

elastic or plastic. Both milling and grinding can be carried out in either up-cut or down-cut modes which differ significantly in the incidence of machining forces and, hence, in the mechanism of material removal. If different cooling environments are in usage, effect of temperature fields can be studied as well [2]. The goal of the performed X-ray diffraction analysis was to obtain detailed information about the state of macroscopic and microscopic residual stress, and mean coherent scattering domain sizes in surface layers of metals which were subjected to either up-cut or down-cut mode of either grinding or milling with various cutting and cooling conditions.

[1] Chen, X.; Rowe, W. B.; McCormack, D. F. *J. Mater. Process. Technol.* **107**, **2000**, 216 – 221. [2] Pala, Z.; Ganev, N. *Mater. Sci. Eng. A* **497** **2008**, 200 – 205.

**Keywords:** real structure; mechanical treatment; surface quality

#### FA2-MS06-P12

**Defects Study of Polymorph B Enriched Zeolite Beta.** Daliang Zhang<sup>a,b</sup>, Junliang Sun<sup>a,b</sup>, Sven Hovmöller<sup>a</sup>, Xiaodong Zou<sup>a,b</sup>. <sup>a</sup>*Structural Chemistry, Stockholm University, Stockholm, Sweden.* <sup>b</sup>*Berzelii Centre EXSELENT on Porous Materials, Stockholm University, Stockholm, Sweden.*

E-mail: [daliangz@struc.su.se](mailto:daliangz@struc.su.se)

High silica zeolite Beta is one of the industrially important zeolites due to the catalytic properties in fluid catalytic cracking, and organic synthesis and separation. Zeolite Beta always exists as an intergrowth of two end-member structures of polymorph A (\*BEA,  $P4_122$  or  $P4_322$ ) and polymorph B ( $C2/c$ ). Stacking faults dominate all over the Beta crystals and even some large pore defects were observed<sup>[1-2]</sup>. It is important to study the defects of zeolite Beta in order to improve the synthesis and investigate new properties and applications of the materials.

A sample of typical polymorph B enriched zeolite Beta was provided by Corma and co-workers, and the synthesis procedure was described in Reference [3]. The polymorph B enriched zeolite Beta crystals show a wedge-shaped rod-like morphology. The surfaces of the crystals are not smooth but ridge-like. Generally speaking, the surfaces of most perfect crystals should be smooth; however they are not in this case. The property of crystal surfaces very often plays a major role for the properties of a material, especially when the surface structure is different from that of the bulk crystals. So the study of the ridges on the surfaces of the polymorph B enriched zeolite Beta sample is very important.

HRTEM images were taken along the [1-10] direction. A TEM image in figure 1 shows that the stacking of 12-ring channels follows the ABC or CBA type stacking mode of polymorph B. These two types of stacking can be considered as two different twin components. It is very obvious that the ridge at centre shows a different stacking mode compared with the other areas. The CBA type stacking in the center ridge and the ABC type stacking for the other areas belong

to different polymorph B twin components, all of them grow from the same crystal  $ab$  plane. Since their frameworks do not fit each other, so they have to produce large pore defects in between. This significantly increases the lattice energy and may speed up the dissolving. Due to the symmetry of the polymorph B structure, there should be also stacking faults in the orthogonal [110] direction. The stacking behavior at the crystal surface should be also the same. The ridges can be observed along both two directions.

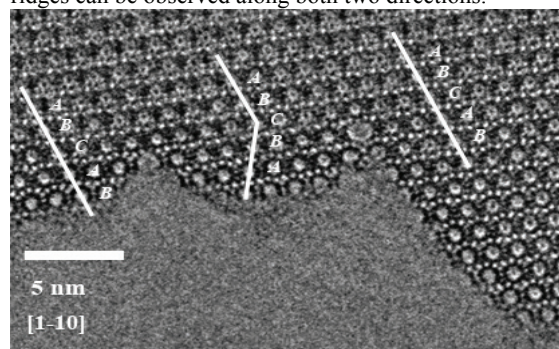


Figure 1. A HRTEM image showing ridges with different stacking modes. Large pore defecta are observed between the ridges

[1] J.M. Newsam, M.M.J. Treacy, W.T. Koetsier, C.B. de Gruyter, *Proc. R. Soc. Lond. A.* **1988**, 420,375-405. [2] Paul A. Wright, Wuzong Zhou, Joaquin Pérez- Pariente and Mar Arranz *J. Am. Chem. Soc.* **2005**, 127,494-495. [3] Corma, A., Moliner, M., Cantín, A., Díaz-Cabañas, M.J., Jordá, J.L., Zhang, D.L., Sun, J.L., Jansson, K., Hovmöller, S., Zou, X.D. *Chem. Mater.* **2008**, 9, 3218-3223.

**Keywords:** zeolite beta; HRTEM; defects

#### FA2-MS06-P13

**Nano Properties and X-ray Crystallographic Analysis of Hard Coatings Synthesized by PVD and IBAD.** Branko Škorić<sup>a</sup>, Damir Kakaš<sup>a</sup>, Gregory Favaro<sup>b</sup>, Aleksandar Miletić<sup>a</sup>. <sup>a</sup>*University of Novi Sad, Serbia.* <sup>b</sup>*CSM Instruments SA, Peseux, Switzerland.*  
E-mail: [skoricb@uns.ns.ac.yu](mailto:skoricb@uns.ns.ac.yu)

In the paper are presented characteristics of hard coatings, type TiN, produced by classic technology PVD (physical vapour deposition) and IBAD (ion beam assisted deposition). The synthesis of the TiN film by IBAD has been performed by irradiation of Ar ions at 1000 eV. Such coatings exhibit improved mechanical properties in comparison with TiN deposited by PVD. The three basic points that are considered fundamental to studies of friction are the surface area and nature of the intimate asperity contacts, the surface adhesion and shear strength, and the nature of deformation and energy dissipation occurring at the asperity junctions. The optimization procedure for coated parts could be more effective, knowing more about the fundamental physical and mechanical properties of a coating, their interdependence and their influence on the wear behaviour. The morphology and characteristics of surface layer structure as well as important properties were

investigated This paper describes the successful use of the nanoindentation technique for determination of hardness and elastic modulus. In the nanoindentation technique, hardness and Young's modulus can be determined by the Oliver and Pharr method, where hardness (H) can be defined as:  $H = P_{\max} / A$ , where  $P_{\max}$  is maximum applied load, and A is contact area at maximum load. In nanoindentation, the Young's Modulus, E, can be obtained from:  $1/E_t = (1-\nu_i^2)/E + (1-\nu^2)/E_i$ , where  $\nu_i$  = Poisson ratio of the diamond indenter (0.07) and  $E_i$  = Young's modulus of the diamond indenter. Therefore, in recent years, a number of measurements have been made in which nanoindentation and AFM have been combined. Indentation was performed with CSM Nanohardness Tester. The results are analyzed in terms of load-displacement curves, hardness, Young's modulus, unloading stiffness and elastic recovery. The nanohardness of coating measured by Berkovich indenter is about 42.4 GPa. The analysis of the indents was performed by Atomic Force Microscope. The composition of the films (nitrogen to metal ratio) was determined by energy-dispersive X-ray analysis (EDAX). The determination of phases was realized by X-ray diffraction. The stress determination follows the conventional  $\sin^2\psi$  method, using a X-ray diffractometer. The analyzed AE signal was obtained by a scratching test designed for adherence evaluation. AE permits an earlier detection, because the shear stress is a maximum at certain depth beneath the surface, where a subsurface crack starts. Characteristics of surface layer structure as well as important properties were investigated. A variety of analytic techniques were used for characterization, such as scratch test, calo test, SEM, AFM, XRD and EDAX.

[1] J.C.A. Batista, C. Godoy, A. Matthews, A. Leyland, *Surface Engineering*, **2003**, 9, 37. [2] Pharr, G.M., Oliver, W.C., Brotzen, F.R., *J. Mater. Res.*, **1992**, 7, 613.

**Keywords: atomic force microscopy; crystal structure; residual stress**