CARBON COATINGS FOR BIO-MICRO-REACTORS

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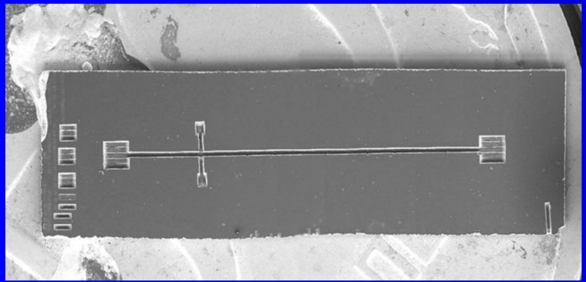
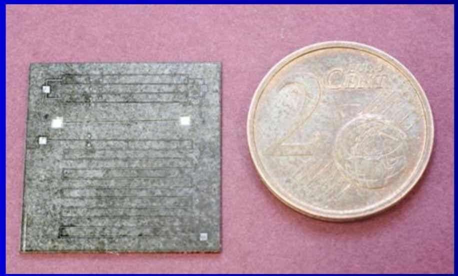
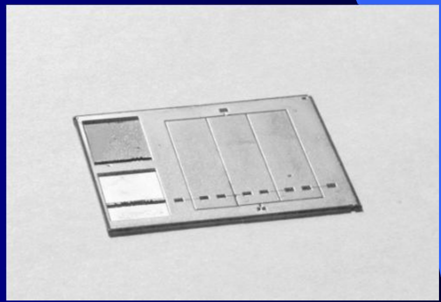
Diamond Microfluidic Devices – previous PROJECTS

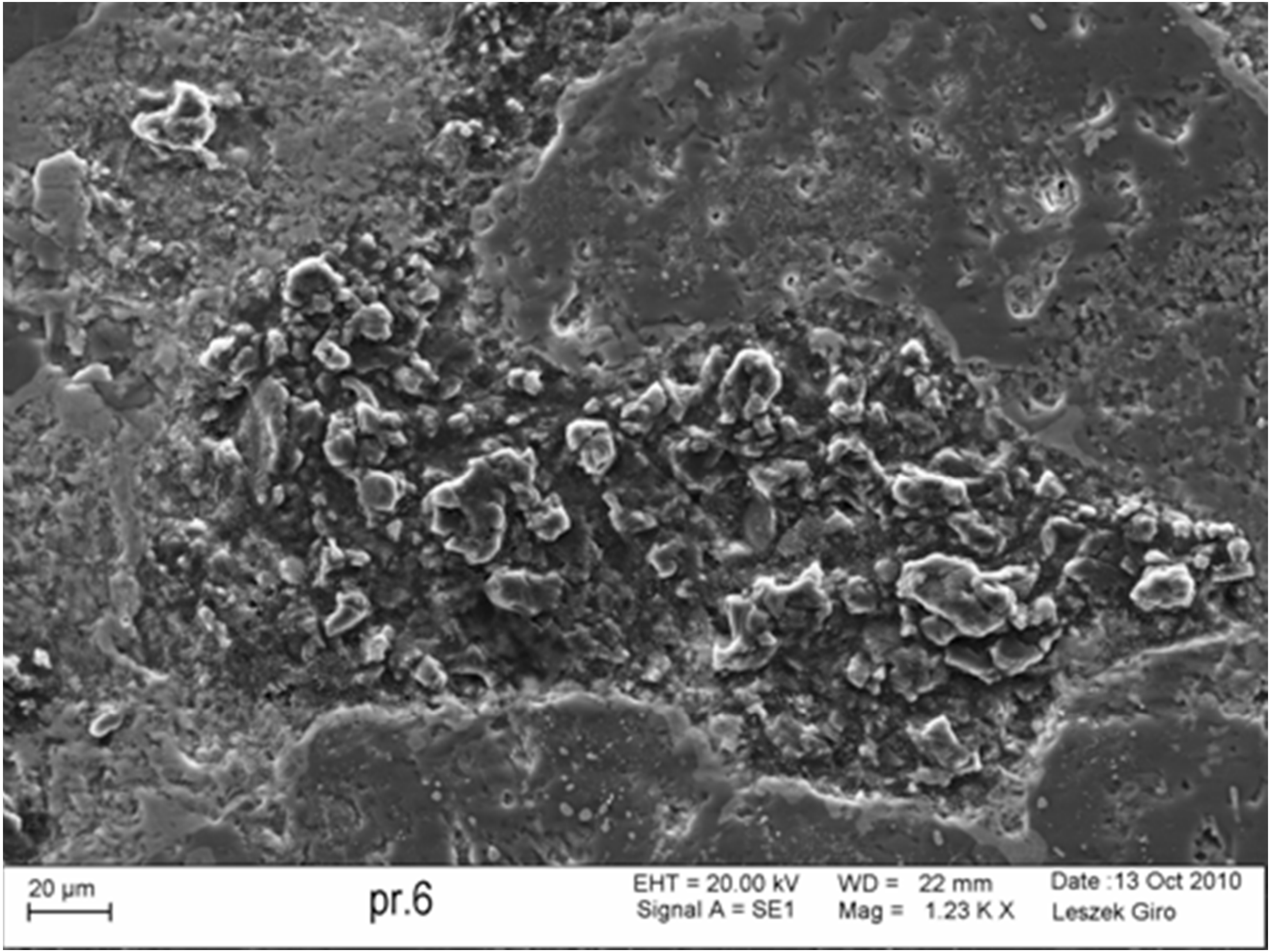
1997-2000 EU project µDiaGene (UK Imperial College, Dr. John Hassard; Italy, Germany, Poland - Technical University of Lodz&ITE)

2007-2009 MNT ERA-NET project „Diamond Microfluidic Devices for Genomics and Proteomics" (Technical University of Lodz, ITE – Poland; Imperial College, DeltaDot – UK; Technical University of Liberec – Czech Republic)





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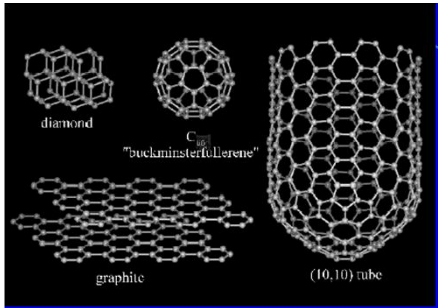
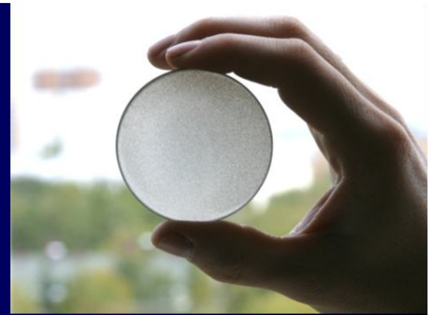
Mag = 1.23 K X
Date :13 Oct 2010
Leszek Giro



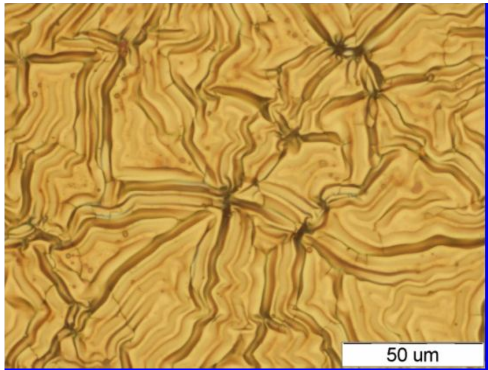
Carbon materials - diamond



Different allotropic forms of carbon posses the unique properties which allow to apply it in many fields of science and technology.
Diamond exhibits good optical properties, it means broad transmission spectral range; it is very hard and stiff, shows the highest known thermal conductivity, low thermal expansion coefficient, is chemically inert, not affected to all bases and acids reagents and together it is bioactive.

Last decades advances in diamond materials technology, especially different chemical vapour deposition techniques, allow both to manufacture large area CVD polycrystalline diamond plates of different grades and different kinds of diamond-like coatings and powders.

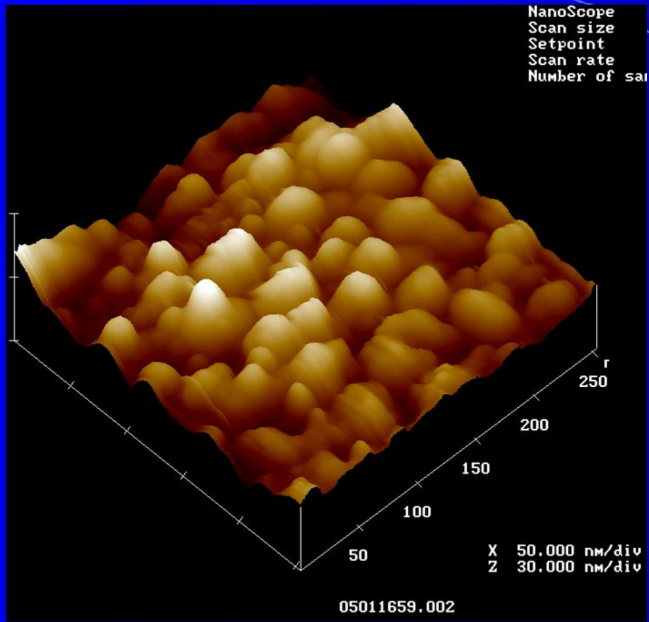
Depending of the grade of polycrystalline material and of the composition (sp^3 to sp^2 content), carbon materials posses wide range of properties. The changes of the properties are due to the reduction of the crystals sizes, existence of defects and the existence of the grain boundries, increase of sp^2 bonds in the material and change of the composition (impurities).



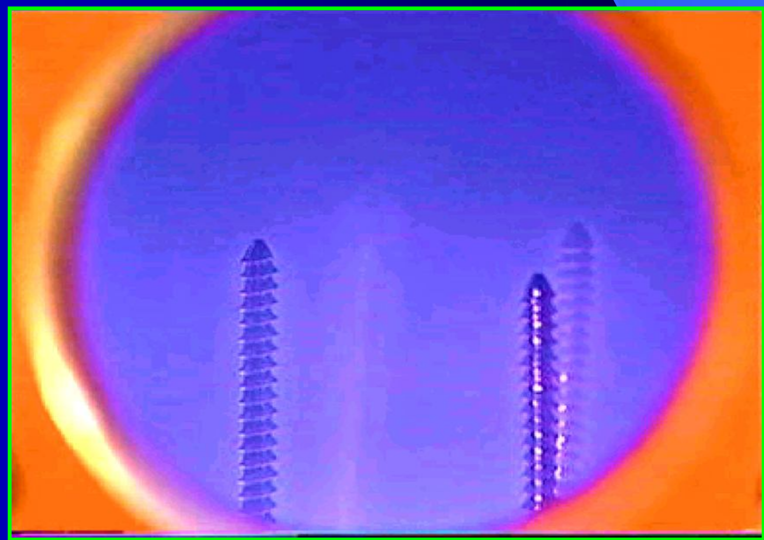
Carbon materials

- Due to their exceptional physico-chemical properties, such as: high hardness, chemical inertness, optical transparency in a wide spectral range, high thermal conductivity and low electrical conductivity, diamond-like carbon coatings (DLC) receive increased attention as a relevant materials for a construction of bioelectronic devices and a prospective substrates for a tissue engineering.

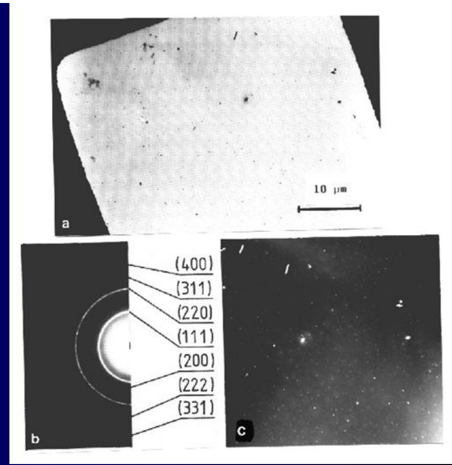
Presented here research is focused on the modification of the surface properties of polydimetylosiloxane (PDMS). The surface of PDMS was coated with diamond like carbon (DLC) films using two different plasma assisted chemical vapor deposition methods: MW/RF PACVD and RF PACVD/MS (combined with magnetron sputtering). By varying deposition parameters such as: time of initial plasma etching (3 to 10 min), composition of gases (CH_4, Ar, O_2), time of deposition (3 to 30 min), autopolarisation voltage and sputtering power supplied respectively to the electrode and magnetron.



CVD technique



Carbon coatings synthesis



RF PACVD/MS

MW/RF PA CVD

Carbon based coatings were synthesized using two techniques: MW/RF PACVD and RF PACVD/MS. Modification processes were carried out to obtain different surface properties of carbon coatings deposited on PDMS surface.

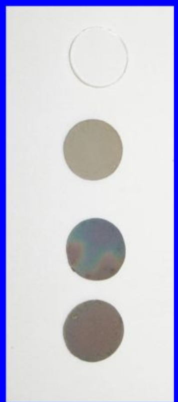
MW/RF PACVD

- The technology is based on two energy sources: MW/RF (*Microwave / Radio Frequency)*. In those solutions microwave plasma characterized by high density of ions stream decides on generating and ionization ratio of active particles, whereas RF plasma controls the stream and energy of the ions. Those systems make it possible to conduct the deposition processes in wide range of the parameters, thus they demonstrate the advantage over the conventional RF processes.

RF PACVD/MS

- Gradient carbon layers were synthesized using hybrid deposition system employing magnetron sputtering and RF PACVD techniques into the one reaction chamber. Presented combination makes it possible to solve the problems with adhesion of carbon layers due to their high residual stress which determines their thickness as well. As the magnetron target titanium cathode was used. a-C:H/Ti layers synthesis process assumes the deposition of thin adhesion promoting Ti interlayer. After that during the magnetron sputtering process methane is introduced into the reaction chamber. High chemical affinity of titanium to carbon lets to obtain a buffer layer between carbon and intermediate layer. As the final stage of the deposition process thick carbon layer is synthesized.

The parameters of the most important processes

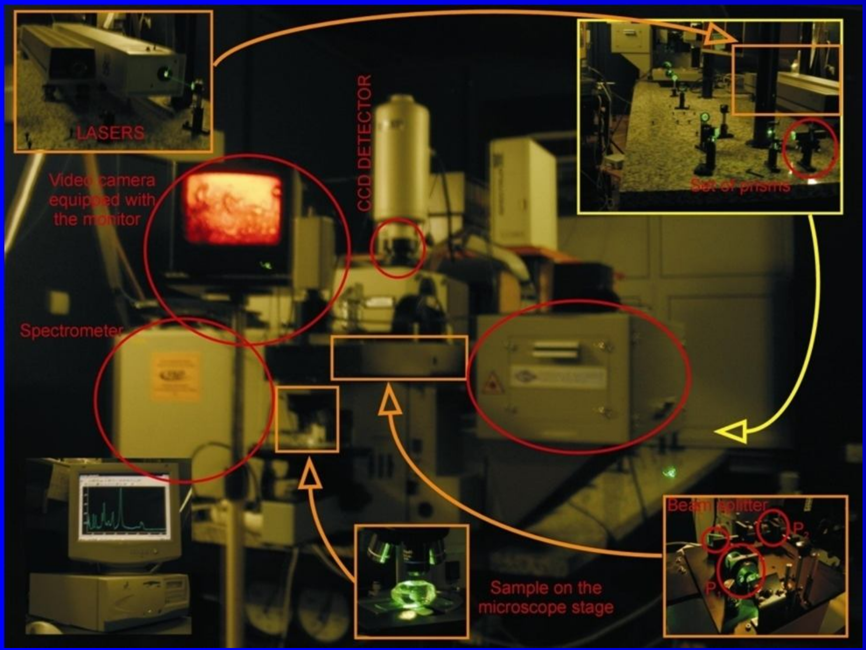


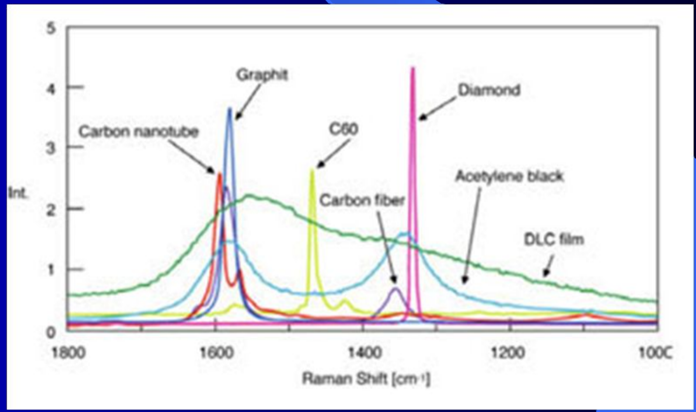
Sample	Parametry procesu			
	Etching	**Time**	**Coating deposition**	**Time**
MS/RF_1 15.06.09_2 (RFPACVD/MS)	**Gases:** 10sccmAr +10sccmO_2, *V_B = 600V	5 min.	**Gases:** 10sccmAr + 10sccmCH_4, **I_G =5A, *V_B = 400V	3 min.
MS/RF_2 15.06.09_3 (RFPACVD/MS)	**Gases:** 10sccmAr +10sccmO_2, *V_B = 600V	10 min.	**Gases:** 10sccmAr + 10sccmCH_4, **I_G =5-4A, *V_B = 300V	5 min.
			Gases: 30sccm CH_4, *V_B = 540V	5 min.
MS/RF_3 15.06.09_4 (RFPACVD/MS)	**Gases:** 10sccmAr +10sccmO_2, *V_B = 600V	10 min.	**Gases:** 10sccmAr + 10sccmCH_4, **I_G =5-4A, *V_B = 300V	5 min.
			Gases: 30sccm CH_4, *V_B = 540V	5 min.
			Gazy: 10sccm O_2, *V_B = 100V	1 min.
MW/RF_1 18.01.10 (MW/RF PACVD)	**Gases:** - *$^{a}M_{RF}$ = 200W *$^{b}M_{MW}$ = 900W	3 min.	**Gases:** 60sccm CH_4, *$^{a}M_{RF}$ = 200W *$^{b}M_{MW}$ = 900W	10 min.
MW/RF_2 16.05.06 (MW/RF PACVD)	**Gases:** 10sccmCH_4 *$^{a}M_{RF}$ = 100W *$^{b}M_{MW}$ = 750W	3 min.	**Gases:** 25sccmCH_4 *$^{a}M_{RF}$ = 90W *$^{b}M_{MW}$ = 750W	20 min.
MW/RF_3 17.06.02 (MW/RF PACVD)	**Gases:** 30sccmCH_4 +18sccmO_2, *$^{a}M_{RF}$ = 500W *$^{b}M_{MW}$ = 650W	5 min.	**Gases:** 60sccmCH_4 *$^{a}M_{RF}$ = 500W *$^{b}M_{MW}$ = 650W	3,5 min

Characterization methods

- The structure of modified samples was examined with use of the Olympus GX 71 optical microscope.
- Surface morphology and topography were measured using atomic force microscope AFM Multimode microscope equipped with Nanoscope V controller (Bruker Corporation, USA) working in tapping mode.
- Contact angles were measured with use of KRUSS Contact Angle Measuring Instrument, using sessile drop method.
- Phase composition, particularly sp^2/sp^3 phase ratio, was examined by means of Raman spectroscopy with use of T-64000 Yobin-Yvon spectroscope equipped with Ar laser working at 514,5 nm argon.
- Studies of X-ray photoelectron spectroscopy were carried out using ESCALAB-210 system (VG Scientific UK) equipped with non-monochromatic Al (K_α = 1486.6 eV) X-ray source operated at 14,5 kV and 20 mA. Experimental spectra were deconvoluted using Avantage 4.84 (Thermo Electric) software.

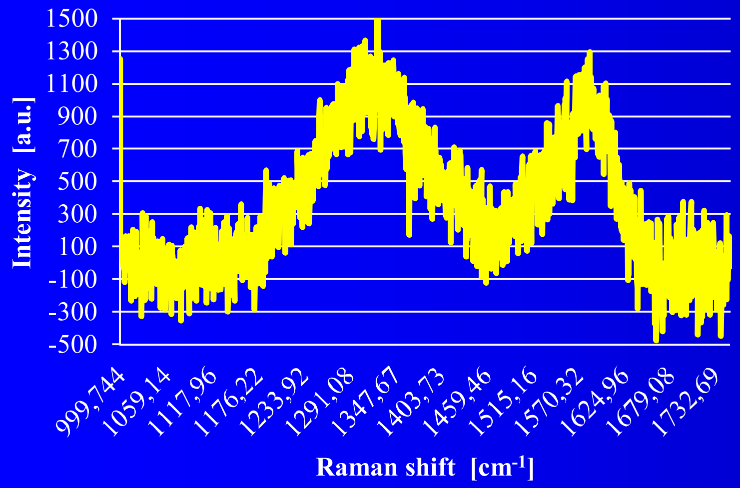
Raman spectra were recorded using the confocal Raman micro-spectrometer T-64000 (Jobin-Yvon) equipped with the microscope BX-40 (Olympus). The 514.5 nm Ar line was used for sample excitation. The other parameters of spectra acquisition (time, laser power) were adjust to obtain good quality spectra. The diameter of laser beam was 1.5 μm, the light intensity across the beam is of Gaussian distribution.



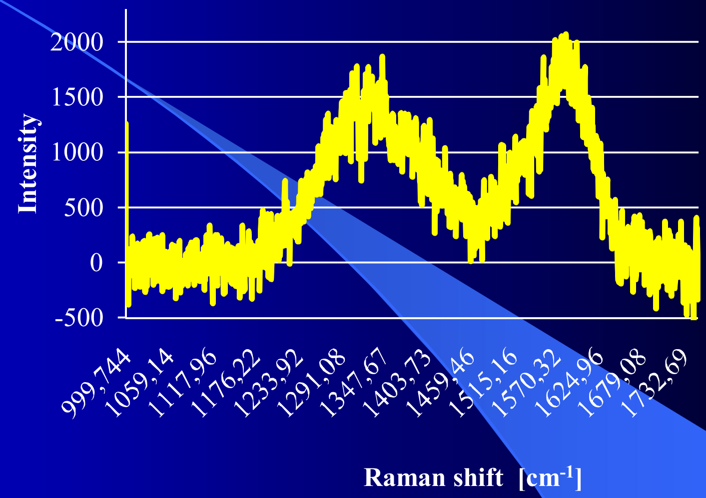


Raman Spectroscopy results

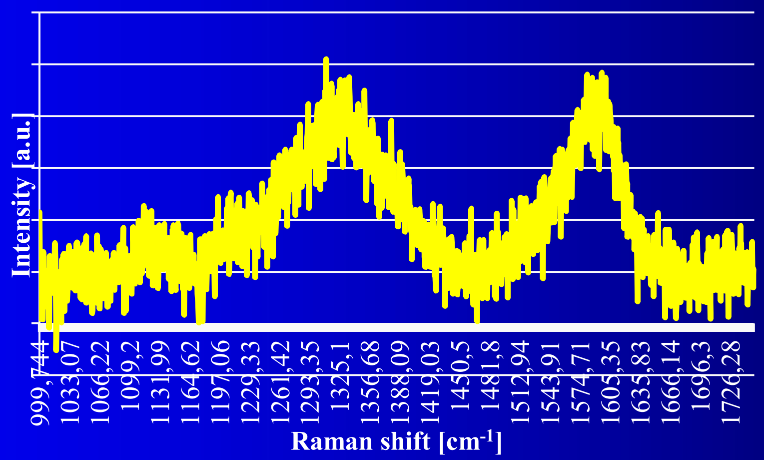
MS/RF_1



MS/RF_2

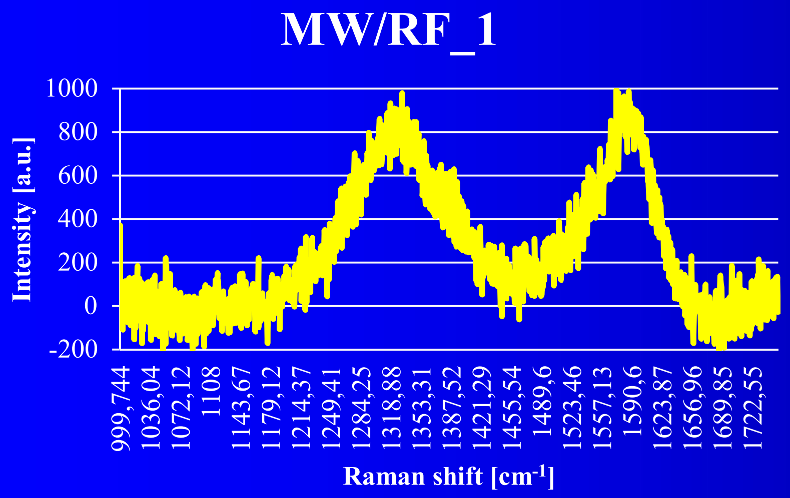


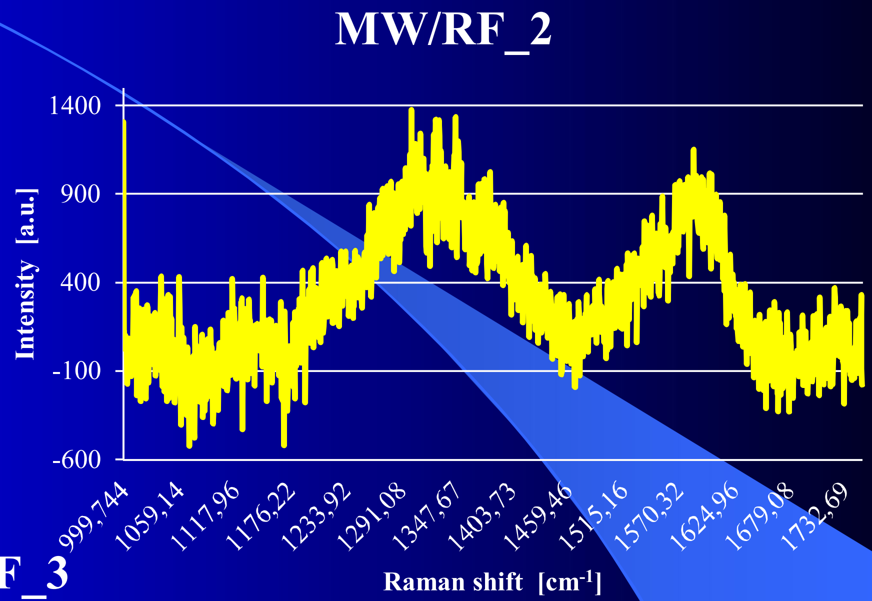
MS/RF_4

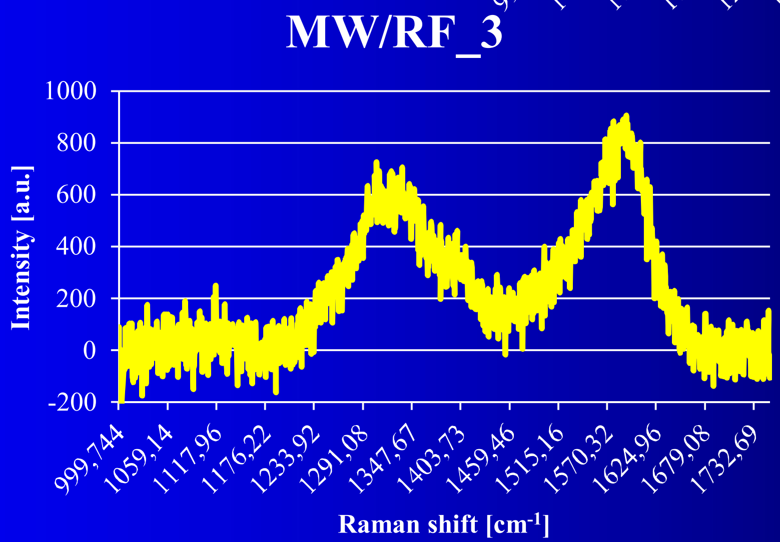


D-band around 1330 cm^{-1} and G-band around 1580 cm^{-1}

Raman Spectroscopy results

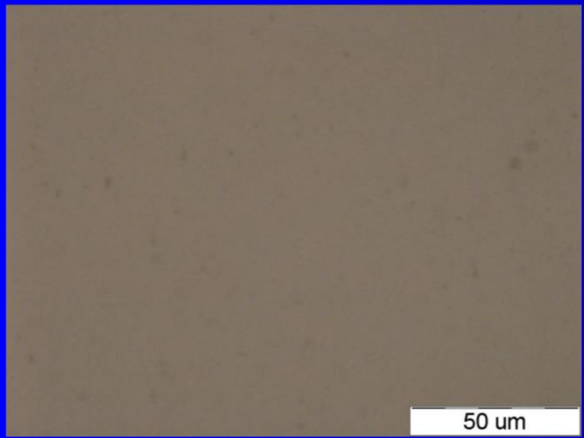




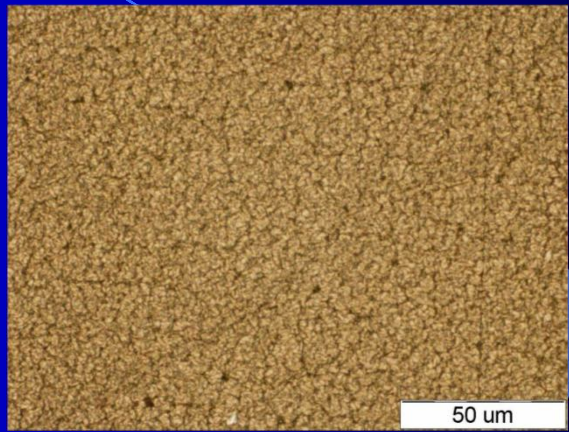


D-band around 1330 cm^{-1} and G-band around 1580 cm^{-1}

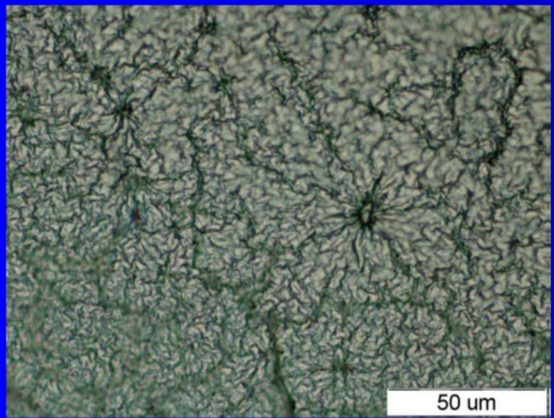
Optical microscopy



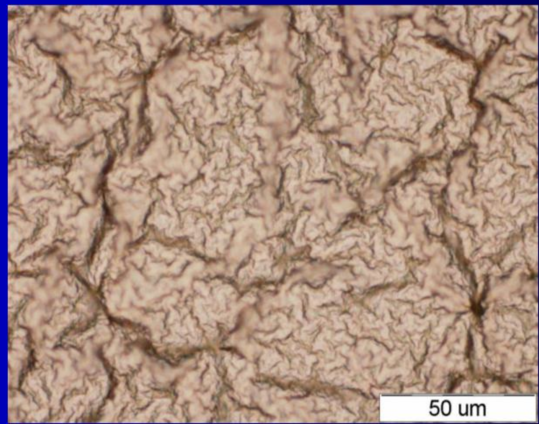
Unmodified PDMS



MS/RF_1 (15.06.09_2)

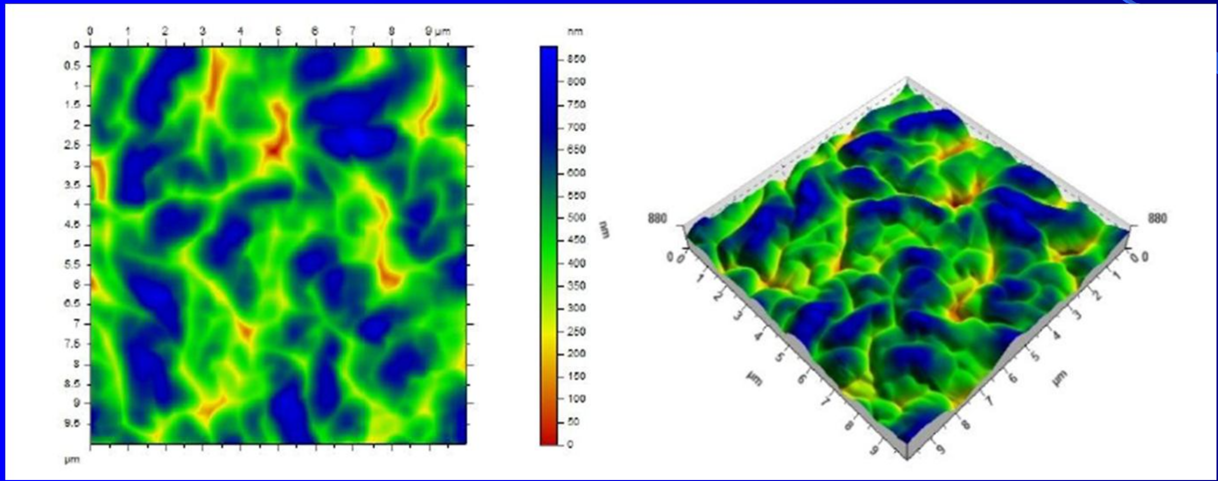


MS/RF_2 (15.06.09_3)

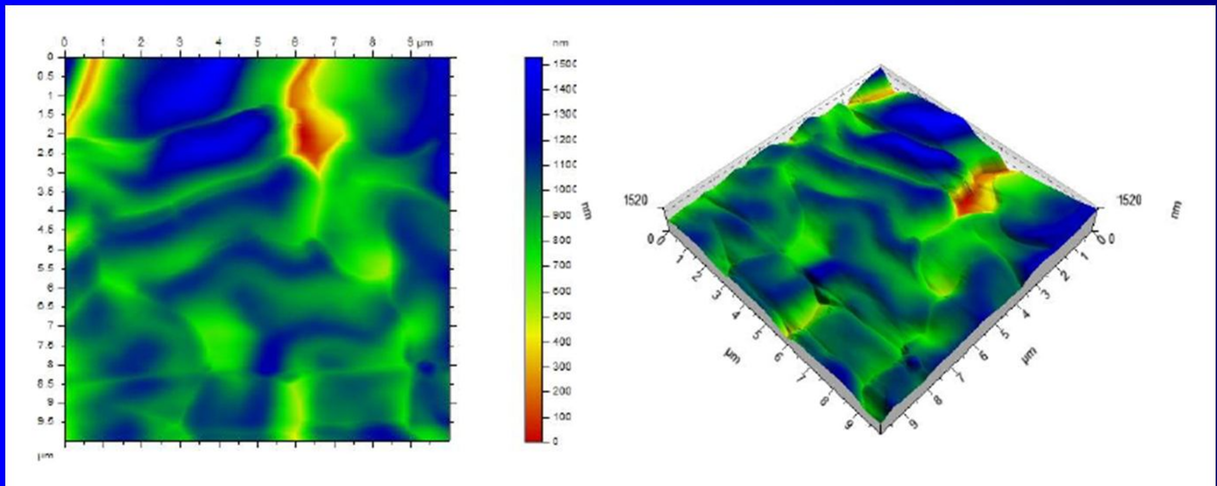


MS/RF_3 (15.06.09_4)

AFM results

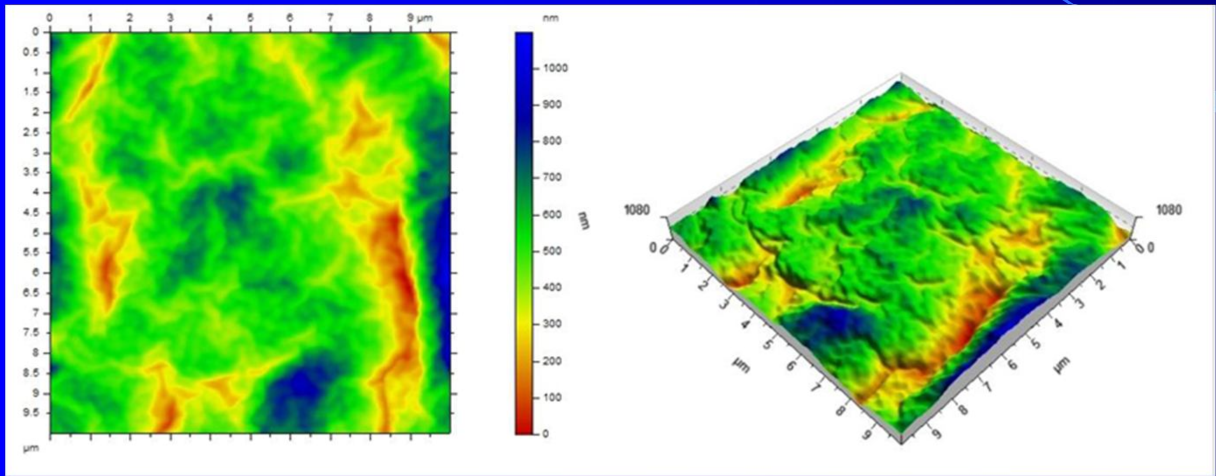


MS/RF_1

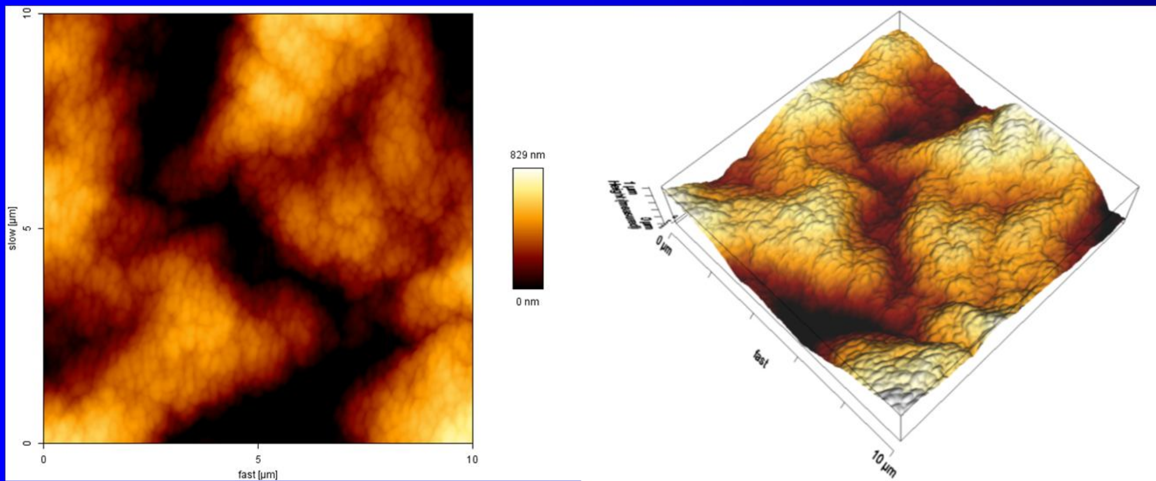


MS/RF_2

AFM results

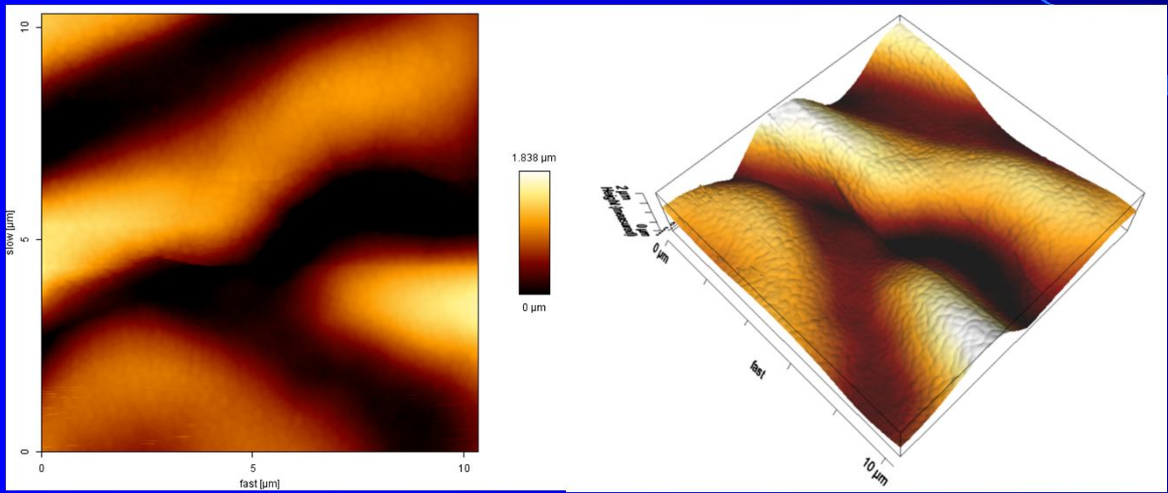


MS/RF_3

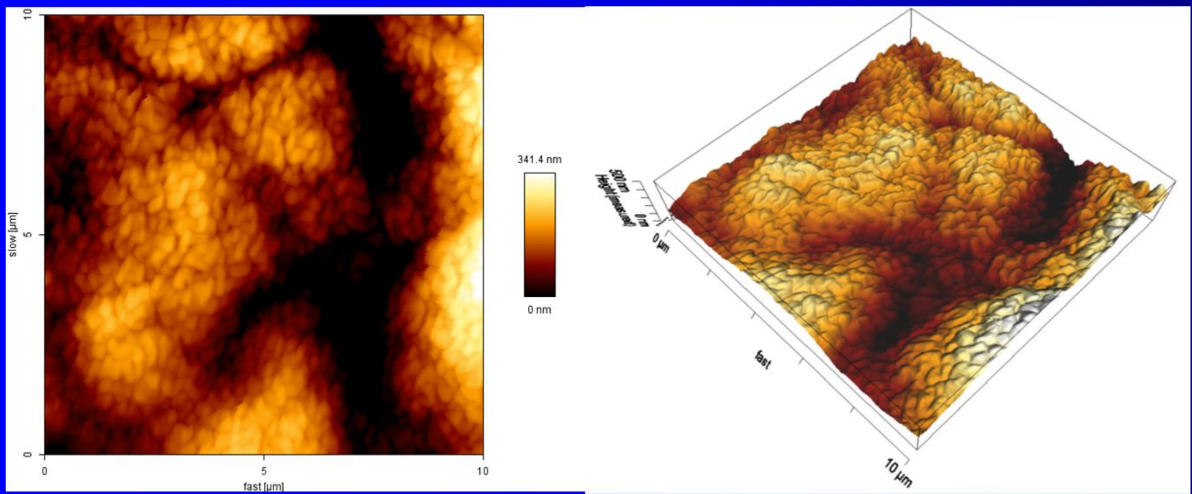


MW/RF_1

AFM results

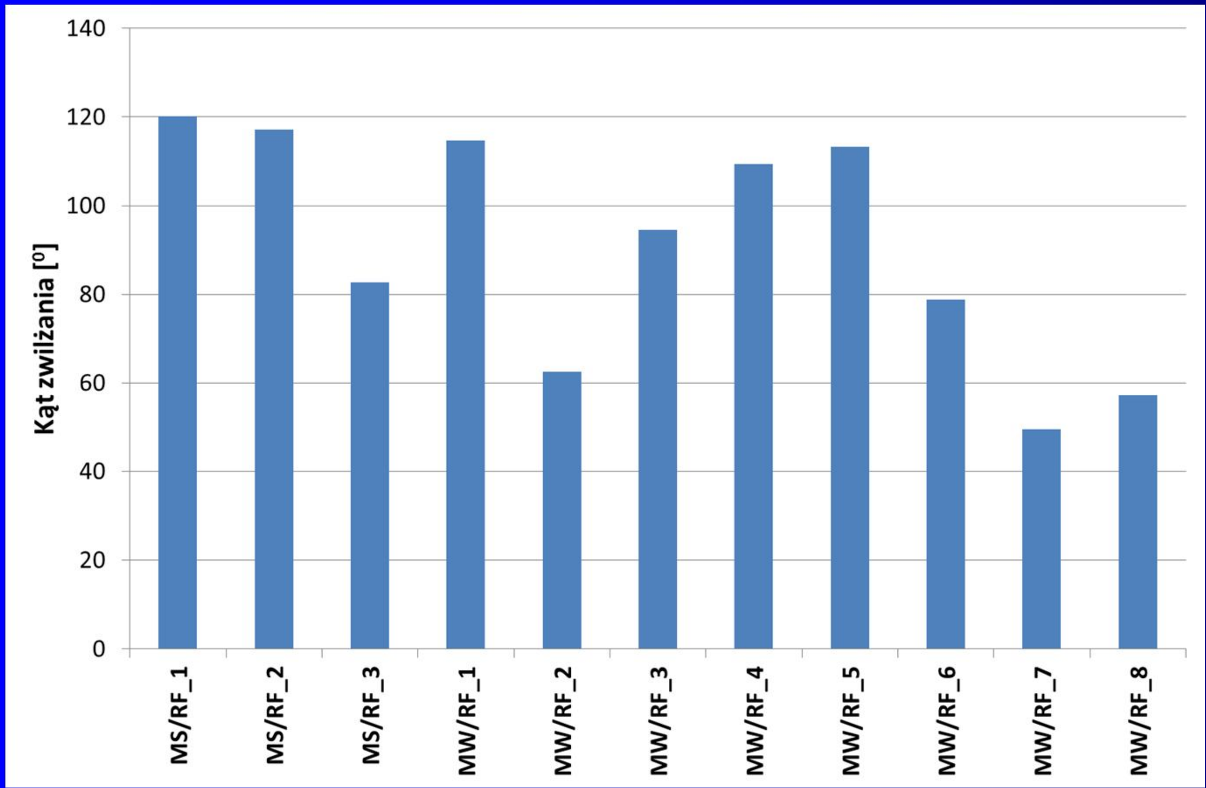


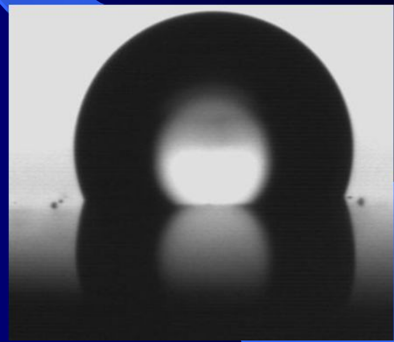
MW/RF_2

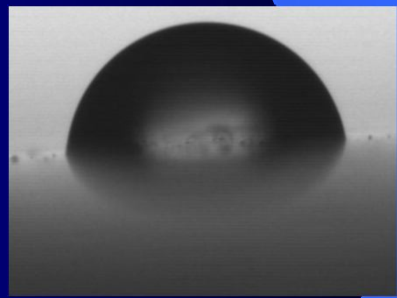


MW/RF_3

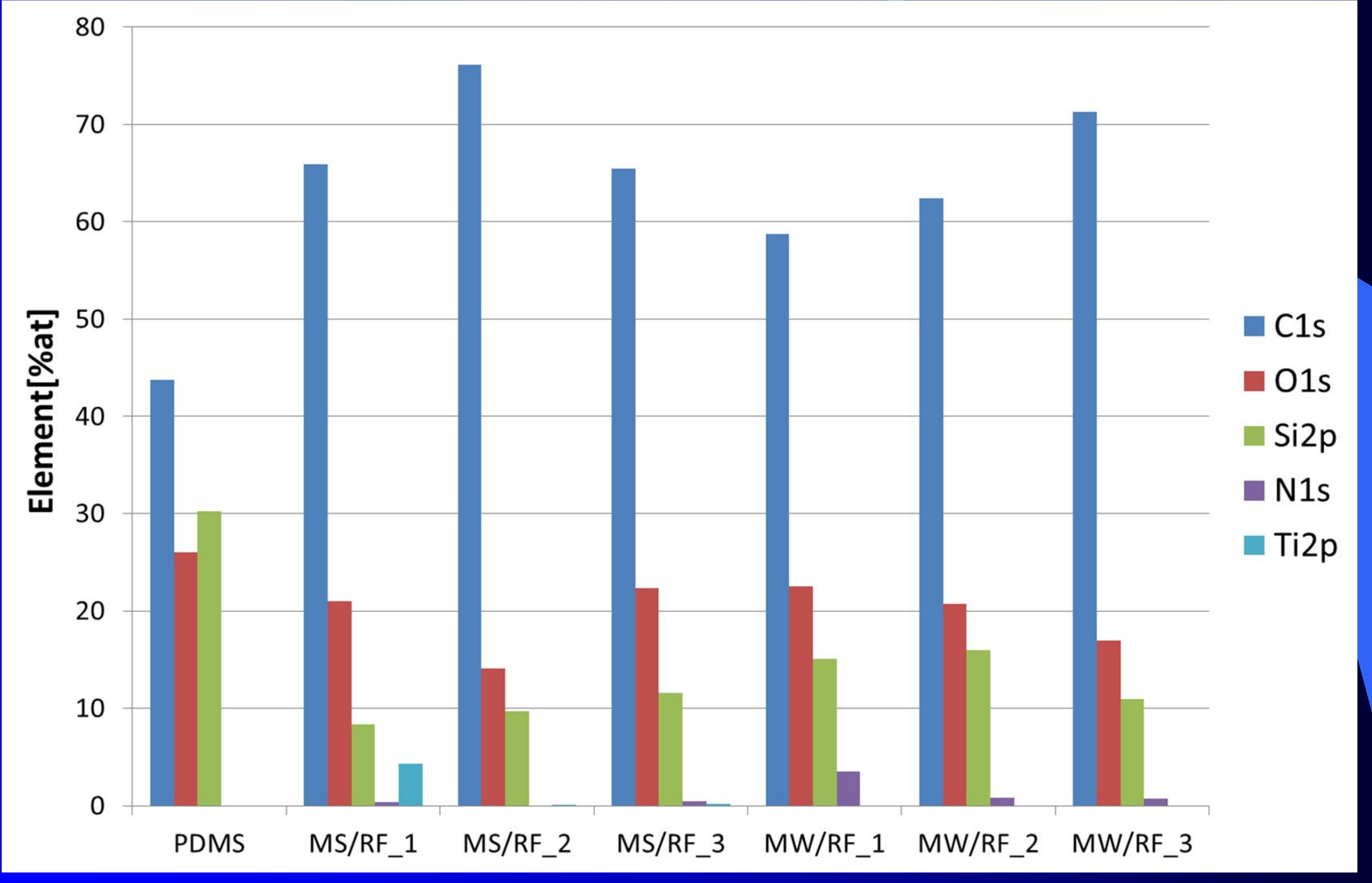
Wettability investigations -contact angle unmodified PDMS – 111,86º







X-ray photoelectron spectroscopy (XPS) results – elemental composition of the coatings



X-ray photoelectron spectroscopy (XPS) results

- Additionally analyzed peaks were decomposed and tested for the appearance of C=C type (284,4 eV), C-C type (285,4 eV) , C-O type (286,1 eV) and C=O type (288 eV) bonds.
- It was found that due to the application of oxygen plasma the share of C-C type bonds is increased for both technologies as well as the share of C=O type bonds. At the same time the opposite situation also for both technologies can be observed for C-O type bonds.

Conclusions

- PDMS samples have been deposited with different carbon coatings of different properties obtained with MW/RF PACVD and RF PACVD/MS techniques.
- The samples have been characterized with optical microscopy, AFM, Raman spectroscopy, XPS, also wettability and surface energy investigations were made.

Conclusions

- The results show that it is possible to modify the PDMS surface by changing its properties in a wide range (morphology, wettability, chemical composition) thanks to the different carbon coatings with different parameters of deposition.

Acknowledgments

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