crystal structure of $Pd_3Pb_2Te_2$ shows many structural similarities to the structure of shandite ($Ni_3Pb_2S_2$, *Rm*) and parkerite ($Ni_3Bi_2S_2$, *C*2/*m*). The phase $Pd_3Pb_2Te_2$ was described as a new mineral pašavaite [3].

**$Pd_3AgSe$**: Space group *Pa*, a = 8.63 Å, *V* = 642 Å$^3$ and Z = 8. The silver atom is surrounded by 12 palladium atoms, 3 selenium atoms and 1 silver atom. 12 of these atoms (9 Pd and 3 Se) form a truncated tetrahedron (Fig. 1). The other atoms (3Pd + 1 Ag) are located slightly above the centers of hexagonal faces of truncated tetrahedron. Two neighboring truncated tetrahedra share the hexagonal faces and thus form a basic structural unit of the $Pd_3AgSe$ structure. Similarly, as was mentioned for the isostructural $Au_3CaGa$ compound [4], the $Pd_3AgSe$ structure show comparable local structural motives as quasicrystals. Nevertheless, $Pd_3AgSe$ is a conventional crystalline compound. Therefore, it can be viewed as an approximant of quasikrystal [5].

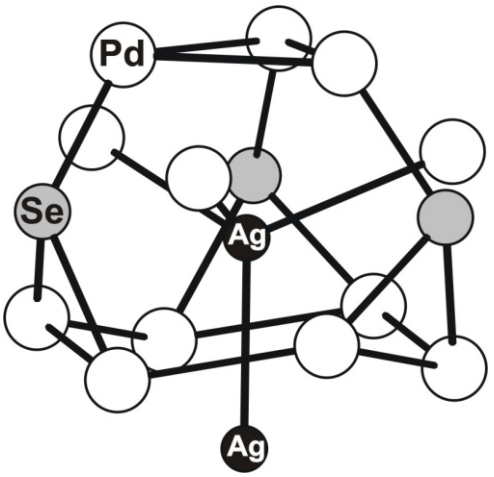


**Figure 1.** Coordination of Ag atoms in crystal structure of $Pd_3AgSe$. Note the truncated.

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**SL5**

## ZEOLITE MEMBRANE - MFI

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### Introduction

The oriented zeolite MFI (ZSM-5 and silicalite-1) layers were already synthesized on different supports and showed their attractiveness for the applications in the fields of membranes [1], microreactors, sensors and optoelectronic devices. For MFI zeolite membranes, the most favorable configuration would be a thin, fully intergrown *b*-oriented layer that would exhibit higher fluxes in comparison with *a*, *c* or random oriented layers. It is also known that the orientation of crystals essentially determines the crack formation during template removal, where different expansion/shrinkage properties of MFI crystallographic axes are responsible. It has been suggested that the preparation of *a*, *b*-oriented layer is more advantageous due to the template removal [2].

### Experimental

The direct hydrothermal *in*-situ syntheses of silicalite-1 layers were carried out on the surface of mercury, silicon wafer, non-porous and porous stainless steel TRUMEM™ supports. The crystallizations of MFI layers were performed in the temperature range of 155-165 C under static conditions with duration of crystallization between 1 to 20 hours.

The synthesized crystal layers were washed in an ultrasonic bath, dried and further characterized with scanning electron microscope (JEOL JSM 5500LV).

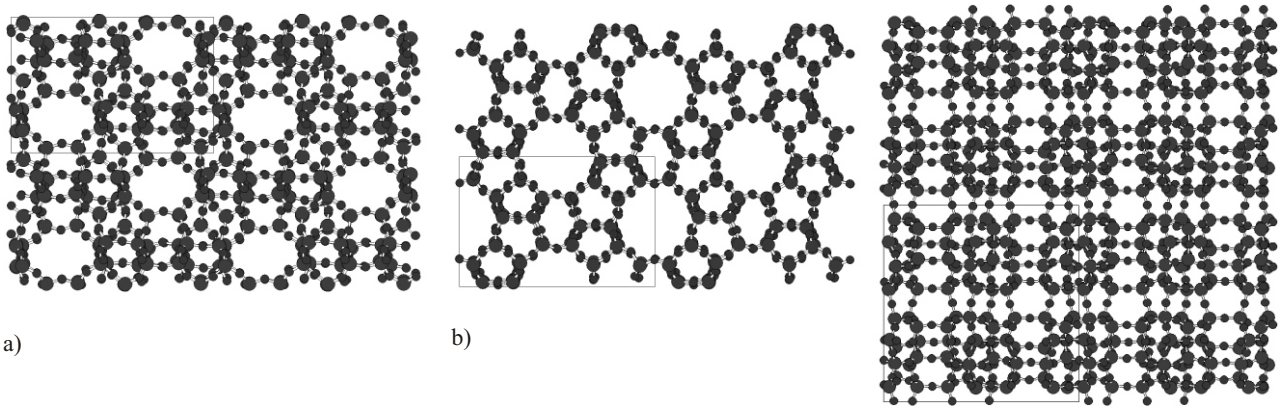
The X-ray diffraction pattern were measured on PANalytical X`Pert diffractometer in Brag-Brentano geometry. The Co radiation was used.

### Structure of MFI

The FMI zeolites crystallite in Pnma space group. The lattice parameters of ZSM-5 are following: *a* = 20.048, *b* = 19.884, *c* = 13.352, = 90°, lattice parameters of Silicalite-1 are a little varied. The structure contains relatively big pores which run through the structure in (100) and (010) directions. The three basic orientations ((100), (010), (001)) are shown in Fig. 1.

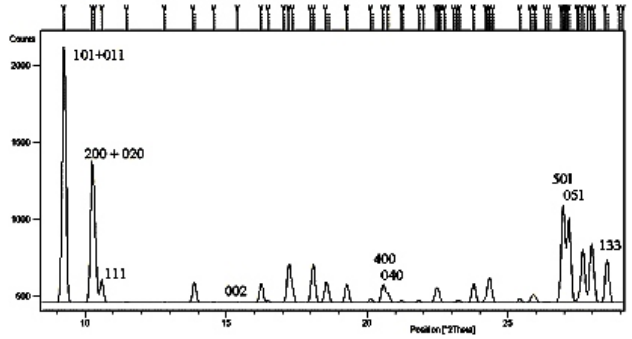
### Diffraction pattern

Besides the low angle region, the diffraction peaks are mostly overlapped because of relatively big orthorhombic unit cell. Some important higher angle diffractions can be

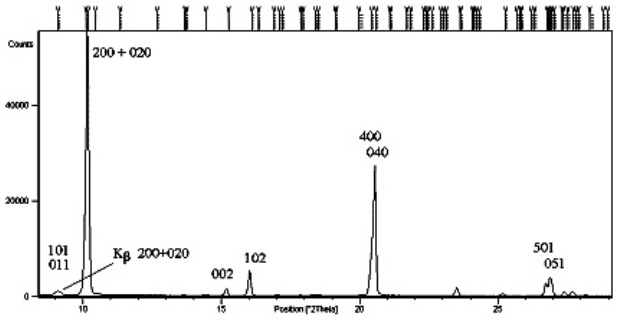


a) b) c)

**Figure 1.** The tree basic orientation of MFI framework, a) (100) direction, b) (010) direction, c) (001) direction. The four unit cells are plotted in every figure.




a)




b)

**Figure 2.** a) The theoretical non-textured diffraction pattern of silicalite-1. b) Strongly orientated silicalite-1 membrane (sample 17-08), a overlapping of (101,110) and K (200,020) diffraction is shown. In both cases Co radiation was used.

observed without undesirable overlapping in strongly textured samples. Fig. 2 shows a computed theoretical non-textured diffraction pattern of silicalite-1 with comparison of strongly textured one (sample 17-08 (floating)), where mainly the (100) and (010) orientation are observed.

The diffraction pattern 2b) shows also small (002) and (102) preferred orientation which corresponds to SEM observation in Fig 3.

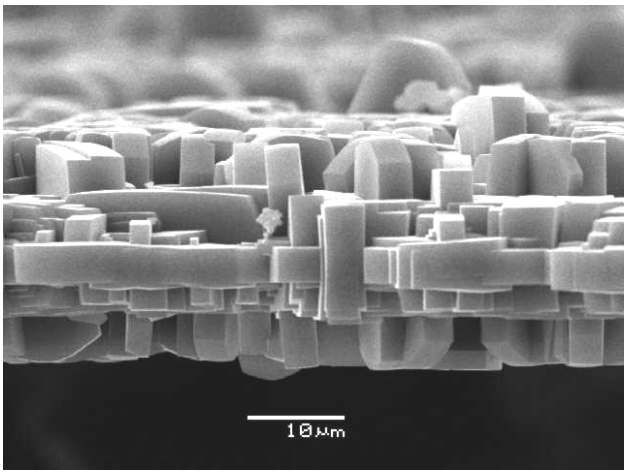



**Figure 3.** SEM micrograph of thin layer of sample 17-08 (floating).

## CPO indexes

The overlapping of diffraction peaks complicated the orientation dependent study of the film therefore *CPO*[X]/[Y] (crystallographic preferred orientation based on the [X] peak and the [Y] peak) was defined in the following way [3]:

$$CPO\ \frac{[X]}{[Y]} \quad \frac{I_S^{[X]} / I_S^{[Y]} \quad I_P^{[X]} / I_P^{[Y]}}{I_S^{[X]} / I_S^{[Y]}}$$

Where [*X*] and [*Y*] are chosen crystalografic direction, $I_S$ is intensity of sample and $I_P$ is intensity of non-textured powder. If CPO index is equal to 1 then the sample has only [X] orientation, for *CPO* = 0 is the sample non-textured and for negative CPO index the sample prefer [Y] orientation.

## Results and discusion

Several CPO indexes was calculated and compared. Two of them are in Tab. 1. The first one CPO [200+020]/[133] enable distinguish between (100+010) and "random" orientation. Due to resolution of X-ray diffractometer the

(200) and (020) reflextion can not be measured separable and are consider as one line. The second one CPO index [0 10 0]/[10 0 10] distinguish between (100) and (010) orientation. Because of overlapping with several others diffraction the second one index can be used only when the first one is close to one, in this case the intensity of (0 10 0) and (10 0 0) diffraction are much more bigger than the other reflection diffracting to this area.

## Conclusions

The CPO indexes given by X-ray diffraction compared with SEM micrograph helps in understanding the grooving of MFI thin layers and their preferred orientation on the surface of studied supports.

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### Acknowledgements

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**Table 1.** CPO indexes for several samples

| Sample | CPO | |
|---|---|---|
| | [200+020]/[133] | [0 10 0]/[10 0 0] |
| 17-08 (floating) | 0.99 | 0.33 |
| 3z - 08 | 0.84 | 0.68 |
| 16-08 | -18 | 0.70 |

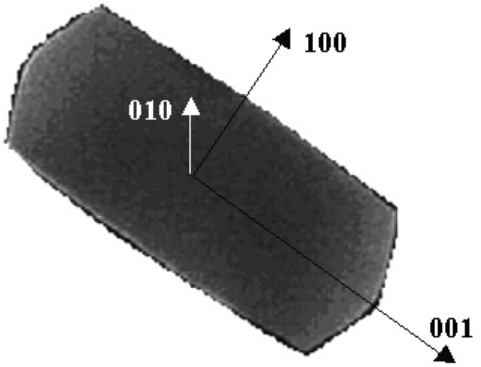


**Figure 4.** The relation between macroscopic grain shape and crystallographic directions.

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**SL6**

# CAN TRYPTOPHAN ENHANCE PROTEIN CRYSTALLIZABILITY?


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X-ray crystallography is a powerful tool in protein tertiary structure determination. For this method the preparation of well-diffracting crystals is inevitable, which usually represents the most problematic and rate-limiting step. The process of protein crystallization is influenced by many parameters and protein itself, its purity, homogeneity and properties, namely the ability of forming crystals, is the most important one. Many attempts were done with the aim to improve protein crystallizability and crystal quality. Protein modifications oriented on improvement in protein homogeneity, solubility, stability and/or crystal quality represent fruitful approach to solving the problem of high-quality crystals preparation. Besides chemical modifications of proteins, their truncations, deglycosylation, limited proteolysis, the mutations of individual amino-acid residues become very popular. A lot of examples of successful mutations can be found in the literature, however, no universal recipe exists and/or no absolute rules can be extracted. Hence the main question - which to which amino-acid should be mutated - is still valid.

Analysis of intermolecular contacts in the crystals of several different proteins crystallized in our laboratory revealed that tryptophans located at the molecular surface can form a number of intermolecular contacts in the crystals. The large size and the mixed hydrophobic/hydrophilic character of tryptophan side chain allow various kinds of interactions with a number of residues simultaneously. Tryptophans are usually mostly buried in the molecules, rather rarely they are exposed to the solvent. It is known that tryptophan residues have an important role in stabilizing the protein structures. Recently it has been observed that tryptophans located on the molecular surface can indicate the binding site.

These facts together with the above mentioned observations inspired us to analyze the role of tryptophan in formation the intermolecular contacts in crystals. For this, the systematic analysis of protein X-ray structures deposited in