

**KEEMIA- JA MATERJALITEHNOLOOGIA TEADUSKOND
MATERJALIUURINGUTE TEADUSKESKUS
TEADUS- JA ARENDUSTEGEVUSE AASTAARUANNE 2013**

1. Struktuur

**Materjaliuuringute teaduskeskus, Centre for Materials Research
Keskuse juhataja Urve Kallavus**

- Materjaliuuringute õppetool, Chair of Materials Research, Urve Kallavus
Materjaliuuringute teadus- ja katselaboratoorium, Laboratory for Materials Research

2. Teadus- ja arendustegevuse (edaspidi T&A) iseloomustus

2.1 Struktuuriüksusesse kuuluvad uurimisgrupid (*kõik uurimisgrupid näidatakse aruandes eraldi, järgides alltoodud ülesehitust*).

Uurimisgrupi nimetus (eesti ja inglise keeles) ja juhi nimi

- uurimisgrupi teadustöö kirjeldus (*inglise keeles*);
- uurimisgrupi aruandeaastal saadud tähtsamad teadustulemused (*inglise keeles*);
- uurimisgrupi kuni 5 olulisemat publikatsiooni aruandeaastal.

Main research directions and results:

1. Investigation of thin films, multilayered structures, fine particles and powders; feature analysis and crystallographic orientation of fine structures (Valdek Mikli, M.S.Nat.Sci. PhD nat.Sci).

The structure and process of ZnO:In thin films grown of spray solution on glass substrates by chemical spray method was investigated. The solution spray rate influences the surface morphology, grain size, film thickness, and electrical and optical properties. According to SEM and AFM studies, sharp-edged pyramidal grains and canvas-resembling surfaces are characteristic of films grown at spray rates of 0.5 and 3.3 mL/min, respectively. carrier concentration in slowly sprayed films is likely due to the indium oxidation.

In the study of surface defects in 4H-SiC Schottky diodes using a scanning Kelvin probe the increased leakage currents in Schottky diodes with an integrated p-n structure was investigated. The SEM investigations revealed that the dislocations penetrating from the substrate into the epitaxial layer play a critical role in increasing leakage currents.

The formation of differently shaped crystallites (spherical particles and plate-like grains) in the Calcium Phosphate Nanocomposites. with an average grain size ranging between 1 and 2 μm was determined from SEM measurements. The roughness of thin films measured by AFM are associated with changes in phase composition of CHA-TCP coating.

In $\text{La}_{0.6}\text{Pb}_{0.4}\text{MnO}_3$ polycrystalline sample synthesized by a solid state reaction in oxygen atmosphere. observed 0.5–1 mm size cubic-shape LaPbMnO grains averagely consisted from 5 to 12 (93 (7) nm in size) crystallites. After the second pelletization and sintering processes, the cubic-shape grains of the sample were found to be closely packed each other. For this reason, thinner grain boundaries and lower energy barriers for the charge carriers can be expected in such a sample.

3. Investigation of hard sintered materials, their formation and structural characterization, metallographic structures, computer-aided feature analysis (Mart Viljus, M.S.Nat.Sci. PhD.Nat.Sci).

High-velocity oxy-fuel (HVOF) sprayed composite powder TiCNiMo and $\text{Cr}_3\text{C}_2\text{-Ni}$ cermet particles reinforced self-fluxing alloy matrix (FeCrSiB) coatings were studied. Sprayed coatings exhibited a structure with a number of defects, like voids and cracks, whereas the latter were more pronounced in reinforced coatings. Cermet particles were deformed at the impact with the substrate, deformation was more remarkable in the case of the TiC-NiMo particles.

Close look-up of TiC-NiMo powder particles with some iron impurities showed only few cracks in them, compared to $\text{Cr}_2\text{C}_3\text{-Ni}$ cermet powder particles, which are full of defects. This indicates to higher ductility of raw material used in experiment. Moreover, the possibility of deformation during high velocity spraying later is lower due to the absence of inter cracks compared to $\text{Cr}_2\text{C}_3\text{-Ni}$ powders and in comparison on spraying of composite powders, consisting of WC-Co hard particles.

The results of SEM-studies showed that sintering techniques have a slight influence on the microstructure of TiC -based cermets. The microstructure of alloys sintered at optimal parameters (sinterhipped at 1430°C) that ensure maximized performance is featured by higher homogeneity (low porosity, uniform distribution of phases).

Processing and properties of bulk ultrafine-grained pure niobium were investigated. There, the Nb with an UFG microstructure has an increased COF and a higher specific wear rate as compared to the as-cast sample.

Microstructure Evolution of WC-TiC-Co Cemented Carbides During Reactive Sintering was investigated. The evolution of the microstructure during solid and liquid state sintering are exhibited. The microstructure of reactive sintered WC-TiC-Co cemented carbides, with optimal carbon content in initial powder mixture, is fine-grained and homogeneous.

Different process methods and parameters together with different amount of additives were used to fabricate WC-Ni-ZrO_2 hardmetals with mechanical properties aiming at improved performance under erosive wear. XRD observation showed the presence of tetragonal zirconia in the cermet matrix after processing.

The micro-structural and compositional properties of the precursor stacked and selenised films of $\text{Cu}_2\text{ZnSnSe}_4$ (CZTSe) were characterised using scanning electron microscopy/energy dispersive spectroscopy, X-ray diffraction and Raman spectroscopy. Selenisation for 60 min resulted in highly crystalline CZTSe films with a grain size of 1.5–4 μm .

4. Investigation of lignocellulosic materials (wood, cellulose, paper, structure timber), natural and man-made stone, and their degradation by natural and artificial causes; objects of cultural heritage, archaeology, art and their conservation problems (Urve Kallavus, Dr.Habil.Chem.). Investigation of the indoor climate influence to the biodeterioration of materials.

In the study of cellulose engineering in nanoscale, with main attention on chemical modification and related kinetics and surface modification and characterization for potentially commercial materials different methods of obtaining nanocellulose based hydro- and aerogels were worked out. This project is compliant with the eligible themes specified in the regulation of the measure – nano- and wood materials.

The microbiological damage of the constructive materials and quality of indoor climate was analysed in Estonian Manor schools, rural houses and medieval sacral buildings. The damage types were identified and classified.

Deterioration plays an important part in the life cycle of infrastructure systems. Among all causes of deterioration - aging, chloride ingress, etc. the action of live organisms has shown to be essential. This phenomenon accelerates other processes that may lead to unacceptable structural performance or cause failure e.g., corrosion, cracking.

Biodeterioration-related structural problems have substantial cost a year in infrastructure maintenance and repair. This is particularly relevant given the large amount of existing infrastructure that has been exposed to aggressive environments for long periods of time. A description of the main organisms that affect each material and the associated biodeterioration mechanisms were described.

2.1.1 Paremad publikatsioonid

Beganskiene, A.; Stankeviciute, Z.; Malakauskaite, M.; Bogdanoviciene, I.; Mikli, V.; Tõnsuaadu, K.; Kareiva, A. (2013). SOL-GEL APPROACH TO THE CALCIUM PHOSPHATE NANOCOMPOSITES. *In: Nanostructured Materials and Nanotechnology VII: The 37th International Conference on Advanced Ceramics and Composites, Florida, USA, January 27 - February 1, 2013. (Toim.) S. Mathur, F. Hernandez-Ramirez, S. Kirihara and S. Widjaja.* John Wiley & Sons Ltd, 2013, (Ceramic Engineering and Science Proceedings ; Volume 34, Issue 7), 1 - 11.

Kübarsepp, J.; Klaasen, H.; Tšinjan, A.; Juhani, K.; Kollo, L.; Viljus, M. (2013). Influence of pressurized sintering on performance of TiC-based cermets. Hussainova, I. (Toim.). *Engineering Materials and Tribology* (56 - 61). Trans Tech Publications Ltd.

2.2 Loetelu struktuuriüksuse töötajate rahvusvahelistest tunnustustest.

2013.a. rahvusvahelisi tunnustusi ei saadud.

2.3 Loetelu struktuuriüksuse töötajatest, kes on välisakadeemiate või muude oluliste T&A- ga seotud välisorganisatsioonide liikmed.

Valdek Mikli – European Microbeam Analysis Society (EMAS), liige

Urve Kallavus - Kaunas University of Technology, Academy of Sciences of Lithuania
"Materials Science - Medziagotyra" Editorial Board, liige.

2.4 Soovi korral esitatakse aruandeaastal saadud T&A-ga seotud tunnustused (va punktis 2.3 toodud tunnustused), ülevaade teaduskorralduslikust tegevusest, teadlasmobiilsusest ning hinnang oma teadustulemustele.