeated here. The crystals had good transmissivity, no inclusions, favorable course of Zn content and in some cases also large grains enabling to cut larger substrates. However, they did not pass the dislocation density and X-ray rocking curves test. These both parameters are connected. If the microstructure is not good, the dislocation density is also high. The most probable reason for this problem, known from literature, is use of quartz ampoules without crucibles. The stress from walls of the ampoules during cooling, caused by different thermal expansion coefficients of quartz and solid CZT, causes the damage. The reason for use of quartz was, that we wanted to make the growth cheaper. Based on the identification of problem of higher dislocation density the switch to a use of pyrolytic boron nitride crucibles, which have thin soft walls substantially decreasing the thermal stress and dislocation generation, is planned. Additionally to this main reason, any instability of temperature or Cd pressure during the growth, especially at the solidification temperature, will also cause problems with microstructure, because it generates additional defects. It is expected, that the two planned changes - use of PBN crucibles and modification of the furnace, will provide more favorable growth conditions leading to high quality crystals with a good microstructure, keeping the advantages of the higher Cd pressure - good transmissivity and low concentration of inclusions.

In parallel with the effort to optimize growth conditions in the new middle pressure setup growth in the old, low-pressure setup continued. From the crystal prepared in this setup, a 3x3cm² (211) oriented wafer was cut and mechanically and chemically polished. The wafer is delivered to NVESD for testing of its properties together with this final report.

2.3 Planned modification of the middle pressure growth setup

Based on the practical experience with the current work of the new setup we plan to modify the growth setup. There are two furnaces in the growth pressure chamber. One for growth (3 zone), the other for Cd pressure control. The theory says, that by change of temperature of Cd

zone the Cd pressure can be set. Therefore, based on the known dependence of pressure of vapors above the pure Cd metal, one should be able to control automatically the Cd pressure above the boule. However, in practice, the result depends on the dynamical flow of Cd vapors in the system. In our case it appeared, that the diameter of the Cd zone is insufficient to enable optimal heat transfer from Cd vapors in the Cd zone. In other words, the real temperature of Cd vapors in the Cd zone was higher, than the nominal value set by the temperature controller. It was still possible to control the Cd pressure in this system, but this had to be done manually, because it was not possible to use the dependence of Cd pressure on temperature from literature. Manual control required almost permanent presence of the grower at the setup. Furthermore, the manual control is never so precise as the automatic one and the growth conditions in the system were therefore less stable than expected. In order to eliminate these problems, Cd furnace with a larger diameter is necessary. This requires to

- a) fabricate new furnace for Cd zone
- b) to cut the high pressure chamber in the factory, drill a larger hole on the top and to weld the new upper part of the high pressure vessel, which will contain the Cd furnace to control the Cd pressure.

3. Conclusion

During the contract growth of CZT under high Cd pressure (2-4atm) was tested in the new middle-pressure setup in order to evaluate the properties of such crystals for application of substrates for MBE epitaxy. The main motivation to increase Cd pressure during the growth was to eliminate the presence of Te inclusions, which were often present at growth runs performed at standard growth conditions (Cd pressure~1-1.3atm). The results of testing of crystals grown in the new setup have shown, that in most cases the presence of inclusions larger than 1 μ m was fully eliminated and the crystals achieved nearly theoretical optical transmission. From this point of view the application of higher Cd pressure is definitely a perspective way to follow. At the same time we observed higher concentration of dislocations (10^5 - 10^6 cm⁻²) and higher FWHM (45-70arcs) than was typical for crystals grown in the low-pressure setup. We connect this problem with the application of quartz ampoules resulting in presence of thermal stress during crystal cooling. Measures were taken to eliminate this problem – use of PBN crucibles and

planned modification of the middle pressure setup in order to provide more stable growth conditions and to enable fully automatic Cd pressure control while keeping the higher Cd pressure to eliminate inclusions. The start of growth using the above mentioned modifications is planned in September.

During the contract period more than 100 polishing experiments testing various combinations of polishing conditions and polishing pads were performed and significant improvement of surface roughness parameters was achieved. While the producer of the polishing machine (Logitech, Scotland) guaranteed rms 7nm, standard result ~ 1.2 nm was obtained after optimization of the polishing process (Zygo, measurement area $170 \times 130 \mu\text{m}^2$). AFM results on smaller area $5 \times 5 \mu\text{m}^2$ (rms ~ 0.5 - 0.7 nm) are practically comparable with Nikko wafers. In order to remove longer range variations of surface, which cause that the rms measured by Zygo is factor 3-4 worse than on Nikko samples, continuation of development of the polishing process is planned. The activity will concentrate on minimization of mechanical damage of the surface during mechanical polishing and on testing of new polishing pads, which guarantee better homogeneity of distribution of the etchant during the polishing process.