KEEMIA- JA MATERJALITEHNOLOOGIA TEADUSKOND
MATERJALIUURINGUTE TEADUSKESKUS
TEADUS- JA ARENDUSTEGEVUSE AASTAARUANNE 2012

1. Keskuse 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. Keskuse teadus- ja arendustegevuse (edaspidi T&A) iseloomustus

(NB! punktid 2.1- 2.6 täidab struktuuriüksus)

2.1 struktuuriüksuse koosseisu kuuluvate uurimisgruppide

2.1.1 teadustöö kirjeldus *(inglise keeles*);

Main research directions:

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).

2. 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).

3. 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, archaelogy, art and their conservation problems (Urve Kallavus, Dr.Habil.Chem.).Investigation of the indoor climate influence to the biodeterioration of materials.

2.1.2 aruandeaastal saadud tähtsamad teadustulemused *(inglise keeles).*

Development and investigation of multiphase tribomaterials (T162)
Within the project the microstructure of a number of different materials was investigated and characterised with the means of scanning electron microscopy and X-ray microanalysis. The list includes ZrO_2-doped cermets, Al-Al_2O_3 nanofibers, TiC based cermets with steel binder (with different sintering technology), recycled WC-Co hardmetals, reactive-sintered TiC-NiMo cermets, Ni-based superalloys and nanocrystalline diamond films.

After sliding tests of the nanocrystalline diamond films (NCD), ripple patterns were found on the wear scar surfaces. These patterns seem to be characteristic for the given type of films, forming in the centre of the wear track regardless of the couter-body. Investigating the Ni-based superalloys, X-ray microanalysis was successfully combined with the nanoindentation technique to determine both the mechanical properties and the composition in the same microscopical location. The mechanism of abrasive impact wear of WC-Co and TiC-NiMo cermets was investigated and compared, finding that the mechanism of both materials consists of plastic deformation of the binder phase and brittle cracking of carbide grains and carbide network.

Hard coatings and coating technology (T191)
In this project different materials and methods were applied to increase the wear and corrosion resistance of materials and industrial parts. Addition of basalt to the FeCrSiB HVOF sprayed coating was studied.

Self-fluxing NiCrSiB and FeCrSiB powders were supplied from Höganäs AB, hardmetal TiCNiMo and Cr2C3-Ni powders were obtained by disintegrator milling at Tallinn University of Technology. PVD single layer TiN (I generation), multilayer (Ti,Al)N-ML (II generation), gradient (Al,Ti)N-G and multilayer nanocomposite FiVIcR (both – III generation) coatings on the nitrided low alloy steel 42CrMo4 are analysed.

PVD coatings were used in a variety of different applications to reduce the wear of tools.
Study the properties of cost-effective self-fluxing alloys based on composite powders and recycled cermets and to examine the properties of thermally sprayed (HVOF) coatings from composite powders based on iron self-fluxing alloy and recycled cermet powders (Cr_2C_3-Ni and WC-Co).

Recycling of materials is becoming increasingly important. Different recycling technologies for composite materials (oxidation, milling, remelting etc) are widely used. The project studies hardmetal/cermet powders produced by mechanical milling technology. The following composite materials were studied: Cr3C2-Ni cermets and WC-Co hardmetal.

For corrosion resistance the influence of substrate microstructure on iron-zinc alloy layer formations was studied.

In the automotive industry, the heat treatment of strip steel safety components is a crucial and highly responsible process in terms of quality. By the help of this project the part austenitization in the industrial furnace was investigated.

Addition of macroparticles of basalt into FeCrSiB-alloy based HVOF sprayed coating has resulted in the reduction of wear resistance under most of the conditions where dynamic loading takes place. Addition of basalt is favourable in continuous sliding conditions with low velocity, low force and with steel counter body that may form surface layer enriched by basalt inclusions.

FeCrSiB alloy based coatings were less dense due to microcracking, induced by the differences in the value alloy and hardmetals. Addition of hardmetal s of thermal expansion coefficients of the *FeCrSiB* particles allows to increase the microhardness of self-fluxing alloy coatings by 1.1 – 1.2 times in comparison with those without the addition of hardmetal particles.

The PVD III generation (Al,Ti)N-G coating demonstrated the best cracking resistance (III class of the cracking evaluation criteria) in comparison with the other coatings (VI class of the cracking evaluation criteria) due to the gradient structure, having the lowest residual stress and thus the best adhesion to the substrate. The 1.6 – 1.7 times higher impact wear resistance was demonstrated by the PVD I generation TiN coating in comparison with the other studied coatings due to the highest modulus of elasticity.

The grindability of hardmetal/cermet using milling by collision in disintegrator was studied and the influence of particle size reduction on specific energy of treatment was clarified. The technology of producing hardmetal/cermet powders from used (recycled) hardmetal consisted of preliminary crushing and mechanical size reduction of hardmetal parts and final milling of pre-treated product by collision in the disintegrator mill. The dependence of grindability (decrease in particle size) on the specific energy of treatment was studied. Hardmetal powders production with a predicted particle size is available.

The thickness of the coating and iron-zinc reactivity is related with the microstructure of the substrate. Ferrite grain size does not affect the morphology of zinc coatings. Differences in the size of carbides influence the coating formation. The higher the hardness of substrate, the smaller the carbide grain size and during hot dip galvanizing thinner coating is formed.

By the selection of austenitization parameters for an industrial furnace, the following inputs must be considered: material chemical composition, furnace temperature, feed rate, layer thickness and heating time. The laboratory test indicated that the wider and narrow sections of the part will austenitisize unequally in time. The geometrical aspect such as wider cross-section of the safety belt tongue requires higher temperature or longer soaking time to achieve uniform hardness. The greater the overheating step from Ac3 transformation line is, the less importance has the soaking time for homogeneous austenite formation.

Synthesis and structure investigation of multifunctional materials (VA428)
Investigation of different types of ceramic materials, like superconductors and magnetic materials.

A $La_{0.6}Pb_{0.4}MnO_3$ polycrystalline sample was synthesized by a solid state reaction in oxygen atmosphere. Thus obtained polycrystalline bulk $La_{0.6}Pb_{0.4}MnO_3$ possessed relatively high magneto-resistance effect (32.3% at4K and 10 kOe).The saturation magnetization M_{sat} (75.8 emu/g and 48 emu/gat 4K and 300K) and the spontaneous magnetization Ms (66.2 emu/g and 26 emu/g at 4K and 300K) values were of the same order as those of the single crystals. At 300K the resistance R showed a small progressive decrease in cyclic magnetic field H which resembled a training phenomenon in magnetic fields.

Thin films
Opto-electronical thin films were prepared by different techniques.
At first ZnO nanorods were prepared by a spray pyrolysis technique on both as-received and etched Indium TinOxide (ITO)/glass substrates. Secondly, characterization of polycrystalline CdTe thin films grown directly on glass, SnO2-coated glass, and CdS/SnO2/glass at relatively low temperatures by employing the close space sublimation technique (CSS). Thirdly the sol–gel chemical spray pyrolysis method was used to deposit samarium and nitrogen co-doped TiO2 films onto a quartz substrate using pulsed spray solution feed.

ZnO nanorods were deposited directly onto ITO/glass substrates by a simple, fast and cost-effective spray pyrolysis method. It was demonstrated that the ZnO nanorod growth in the spray pyrolysis process on transparent conductive ITO electrodes is largely controlled by the amount of nucleation centers (grain boundaries, intersections) on the substrate.

Using the CSS technique, polycrystalline CdTe thin films were grown on substrates of three kinds: glass, SnO_2/glass, and CdS/SnO_2/glass. According to the XRD, SEM, and the absorption spectra analysis, the CdTe/CdS/SnO_2/glass thin films exhibit high crystallinity and reproducibility in comparison to other CdTe films grown on glass and SnO_2/glass. All the as-deposited CdTe samples are textured with the preferential orientation in the [111] direction.

The results of the study indicate that sol–gel chemical spray pyrolysis is a promising method for preparation of rare earth and nitrogen co-doped TiO_2 nanocrystalline thin films. The N and Sm co-doped TiO_2 films exhibit anatase structure, independent of the doping level. According to AFM, the undoped TiO_2 film consisted of agglomerates with a size of 30–200 nm. N-doping (25 at%) transformed the agglomerates into individually distinctive grains with a size of ca. 30 nm. Sm doping (5 at%) caused a significant decrease in the average diameter of the agglomerates to ca. 100 nm. The Sm and N co-doped TiO_2 films exhibited surface features characteristic of both the Sm-

doped and N-doped TiO_2 films. The RMS roughness of the undoped TiO_2 film was 1.7 nm; doping lead to the formation of smoother films with an RMS roughness of 0.9–1.4 nm.

Residual stresses

Different coatings were studied. As in the case of many coating technologies, residual stresses in deposits can lead to a detrimental effect including a decrease in fatigue strength, cracking under service conditions, and delamination.

Brush-plated gold and silver coatings are mainly used for decorative (e.g. copper domes of churches, adornments, etc) or electric applications. Today, nickel-hardened gold layers are used in electric and electronic equipment, as contacts for high-reliability separable connectors, switches and in other applications, where hardness and resistance to mechanical wear are key properties for achieving reasonable service life in compliance with reliability standards. Considering the cost, gold is sometimes replaced by silver in manufacturing electronic components.

Residual stresses in freshly plated coatings represented high tensile stresses that were significantly influenced by current density for gold coatings but not significantly influenced by current density for silver coatings. Higher deposition temperatures caused temperature stresses whose proportion in residual stresses was insignificant, accounting for not more than 2.5 %. Brush-plated coatings have a fine crystalline nano-structure which is caused by the high deposition current and by the short-term growth of formed crystals.

The values of residual stresses in coatings decreased markedly and after two months they were two times lower for silver coatings and to some degree lower for gold coatings compared to the respective values obtained for freshly plated coatings. Residual stresses stabilized after 1300 and 400 days for gold and silver coatings, respectively.

Diffusion welding for electronics

This study presents the characteristics of solid-phase interaction of aluminium with silicon carbide in the process of creating a diffusion welding of contacts to semiconductor devices.

The results clearly demonstrate that the interaction of the metal with the semiconductor material during the process of diffusion welding is individual. Thus, the interaction of aluminium with silicon crystals is accompanied by anisotropic solid-phase etching, followed by solid-phase epitaxy in the cooling. In the case of silicon carbide solid-phase etching is looks like isotropic, followed by precipitation of carbon on the contacting surfaces.

Hydroxy-Apatite doping

The apatites were modified by transition metal ions.

Apatites are of considerable interest in numerous research areas including synthesis in organic chemistry. The mechanism of metal ions sorption on apatite depends on solution equilibrium pH value as well as on ion nature. M^{3+} ions lead in aqueous solution always to a new solid phase at the surface of apatite through a dissolution-re-precipitation process. Results concern the sorption mechanism of M^{2+} (Zn^{2+}, Cu^{2+}, Cd^{2+}) and M^{3+} (Ru^{3+}, Fe^{3+}, La^{3+}) ions on apatites of various composition in aqueous solutions in pH region 4-10. Some ions, mainly M^{2+} type can change Ca^{2+} in apatite crystal surface, whereas M^{3+} ions lead to a new solid phosphate phase at the surface of the apatite through a dissolution- re-precipitation process.

Smart aerogels based on the nanostructured wood Cellulose - SMaCell (AR12138)

Project started in September, 2012. he overall goal of the project is to study the cellulose engineering in nanoscale, particularly in hydro- and aerogel formation using highly fibrillated

cellulose, with main attention on chemical modification and related kinetics and surface modification and characterization for potentially commercial materials. This project is compliant with the eligible themes specified in the regulation of the measure – nano- and wood materials.

The microbiological deterioration of constructive materials

The microbiological damage of the constructive materials and quality of indoor climate was analysed in Estonian apartment houses, rural houses and sacral buildings. The damage types were 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.

2.2 Uurimisgrupi kuni 5 olulisemat publikatsiooni läinud aastal.

Dedova, T.; Oja Acik, I.; Krunks, M.; Mikli, V.; Volobujeva, O.; Mere, A. (2012). Effect of substrate morphology on the nucleation and growth of ZnO nanorods prepared by spray pyrolysis. Thin Solid Films, 520(14), 4650 – 4653.

Viljus, M.; Pirso, J.; Juhani, K.; Letunovitš, S. (2012). Structure Formation in Ti-C-Ni-Mo Composites during Reactive Sintering. Materials Science (Medžiagotyra), 18(1), 62 - 65.

Potlog, T.; Spalatu, N.; Maticiuc, N.; Hiie, J.; Valdna, V.; Mikli, V.; Mere, A. (2012). Structural reproducibility of CdTe thin films deposited on different substratesby close space sublimation method. . Physica Status Solidi A - Applications and Materials Science, 209(2), 272 - 276.

2.3 Loetelu struktuuriüksuse töötajate rahvusvahelistest tunnustustustest.
2012.a. rahvusvahelisi tunnustusi ei saadud.

2.4 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" Ediorial Board, liige

2.5 Aruandeaasta tähtsamad T&A finantseerimise allikad.

- T162 „Mitmefaasiliste tribomaterjalide arendamine ja tehnoloogia“
- T191 „Kõvapinded ja pinnatehnika“
- RP12112 Eesti Pühakodade kivimüüride niiskuskahjustused ja sooldumine
- AR12138 Smart aerogels based on the nanostructured wood cellullose – SmaCell.

2.6 Soovi korral lisada aruandeaastal saadud T&A-ga seotud tunnustusi (va punktis 2.3 toodud tunnustused), ülevaate teaduskorralduslikust tegevusest, teadlasmobiilsusest ning anda hinnang oma teadustulemustele.

Teaduseskuses jätkati edukalt eelnevate aastate teadustööd. Alustati uute projektidega SA Archimedese materjalitehnoloogia programmi raames. Välislepingutest osavõtt oli stabiilne. Jätkuvalt on kõrge publikatsioonide arv.

	2012	2011	2010	2009
Publikatsioonid (CC)	38 (13)	25 (15)	21 (8)	32(13)
Kaitsmised	2 magistritööd	0	1 magistritöö	1 magistritöö

Keskuse Nõukogu otsustas hinnata keskuse 2011.a. teadustöö tulemused hindega “4”.

2.7 Keskuse teadus- ja arendustegevuse teemade ja projektide nimetused *(Eesti Teadusinfosüsteemi, edaspidi ETIS, andmetel)*

- Haridus- ja Teadusministeerium

– sihtfinantseeritavad teemad:
SF0140062s08 „Mitmefaasiliste tribomaterjalide arendamine ja tehnoloogia“ (T162) (01.01.08 - 31.12.13)
SF0140091s08 „Kõvapinded ja pinnatehnika“ (T191) (01.01.08 - 31.12.13)
– baasfinantseerimise toetusfondist rahastatud projektid (sh TTÜ tippkeskused):
– riiklikud programmid:
RP12112, Eesti Pühakodade kivimüüride niiskuskahjustused ja sooldumine, Kallavus Urve (4.05.2012 - 1.12.2012)

- Teiste ministeeriumide poolt rahastatavad riiklikud programmid:

- Uurija-professori rahastamine:

- SA Eesti Teadusfond/Eesti Teadusagentuur

– grandid:
– ühisgrandid välisriigiga:
– järeldoktorite grandid (SA ETF ja Mobilitas):
– tippteadlase grandid (Mobilitas):

- Ettevõtluse Arendamise SA

– eeluuringud:
– arendustoetused:

- SA Archimedesega sõlmitud lepingud

– infrastruktuur (nn „mini-infra“, „asutuse infra“):

– Eesti tippkeskused:
– riiklikud programmid:

AR12138, materjalitehnoloogia, Smart aerogels based on the nanostructured wood cellullose - SmaCell, Urve Kallavus (1.09.2012 - 31.12.2014)

AR12118, materjalitehnoloogia, Effiicient plasmonic absorbers for solar cells, Alvar Kurrel (1.07.2012 - 31.12.2014)

– muud T&A lepingud:

- SA Keskkonnainvesteeringute Keskusega sõlmitud lepingud:
- Siseriiklikud lepingud:
- EL Raamprogrammi projektid:
- Välisriiklikud lepingud:

VA428, Multifunktsionaalsete materjalide süntees ja struktuuri uurimine, Mikli Valdek (1.01.2009 - 31.12.2012)

2.8 Struktuuriüksuse töötajate poolt avaldatud eelretsenseeritavad teaduspublikatsioonid *(ETIS klassifikaatori alusel 1.1, 1.2, 1.3, 2.1, 2.2, 3.1, 3.2, 3.3, 4.1 ja 5.1).*

1.1

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1.2

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1.3

2.1

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3.1

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3.3

3.4

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4.1

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2.9 Struktuuriüksuses kaitstud doktoriväitekirjade loetelu *(NB! struktuuriüksus lisab struktuuriüksuse töötaja juhendamisel mujal kaitstud doktoriväitekirjade loetelu)*

2.10 Struktuuriüksuses järeldoktorina T&A-s osalenud isikute loetelu *(ETIS-e kaudu esitatud taotluste alusel)*

2.11 Struktuuriüksuses loodud tööstusomandi loetelu

4. Struktuuriüksuse infrastruktuuri uuendamise loetelu