Higher Education Quality Assessment Centre of Estonia

Chemical and Material Science

Tallinn Technical University

Institutes evaluated:

Centre for Materials Research
Department of Polymer Materials
Department of Materials Technology

Visit Dates: February 12-17, 2002

Expert Team

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Part I
General Overview

The evaluation team consisted of Prof. Jorma Hölsä (University of Turku, Turku, Finland; team chairman), Prof. Veikko Komppa (VTT Chemical Technology, Espoo, Finland), Prof. Mamoun Muhammed (Royal Institute of Technology, Stockholm, Sweden) and Prof. Heikki Tenhu (University of Helsinki, Helsinki, Finland).

The evaluation was organized by the Estonian Higher Education Accreditation Center (EHEAC). The evaluation was carried out through an examination of documents and a series of site visits, interviews and consultations with the research staff and with students over the period February 12 - 17, 2002. Each evaluator had previously received self-assessment reports from the units to be evaluated:

Centre for Materials Research, Tallinn Technical University (TTU)

Department of Polymer Materials, Tallinn Technical University (TTU)

Department of Materials Technology, Tallinn Technical University (TTU)

The evaluated institutes, laboratories, research groups and individual scientists provided additional material during the visit.

The visits to the institutes started with a general introduction of the institute organization, the financing and the main research topics given by the directors of each institute. The second phase consisted of topic-by-topic presentations of research activities by the team leaders. Finally, the evaluators visited laboratories and interacted with individual researchers.

The evaluators were asked to:

1) Evaluate the quality of the research, the overall capacity of the research teams and the possibilities for application of research results in Estonia;
2) Identify deficiencies in the activities of research and development institutions;
3) Give recommendations on the development concerning research and development, and on research areas necessary to the State of Estonia.

The team was given the following materials: working schedule, principles and criteria for the evaluation of research and development institutions, guidelines to the experts for the research evaluation, and self-evaluation reports as discussed above.

The team arrived on February 12, 2002 in Tallinn; was shortly briefed at the EHEAC and visited the Centre for Materials Research, Tallinn Technical University (TTU) the same afternoon and evening. The team visited the Department of Polymer Materials, Tallinn Technical University (TTU) on

February 13, 2002. On February 14, 2002 the team had its site visit to the Department of Materials Technology, Tallinn Technical University (TTU).

On a first evaluation point, the *quality of the research activities* was considered. This assessment is largely based on the records of scientific publications. Regarding the grading of the research activities, the evaluation team was instructed by the EHEAC to reserve the term **excellent** for groups, which were found to be among the best 10% of the European groups in the corresponding field. Similarly, the term **excellent to good** should be used if the evaluated group was found to be among the best 25 % of corresponding European groups. The full scale comprised 7 levels, in addition to the highest ones the grades are **good, good to satisfactory, satisfactory, satisfactory to unsatisfactory,** and **unsatisfactory.**

The *over-all capability* of a research unit was evaluated based on a the combined assessment of the following criteria (each graded in three levels):

- The originality/novelity of past and ongoing research activities
- The strategy and perspective of the research
- Multidiciplinarity and relevance for other research areas
- The competence of the research groups and their capacity for development
- National and international co-operation
- Success in applying for grants

As the result of this assessment one of the four grades **excellent, good, satisfactory** or **unsatisfactory** was given for the group.

Finally, on a third evaluation point the implementation opportunities for the research results and their importance for the Estonian society was assessed. Here we adopted a three level grading: no comment, good or very good.

The evaluation Team visited the institutes and their evaluation appears below in the same order as they were visited. The visits were kindly hosted by Prof's. Urve Kallavus in the Centre for Materials Research, Antti Viikna, Peep Christjanson, Tiit Kaps in the Department of Polymer Materials as well as Priit Kulu, Jakob Kübarsepp and Enn Mellikov in the Department of Materials Technology. The Team had possibility to openly discuss with researchers and engineers, and the whole staff was very helpful to assist the Team in the evaluation process.

Part II
Evaluation of Individual Institutes and Research Groups

A. Centre for Materials Research, CMR

The Centre for Materials Research was founded in 1992. The Chair of Materials Research was started at CMR in 1995. In 1997 a testing laboratory, was created by the CMR. Thus, CMR comprises a scientific laboratory, teaching laboratory, and a testing laboratory. Eight departments of TTU are

the associate members of CMR. 23 Estonian and foreign universities, institutions, and companies are reported as research partners during 1997-2001.

CMR is a research unit with qualified personnel and instrumentation, which serves various departments in TTU and institutions outside by conducting structural studies of various materials.

The ***personnel*** of CMR includes one professor, Dr. Urve Kallavus, four research scientists of whom three are doctoral students, and three engineers. Four scientists from the Department of Physics and the Department of Fundamental and Applied Chemistry also conduct their studies in CMR.

CMR houses good ***instrumentation*** for materials research. In the research laboratory, the oldest instrument is a scanning electron microscope TESLA BR 300, purchased in 1980. Other, newer instruments include optical microscopes NIKON Microphot FX and SMZ 800 with image analysis system, a scanning electron microscope Jeol JSM 840A with an image capturing system, EDS and WDS spectrometers for X-ray microanalysis with AN 10000, a transmission electron microscope EM 301G, an optical emission spectrometer Spectrolab M5, X-ray diffractometer Bruker D5005, X-ray fluorescent spectrometer PW1400, and a thermal analysis equipment Derivatograph 102. In addition, instruments needed for sample preparation are of good standard.

Teaching and supervision of students from Chemical, Mechanical Engineering, and Information Technology Faculties have started to a limited extent while lecturing courses with tutorials will start during the academic year 2002/2003. At present, three doctoral students (Valdek Mikli, Mart Viljus, Rainer Traksmaa) and two Master students (Kairi Kriiska, Heidi Jänes) work at CMR. ***Rate of publishing*** is indicative of high research activity.

Research topics in the CMR

Prof. Urve Kallavus:

Structural investigation of paper

The main goals of the research were to find out the structure-properties dependence in paper, investigation of the possibilities to enhance the paper quality by improving the internal and surface structure of paper and the application of the new surface sizing technology. Structural investigations were performed with optical and electron microscopy, X-ray diffraction and microanalysis technique.

Chemical decay of Estonian book heritage

Optical and polarization microscopy for the identification of the paper fibres were used. The distinction of the fibres allows determining the origin of paper and gives valuable information for its restoration. The quality of the surface, i.e. the orientation of the fibres, sizing quality, and the distribution of fillers, were studied by scanning electron microscopy.

Investigation of the microstructure of steam-exploded biomass

The steam explosion (SE) autohydrolysis treatment has been used as a method to obtain various value-added products from sugar cane (*Saccharum Officinarum*) bagasse. The changes occurring during SE treatment were evaluated using chemical composition analysis, solubility data, scanning electron microscopy and X-ray diffraction data. Both two-stage extractions (with hot water and subsequently with 90% ethanol) and one-stage extraction with 90% ethanol allowed the separation of one quarter of the material obtained after SE treatment.

Service life of rot-damaged buildings

In this study, the wooden structure of a building, the service lifetime of which has been exceeded, was analyzed. During the analysis some structures were opened, wood moisture content measured and the spreading of dry rot was estimated. In laboratory the type of fungi that caused the decay of the wood was identified and the damaged wood cell structure was studied. The main reason for the end of service lifetime was the damage done by dry rot (*Serpula lacrymans*). Dry rot was spread to such an extent that it cannot be stopped and it is not economically reasonable to treat the structure.

Other topics include biodegradation of wood, the structure of ancient silver, and the corrosion of roofings of old churches.

Principal investigator Mart Viljus (collaboration with the Department of Materials Technology):

Research topics include several topics, e.g. the following: structure formation of TiC-NiMo cermets during the sintering process, carbide grain growth in Cr_3C_2 based cermets during sintering, synthesis of titanium carbide by mechanical alloying, corrosion of Cr_3C_2 and TiC based cermets, erosion-corrosion wear of WC-Co hard metals in a solid-liquid mixture, and the abrasive wear of Cr_3C_2 based cermets.

Principal investigator Valdek Mikli (collaboration with the Department of Materials Technology):

The topics are the following: evaluation of the structure of Co powder particles by electron microscopy and image analysis, electron microscopy studies of the wear and corrosion resistant duplex TiN coatings, SEM and EDS studies of chemically sprayed optoelectronic thin films, and TEM and RHEED investigations of various materials with commercial potential.

Principal investigator Meeme Põldme:

Investigations of structure and aging of natural and artificial stones are conducted, as well as those on the efficiency of treatment with hydrophobic coatings to protect Estonian limestone.

Principal investigator Aadu Paat:

The topics include e.g. the following. Electron microscopy studies of halogen sodalite powders and ceramics synthesised by low temperature

hydrothermal method, sintered materials, investigation of Estonian power station ashes by X-ray diffractometry, investigation of the structure of ozonised polyethylene.

Conclusions

The list of research projects presented above indicates a wide range of topics shows broad competence of the group for the study of different types of materials. Both biological and synthetic (organic and inorganic) materials are investigated. Evidently, numerous methods of sample preparation, especially for TEM, are mastered in the group. The research projects are carried out in cooperation with affiliated Departments.

CMR is a unit worth supporting, owing to its activity and capability. In future, the question of the supervision of the doctoral students needs to be addressed. Students from different departments should have joint supervision and guidance from their own disciplines, as well as from CMR.

CMR has a good potential to serve the Academia and Estonian industry. The Centre could also increase the number of collaborators in the Nordic countries, assuming it retains close contact to the basic research in TTU. In future the university meets the question whether CMR will be developed as a full unit/department or should it be more like a service center. However, at present it is reasonable to recommend that all expensive instruments (TEM, SEM, XRD, XRF) purchased into TTU will be located in CMR. The role of the Centre should be a service unit but with very active participation in research.

The research activity, as well as the overall capability of CMR are evaluated as **good to excellent**. The implementation opportunities are good.

B. Department of Polymer Materials, PMI

PMI consists of three teaching chairs:

- Teaching chair of **polymer technology**. The head of the chair is professor Peep Christjanson. The main subjects of teaching of the chair are: polymer chemistry and physics, polymer synthesis, polymer materials, polymer process engineering, adhesion and adhesives, finishing materials, and high performance polymers.
- Teaching chair of **woodworking**. The head of the chair is professor Tiit Kaps. The main subjects of teaching of this chair are: wood science, woodworking machines, sawmill technology, drying and hydrothermal treatment of wood, woodworking technology and products, wood-polymer composites, pulp and paper technology, and chemical processing of wood.
- Teaching chair of **textile technology**. The head of this chair is professor Anti Viikna who also holds the position of director of PMI. The chair teaches the following subjects: fibre science, textile materials, textile testing, garment design, clothing engineering, installation of apparel

industry, spinning and weaving technology, knitting technology, non-woven technology, textile chemistry.

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PMI was founded in 1992 by combining three above mentioned units, together with a research unit of oil-shale chemistry. The research and teaching topics of the Department are diverse, but to a certain extent combined together by polycondensates synthesised from water soluble alkylated resorcinols. These condensates find applications e.g. as adhesives and coating materials. In addition, some investigations on polyethylene (morphology, surface treatment, recycling) as well as on the surface treatment of wood have been carried out.

The obvious problem, also pointed out in the self-assessment report of the Department, is the age structure of the scientific staff. The senior scientists are about to retire, and great expectations on the continuation of the research and teaching are on a few doctoral students. A general impression is that to survive, the Department needs to be strengthened by developing close international contacts but inevitably also by cooperation within TTU and with the industry. If the traditional research field of phenolic resins and their applications is considered a field with a future, more emphasis on polymer synthesis in general would be important. Collaboration with the Faculty of Chemistry, as well as with the Department of Materials Technology is beneficial for all units.

Polymer resins and adhesives
Principal investigator: Peep Christjanson

The research group of Christjanson concentrates on polycondensation reactions as well as on the chemical and structural studies of product polymers. An important topic in Estonia is the utilisation of resorcinols derived from oil shale, in the syntheses of polymeric materials. Applications of the polymers as adhesives and coatings, especially in treatment of wood and in wood composites, have been studied. Textile laminates and adhesion properties of polyethylene have been studied as well.

The research group has gained high level in the chemistry of phenolic resins. However, the number of publications is rather low. The scientists involved in this research are all seniors and, unfortunately, there does not seem to be persons capable to continue the work. This point is of utmost importance because the group of Christjanson is the only one in the Department practising synthetic polymer chemistry. A total of 7 master theses have been produced in these projects. Christjanson has also supervised the doctoral studies of Andres Krumme, concerning bimodal polyethylene (this will be considered separately). The research activity is evaluated to be **good** and the overall capability of this team is **good**. The implementation opportunities are good.

Factors influencing the adhesion of resorcinolic resins
Principal investigator: Tiit Kaps

In this project, the influence of various factors on the adhesion of resorcinol-formaldehyde adhesive resins is studied. Various resorcinolic raw materials are used for the synthesis of resins with various molar ratios of the main components. The type and content of the catalyst and solvent are varied as well. Influence of the adhesive on e.g. wood light resistance is also studied. The results are used to improve the wetting of wood surface, and to guarantee that the strength of glued wood is determined by the cohesive strength of the wood itself.

The research fits well the profile of the Department, as an integral part of studies on phenolic resins. Two master theses have been defended. No reports in scientific journals have been published, however. The research activity is evaluated to be **satisfactory**, and the overall capability of the team is **good**. The implementation opportunities are good.

Use of short flax fibre and tow for producing of fleeces, mulch matting and geotextiles
Principal investigator: Anti Viikna

Production of mulches with different surface densities is studied using short flax fibre as raw material. Mulch materials are produced by wet and needle punching methods. The influence of different adhesives on the strength of bonded fibre materials and their water resistance is studied. The produced batches of mulches are tested in sea buckthorn plantations. Thin fleeces with different content of flax fibre and low melting polyester fibre are also prepared and tested. The strength properties for all the produced materials are determined. The project aims to applications e.g. in agriculture and geotextiles, and may turn out to be of considerable economic importance in Estonia. Only one conference paper is based on this project run by one student and a senior engineer. The research activity is evaluated to be **satisfactor**y, and the overall capability of the team is **good**. The implementation opportunities are good.

Other research projects

Studies on melt properties of polyethylene, and especially on the crystallisation of bimodal polyethylene

These studies comprise a part of doctoral thesis of **Andres Krumme**. Anti Viikna, and evidently also Tiit Kaps, have supervised the work, which seems to be inspired by the needs and interests of Borealis Polymers Ltd (Porvoo, Finland). There has also been supervisor in Borealis Polymers Dr. Arja Lehtinen. Melting of polyethylene has been studied by optical microscopy to detect gel fractions in the polymer. More importantly, crystallisation of polyethylenes with bimodal molar mass distributions has been studied. Of interest has been e.g. the kinetics of crystallisation, and the shrinkage of the crystallising polymer. Various methods have been used in the research, e.g.

scanning calorimetry and rheological methods. This research has most probably been highly challenging for Krumme and should lead to a good doctoral thesis. It should be pointed out, however, that this type of research has been conducted in Borealis Polymers for years. After finalising the PhD in 2002, it is highly recommendable for Krumme to find his own research field, preferably during a post doc period at some foreign university/ies. Krumme's plans to finalize his thesis by the end of the year 2002 but has not yet published his work as research papers. It was noted that several Master theses are being done on the topics similar to Krumme's thesis.

Study of paper structure

This research is conducted by **Rein Reiska** (PMI) under the supervision of Urve Kallavus (Centre for Material Research). The aim is to establish the dependence of paper properties on the structure, and to find out possibilities to enhance the paper quality by improving the internal and surface structure of paper and by application of new surface sizing technologies. Structural investigations are performed using optical and electron microscopy, X-ray diffraction and microanalysis technique.

Conclusions

Certain synergism seems to be found in the diverse research projects conducted in the Department of Polymer Materials. However, research activity is rather weak and the productivity has been moderate in terms of Master and Doctoral theses, and especially in terms of papers published in highly reputed international journals.

Extensive material testing is conducted in the Department. This is considered beneficial because it facilitates the contacts with Estonian and foreign industry. The accumulated know-how in the properties of polymeric materials is obviously of vital importance for the growing industry in the country.

The Department (PMI) is approaching a state of a profound transition. According to the self-assessment, 18 Master theses are in the pipeline and are expected to be defended ("in future") by students born between 1972 and 1976. Four Doctoral theses are expected, two of them during 2002-2003. However, when the senior scientists retire, there is a need for experienced personnel to supervise young scientists. Further, upon the process of the young generation to enter the Department, much of the traditional research will be finished. There do not seem to be qualified scientists capable of continuing the synthetic work. Strengthening the research on polymer synthesis most probably would be of utmost importance for the education of polymer scientists in general and correspondingly, for the Estonian industry. Young scientists finalising their doctoral studies are strongly encouraged to find foreign post doc positions to vitalise the research in the Department. As stated earlier, close international contacts and collaboration with other units in TTU is necessary.

Based on the above reasoning, the research activity of the Department is evaluated to be **satisfactory to good**, and the overall capability as **good**.

C. Department of Materials Technology

In 1998, a new system of financing science was adopted in Estonia, and the following main research themes and principal investigators were defined:

- *Technology of composite materials and coatings* (Prof. Kulu)
- *Tribomaterials and -systems* (Prof. Kübarsepp)
- *Polycrystalline semiconductors* (Sen. res. Krunks)
- *Chemistry and technology of electronic materials* (Prof. Mellikov)

R&D activities of the Department of Materials Technology (professorships of materials science, metal processing and technology of semiconductor materials) are concentrated on wear-resistant composite powder materials and coatings and optoelectronic materials.

During the next few years the Chair has planned to invest approximately 5 million EEK to strengthen its infrastructure through development contracts financed by foreign and domestic industries and through national resources of financing fundamental sciences in the "target financing". The Chair has planned completion of 4 master and 2 doctoral theses during the next four years. Effective master and doctoral studies in the fields of chemistry and materials technology and production engineering and active participation in the activities of the projected Graduate School of Materials Technology (degree studies) will guarantee its successful performance. Efforts will be made to intensify activities related to the participation in the research projects of the European Union and in the common European research environment.

This evaluation report covers the research done in the above groups during the five year period of years 1997 – 2001. The evaluation was carried out by the Team including a site visit on February 14, 2002.

CHAIR OF MATERIALS SCIENCE

Prof. Priit Kulu, D. Eng. Sc.

Development, Testing and Reuse of Industrial Materials

Principal investigator: Prof. P. Kulu, D. Eng. Sc.
Main investigator: Prof. J. Kübarsepp, D. Eng.

The aim of the research group is to create new materials and coatings for protection against corrosion and wear, develop materials testing systems, and develop technologies for utilisation of industrial wastes.

The R&D activities are in the following technological problems:

1) Wear resistant powder materials and coatings with designed characteristics for wear-corrosion working conditions
2) Development of materials testing and quality assurance system in Estonia
3) Reuse of industrial materials

The group has studied the structure and formation processes of titanium and chromium carbide hardmetals, the theoretical bases of disintegrator milling and modelling of milling process, granulometry and morphology of ground product. The Laboratory of Metals of TTU Testing Center has prepared for its accreditation and received it for certain physical tests. TTU has also actively developed new testing systems for wear measurements.

The academic staff consists of 7 researchers, one doctoral student and 4 master degree students. The group conducts research in an area that has great technological applications. The materials studied have applications in the areas of anti-skid studs of automobile tyres, disintegrator milling systems for treatment of different materials. The group has not been very active in publishing scientific papers and there is only one Master theses produced during the evaluation period. The activities in testing are very good and probably important for the whole country. The instrumentation is modern and the test methods are accredited and the group participates in training courses and examinations at AEL in Finland in the NDT testing area. The group has received substantial financing from PHARE projects.

The evaluation team values the efforts of the group in the testing and quality related matters very good. The research activity is considerd low and rated as **good to satisfactory**. Some of the instruments and facilities are good and modern and staff has good knowhow about them. The danger in this group is that contract testing leads easily to short sighted money hunting and real research is given low or no priority. The research is very applied and the technology can be directly and readily usable.

Technology of Composite Materials and Coatings

Principal investigator: Prof. P. Kulu, D. Eng. Sc.

The main aim of research is to create advanced technologies (powder metallurgy, thermal spray, laser and plasma technology, disintegrator technology), metal composite materials and coatings with unique nano and micro structures.

The research concentrates on disintegrator technology, powder composites for thermal spray, PVD and laser technology and joining of ceramics and hardmetals. The group has developed technologies for production of powders of hard metals and ceramics. Based on laser and PVD technologies special thin films are manufactured. Disintegrator milling technique is used to make new composite powders for wear and corrosion resistant thermal sprayed coatings.

Porous PVD coatings are developed for medical and catalytic applications. New coating technologies (HVOF spraying, selective galvanizing) are implemented in Materials Processing Technology Transfer Center and services are offered in the field of strengthening new and restoration of worn parts. Joining technology of new type of hardmetals are developed for AS Mäetehnika for joining of hardmetals with steel of cutter elements of oil shale treatment combines.

The research group consists of 8 researchers and one lecturer and they have produced 8 master theses and 3 doctoral theses. The number of publications is 33 of which majority are in respected international Journals. The group has active collaboration with Finnish Universities and Institutes. The disintegrator research and development work is valuable and rather unique in character.

The evaluation Team appreciates the research and technological services and skills of the group and considers the overall capability to be **good** and research to be competent and scientifically **good to excellent**. The research has been implemented for industrial application in a positive way.

Tribomaterials and Systems
Principal investigator: Prof. J. Kübarsepp, D. Eng.

The purpose of the research in this group is to characterize and test new tribological materials, develop fundamental theories for their selection for different conditions of wear and friction. Cermets (ceramic metal composites) have been studied to optimize their chemical composition, production technology and sintering behaviour. The mechanical and wear characteristics of TiC-FeNi cermets are compared with WC-base hard metals.

TiC-based Fe-Ni alloy bonded cermets have already proved itself as suitable material for the production of tools for metal plastic forming (blanking, heading etc). TiC-NiMo-alloys are mainly prospective as wear resistant high-temperature materials. Anti-skid studs of automobile tyres have been produced in collaboration with Finnish and Swedish companies (Kovametalli Oy and Sandvik). Production in China has also been discussed.

Cermets containing chromium carbide possess a unique combination of wear and corrosion resistance, making them thus useful for certain tool and chemical applications. Components from Cr_3C_2-based cermets have been produced by the firm SUMAR AS (Estonia).

The research group has produced 7 master theses and 2 doctoral theses and 16 scientific papers the majority of which in respected international Journals. The evaluation team finds the research of this group interesting and succesful. The group has contacts with industry and nearby research institutes and universities. The overall capability of the group is rated as **good** and its scientific research comparable with **good to excellent** international research. Cermets are materials having interesting advanced applications.

Sprayed and Deposited Coatings
Principal investigator: Prof. P. Kulu, D. Eng. Sc.

This research area is very new and it was started in 2002. Hence it is not so easy to evaluate its research. The evaluation Team found this research topic to be innovative and important for the University.

The main objective of research is to study coatings materials, technologies and properties aimed to create hightech coatings with specific properties for extreme operation conditions. The research is planned in two directions:

- elaboration of corrosion and abrasive-erosive wear resistant powder coatings
- study of the technology and properties of advanced structural layers

The new wear resistant tungsten carbide (WC)/titanium carbide (TiC) – metal coatings are corrosion resistant. The structural layers, including multilayers, porous layers and layers with open columnar structure could be used as electrode surfaces in medical industry and catalysis. The multilayers obtained by interfacing NiAl/Al_2O_3/ferromagnetic metal layers. are expected to find use in data storage devices. Use of selectively plated coatings with combination of sprayed powder coatings enable to create layers with unique properties for different applications.

The research group consists of 3 doctors, 3 researchers and 3 engineers. There are also 6 Master students and 2 Doctoral students.

CHAIR OF SEMICONDUCTOR MATERIALS TECHNOLOGY

Prof. Enn Mellikov, D. Eng. Sc.

The research group of the technology of the semiconductor materials originates from 1968 and it focuses on studies of A_2B_6 compounds. During the 1990s the research of more complicated ternary compounds ($CuInSe_2$ type materials) was started. The group acquired financing from the Volkswagen Foundation in 1992 and in 1994 the group was the first at TTU to participate in the European Union financed project.

The research programme in the field of electronic materials focuses on different physical, chemical and technological aspects of A_2B_6, copper indium selenide (CIS) and electroconductive polymer materials (ECP) to acquire a better understanding of the processes leading to the formation of materials with the predicted properties. The research group has without doubt good resources and technological know-how and its research results are internationally acknowledged. The research group has a joint project with Scheuten Glasgroep BV aiming at the creation of solar cells based on powder materials.

Chemistry and Technology of Compound Semiconductor Materials
Principal investigator: Prof. E. Mellikov, D. Sc.

The main goal of the research is to study regularities of recrystallization of A_2B_6 and CIS ($CuInS_2$, $CuInSe_2$) powders in different molten fluxes ($CdCl_2$, NaCl, Te, Na_2S_x,, Se). The aim is to establish the correlation between the preparation conditions and the electrical, optical properties and granulometric characteristics of the final product and to develop materials with improved characteristics for applications in solar cells, radiant detectors and luminescent materials.

Disintegrator system for chemical spraying of A_2B_6 and $A_1B_3C_6$ semiconductor thin films was built. The size and orientation of crystallites in the A_2B_6 and $A_1B_3C_6$ films, the optical and electrical properties of the films were determined and their dependence on process parameters like concentration of precursor solutions, droplet size, substrates temperature and spraying time were studied.

The control of defect structure allows to produce materials with predicted composition and practically important physical parameters. He group syntesised powders of different semiconductor compounds whch are potential candidates for Photo Voltaic (PV) solar energy and sensor techniques.

The group consists of principal researcher and 7 PhD researchers and it has published 13 scientific publications. Possibly the collaboration with industry is slowing down group's publishing activities. No theses have been produced during the evaluation period.

The capabilities of the research group are **excellent** and research activity is **excellent to good**. The risk in their work is in the fact that their work is very much industry driven, covered by secrecy agreements and other contracts. Because potential breakthroughs can have great financial impacts it is in the interest of TTU and researchers to have all the agreements regarding Intellectual Property Rights (IPR) done in the best possible way to all the parties involved.

The research of this group is evidently at very high international and scientific level. The group receives substantial financing from abroad and has very good equipments and instruments available. The laboratory facilities are new and build in purpose for their research. The group is planning to buy a SEM just for their own use. Because there exists a separate Centre for Materials Research within TTU the Team recommends that CMR should be utilised as much as possible and as long as its research capacity is fully used. The Team recommends that new instruments and analytical equipments should be purchased preferably for CMR, not for individual research teams and groups.

Chemistry, Physics and Technology of Electron Materials
Principal investigator: Prof. E. Mellikov, D. Sc.

The new, cheap and convenient production method of CdTe nanosize particles by gas phase condensation was developed and the produced materials studied. Formation of CIS and CdTe monograin powders in the

process of isothermal recrystallization of initial powders in different fluxes, the dependence of various technological parameters on film formation were studied. Different technologies for formation of new design of solar cells on the basis monograin layers of these materials were developed. The recombination of near dislocations and complexes with deep donor - deep acceptor in CdTe and different CIS materials were studied. Photoluminescence measurements were made for CIS materials produced by different techniques. Vapour-liquid-solid equilibria were studied in the systems $(CdSe_xTe_{1-x})_{solid}$-$CdCl_2$(l)-CdI_2(l) and $(CdSe_xTe_{1-x})_{solid}$-$CdCl_2$(g)-CdI_2(g).

The inorganic-polymeric composites for solar energy using inorganic photoactive materials and conductive polymers (CP) were prepared. The different CPs, inorganic photoactive coatings, the preparation methods of thin films and inorganic-CP composites were developed.

The group is very active and its research is on a very high and competent international level. The research is financed to some extent by EU and there are new proposals for the 5th Framework Programme of EU. The group consists of a principal investigator and 6 PhD researchers and it has published very actively (45 scientific papers; no theses, however) in respected international Journals. There is no doubt within the evaluation Team that the group's capabilities are **excellent** and its research is **excellent**.

The words of warning expressed above by the Team in the case of Prof. Mellikov's other research activity apply also to this research. The research results have lead to international collaboration project with Schouten Glasgroep BV (the Netherlands) which helps much with investment possiblities of the group. However, the Team does not recommend to purchase valuable instruments for their own use only but rather to the Centre for Materials Research.

Polycrystalline Compound Semiconductor Materials
Principal investigator: Senior researher Malle Krunks, Ph. D.

The main goal of the research in the field of $A_1B_3C_6$ and A_2B_6 materials chemistry and technology is to understand better chemical spray and electrochemical deposition processes and mechanisms of formation of homogeneous thin films with predicted properties.

The research group has studied the formation of CdS, $Cu_{2-x}S$, ZnS and $CuInS_2$ thin films by pyrolysis using metal chalogenides and thiocarbamide aqueous solutions as starting materials. The conditions of spray pyrolysis for deposition of thin ZnO films for solar cell application have been studied. The possibility to apply the spray pyrolytic process for deposition of thin films with nanosize crystallites is proved on the basis of ZnO films.

The group consists of a principal investigator and 4 researchers. The group has published 18 scientific papers and they have EU Human Potential Programme financing for researcher exchange. The overall capability of the research is rated **good to excellent** and research activity also **good**. PhD

Vello Valdna from the group has made innovations concerning luminescence Cd(Se,Te) materials that are in use in CCD detectors manufactured by a US company (Bruker AXS and Phosphor Technology Center, Atlanta, GA, USP 6,254,806).

Part III
Summary

To summarise, the ratings of the various units/research groups are tabled below. The Team has tried to find the best balance of earlier evaluations and formed a general overview of the units and their future possibilities. Productivity has been rated based on the number of examinations and papers published in high level international Journals. There is one research area (Semiconductor Materials Research) that is performing well when compared to the other units. However, the production of graduate students should be better. The Department of Polymer Materials has problems with the age of the staff, hence its future has been ranked as satisfactory only. The Department and University should really try to attract younger researchers to be able to overcome the problems and to guarantee the continuation of teaching and research of polymer science in Estonia. The present situation is jeopardizing the future of polymer research in the whole country. Practically the problem can be solved within the University with proper adjustments in money allocation.

Table Summary of ratings of various units.

Unit of TTU	Present Level of Research	Future Perspectives	Productivity
1. CMR	G/E	G	G/E
2. PMI Christjanson	G	S	G/S
3. PMI Kaps	G	S	S
4. PMI Viikna	G	S	S
5. MT Kulu	G	G	S/G
6. MT Kübarsepp	G/E	G/E	G/E
7. MT Mellikov	E	E	G/E
8. MT Krunks	G	G/E	G/E

CMR: Centre for Materials Research; PMI: Department of Polymer Materials, MS: Department of Materials Technology
E: Excellent, G: Good, S: Satisfactory

Part IV

Recommendations regarding research in the evaluated units:

- Internal and external collaboration needs strengthening
- Increased collaboration with neighboring countries should be strengthened
- Expensive equipment should be preferably placed at CMR and to be made available for research groups
- CMR needs strengthening and support from the Chairs and research groups
- Better possibilities and encouragement should be given to young post graduates and post-docs to do research and collaboration abroad
- Secrecy agreements and contracts with industry should be carefully made and the interests of all parties must be taken into account
- Teaching programs should include practical quality system training (ISO 9000, ISO 17025, ISO 14000, GLP) in the curricula as well as modern measurement uncertainty issues (ISO Guide on Uncertainty of Measurement, GUM)