Background and Mission

The EMPART (Electronics Materials, Packaging and Reliability Techniques) research group is a multidisciplinary research unit with specialists in microelectronics, materials engineering, measuring techniques and in chemical and physical sciences; six professors, three docents, three other doctors and 20 doctoral students. The group leaders, Professor Heli Jantunen and Professor Osmo Hormi supervise the postgraduate studies in the group with co-operating professors from the University of Oulu and from the Technical Research Centre of Finland in Oulu (VTT Electronics).

The research activities concentrate on new materials, components and future manufacturing and packaging technologies required in the development of multifunctional smart electronics:

- Electronics materials of thin and thick films (ferroelectrics, dielectrics, organic luminescent materials, porous semiconductors and carbon nanotubes) are synthesized using powder synthesis, synthetic organic chemistry, electrochemical etching, laser-assisted and chemical vapour deposition methods.
- The above advanced materials are integrated in novel components applying the state of the art manufacturing and packaging methods towards multifunctional silicon, ceramic and hybrid packages and micromodules.
- The results of the research include components and devices in the fields of electronics, RF (radio frequency) applications, piezoelectric sensors and actuators and optical components. Currently the field of interest is also being extended towards thermal management of the electronic circuits by nano-material assemblies, chemical sensors / catalysts and biochemical carriers based on nanostructured materials.

The electrical, optical and thermomechanical design and modeling in conjunction with standard measuring and materials characterization techniques as well as fault and reliability analysis are organic elements of the research.

Scientific Progress

During the research year 2005, in accordance with the long term research targets of the group, we have continued the integration of interdisciplinary topics towards future advanced device and component implementations. In the following sections, selected research areas of the group are presented.

1. Materials

The long term experience of the group in the research of linear and nonlinear materials (dielectrics and electrically tunable ceramics for telecommunication devices and piezoelectric and pyroelectric materials for sensors and actuators) as well as organic materials for OLEDs (organic light emitting diodes) enabled a natural extension of the research to nanostructured materials and micro modules.
Nanostructured materials

Nanotechnology offers and eventually requires, the collaboration of different disciplines by which a colorful mixture of thinking is formed in the research team. The team consists of chemists, physicists and engineers, and the topics researched include synthesis and application of nano-porous silicon structures, carbon nanotube assemblies, metal and metal oxide thin films and nanoparticles, nanofabrication technologies, polymer/ceramic nanocomposites and varistor materials with doping, enabling their cofiring with commercial LTCC (low temperature co-fired ceramic) materials.

Thermal oxidation of porous silicon: Study on structure

The structural changes of porous silicon samples during oxidation are being investigated and analyzed using various microscopy techniques and x-ray diffractography. The surface roughness of oxidized PS layers increases with the oxidation at 200 - 400 °C and decreased at 600 - 800 °C. At 800 °C a partially fused surface is observed. The oxide formed on the wall of a porous silicon skeleton is amorphous. The shifts of Si (400) peaks are observed in the x-ray diffraction patterns, which are correlated to the lattice deformation induced by a thermal expansion coefficient mismatch between the grown SiO₂ and the residual Si, and to the intrinsic stress caused by the Si-O bonds at the Si-SiO₂ interface. These explanations are supported by thermomechanical modeling using a three-dimensional finite element method.

Laser-induced gold deposition on p⁺-Si from liquid precursors

Gold micropatterns are deposited from aqueous solutions of NaAuCl₄ on boron-doped Si (100) surfaces (1.5 · 10⁻⁴ Ω·m) using a focused Ar⁺ laser beam (TEM₀₀, λ = 488 nm, w₀ = 1.5 µm, P = 20 - 80 mW). The maximum temperature at the precursor/silicon interface increases only to the range of 316 - 372 K, which is not high enough for chemical reactions with formaldehyde in the precursor. This suggests a different mechanism is responsible for the reduction of gold ions; namely, changes in the surface potential of Si caused by the Dember and Seebeck effects.

Carbon nanotube synthesis on oxidized porous silicon

Carbon nanotubes were grown on thermally oxidized porous silicon by catalytic chemical vapor deposition from a mixture of ferrocene and xylene precursor. The growth rate of carbon nanotubes showed dependence on the oxidation extent of porous silicon. On pristine porous silicon surfaces, only poor nanotube growth was observed, whilst samples oxidized in air at 200, 400, 600 and 800 °C prior to the deposition process proved to be suitable substrates for carbon nanotube synthesis. Networks of carbon tubes with diameters of ~ 40 and ~ 10 nm observed on the surfaces of samples were investigated by electron microscopy and by energy dispersive X-ray analysis.
Integrated ferroics

The main effort of the research was directed to the development of thin-film integrated varactor in the frame of the Tekes project “Adaptive RF circuits based on ferroelectric thin films” (in cooperation with VTT Information Technology). The RF varactor with satisfactory performance (tunability to 2.5 at bias 5 V and frequency to 2 GHz) has been prepared using thin films of Ba$_{1-x}$Sr$_x$TiO$_3$. The epitaxial quality of Ba$_{1-x}$Sr$_x$TiO$_3$ films, and the effects of buffer layers, of sequence and composition of the layers on the microstructure were analyzed.

A combined methodology of laser ellipsometry and optical reflectivity spectroscopy has been developed for studies of ferroelectric thin-film multilayers (in cooperation with the Institute of Solid State Physics, University of Latvia, and supported by a CIMO grant to I. Aulika). Studies of the thin-film nonlinear dielectrics have been continued.

Materials for multilayer modules

LTCC compositions for varistor components meant for protection of electrical circuits against transients have been researched. Because of its excellent non-linearity, zinc oxide has been applied as a basic material in the varistors. The group has been able to develop a novel composition suitable for the LTCC process (~ 900 ºC) by light doping of ZnO with semi-conductive glass. The work was done in the project “Advanced Devices through Light Element Doping of Electroceramics” (ADLE) financed by the University of Oulu in cooperation with the Inorganic Chemistry and the Optoelectronics and Measurement Techniques Laboratories (University of Oulu). A new composition based on ZnO with 10 wt% of added glass showed good varistor characteristics: $V_{bk} = 378$ V/mm, $\alpha = 33$ and $J_L = 15$ µA/cm$^2$. Currently, multilayer components utilizing this material with commercial dielectric LTCCs are under research with the Wroclaw University of Technology, Poland.

Additionally, research on composites made of ceramic micro- and nanoparticles and polymers has been conducted to develop novel multifunctional materials, which can be used as discrete components or integrated with other materials. This project (Composites with nanoparticle addition) is funded by TEKES and industrial partners. For deeper understanding of the dielectric properties of 0 - 3 composites, a microstructure-based numerical dielectric modeling method for ceramic/polymer composites has been studied in collaboration with the Electromagnetics Laboratory of the Helsinki University of Technology. The microstructure of Epoxy/TiO$_2$ composites with ceramic loading of 35 vol% was studied to reveal the correlation between the theoretical and experimental permittivities.

Organic light emitting diodes

The use of organic compounds in the preparation of LEDs has created completely new approaches to displays which are thin, flexible and bright. The research is financed by the Academy of Finland (Compounds for Organic Light Emitting Diodes) which concentrates on the organic chemistry of thermally stable emitters in organic LEDs. The group has also won a prize for excellence in team work.

The focus of the research is to concentrate especially on electron transmitters and emitters used in organic
LEDs in order to enhance the thermal and chemical stability of the final LED device. Suitably substituted derivatives of monomeric Alq3 and poly(thiophene) with enhanced light emitting and electron transmitting ability will be prepared. In the case of monomeric emitters, they will be bound to a polysiloxane matrix prior to the preparation of the final device. The methods used in the organic LEDs sub-project will concentrate on organic and polymer synthesis. The preparation of final devices and their testing will be carried out at VTT Electronics (Oulu), OSC (Optical Sciences Center, University of Arizona), State University of Arizona and at Braggone company (Oulu).

2. Processes

LTCC line

Utilizing the LTCC prototype manufacturing and sample production line, the EMPART group offers an attractive platform for customized RF and microwave applications. Potential support in the fields of mobile communications, wireless, automotive, sensor technology, space and various consumer products is enabled. The transition from research to the final product/demo is helped by long-term cooperation with materials suppliers. In addition, our LTCC process team is composed of experienced researchers capable of designing, modeling and developing novel customized/engineered LTCC materials and processes. Final testing/characterization and post-processing such as surface mount techniques, wire bonding and dicing complete the prototype service.

Laser-assisted surface processing

When rapid mask-less laser processing with an accuracy of a few micrometers is aimed, pulsed solid state lasers (Q-switched Nd:YAG, Nd:YVO₄, Er³⁺:YAG, Ti:Sapphire) equipped with harmonic frequency generating optics (non-linear optical crystals) give the best results. Our group has a diode pumped high-performance frequency-tripled neodymium-doped yttrium vanadate laser system (Siemens, Microbeam 3200 model, 3ω Nd:YVO₄, λ = 355 nm, P = 0.2 - 3.2 W, τ = 30 ns, f = 20 - 100 kHz, w₀ ~ 15 µm, scan rate up to 2000 mm/s, work table 610 × 710 mm², accuracy 1 µm in the galvanometric scan field of 50 × 50 mm²).

Due to the short wavelength (UV, 355 nm) and short pulse duration (30 ns), the laser-matter interaction is rather photolytic than thermal, which makes the materials processing “clean”, avoiding thermal reactions (oxidation, re-crystallization, crack formations) in the residual materials and resulting in a very precise processing of most materials used in the microelectronics. The high pulse repetition rate, high average power and the fast scanning capabilities enable extremely fast mask-less materials processing (e.g. in a PCB having 65 µm RCC and 12 µm Cu layers ~ 200 pieces of micro-vias can be drilled within a second). The controlling software package installed on the facility enables fine tuning of the laser and materials processing parameters. The different sequential process tool options provide excellent opportunities to structure/cut/drill/modify a large variety of single and multilayer materials. The files containing the patterns can be made using any CAD software.

Chemical vapor deposition of carbon nanotubes in an Si environment

Both porous silicon (PS) and carbon nanotubes (CNTs) are considered as alternative building blocks in future nanoelectronic, field emission, photonic and nano-sensor devices. Currently, it is a challenging task to integrate CNTs into an Si environment and to find selective synthesis methods compatible with standard fabrication processes. Recently, at one of our collaborating laboratories (Rensselaer Polytechnic Institute, Troy, USA), a highly selective synthesis method of multi-walled carbon nanotubes MWCNTs on Si wafers having SiO₂ patterns on its surfaces was demonstrated in a catalytic chemical vapor deposition (CCVD) batch reactor, using ferrocene and xylene as sources for Fe nano-catalyst and carbon, respectively.

Growth takes place at temperatures above 750°C. The substrate materials have to fulfill two important conditions: (1) They should stand the processing temperature without melting, and (b) they should show chemical inertness with the precursor and with the
catalyst in order to avoid catalyst poisoning. Therefore, the combinations of materials in the substrates proved to be quite critical. The best performance synthesis of CNTs was achieved on SiO$_2$ and on Al$_2$O$_3$ surfaces masked with Cr/Au, i.e. the process is compatible with other Si fabrication methods.

Nanostructured porous materials by anodization

Highly porous SiO$_2$ and Al$_2$O$_3$ templates are made by the anodization (electrochemical etching in acids) of Si and Al surfaces, respectively. Since the porosity and pore dimensions of the substrates depend on the anodization conditions, a number of different porous templates with pre-defined properties can be created. Because both processes are compatible with conventional Si fabrication, the porous structures and their derivatives enable component integration on the chip level, by which new sensor and catalyst devices will be accomplished.

Injection molding and extrusion for integrated electronics and functional composites

Integration of electronics components into injection mold plastic parts is an interesting possibility for added value plastic products. In a project, funded by Tekes and industrial partners, the group has researched, in cooperation with research partners at VTT and the North Karelian University of Applied Sciences, injection molding of plastic miniature parts with micro scale features. The two main subjects of the project are to reveal the limitations of plastic molding and to integrate microelectronics enabling smart plastic devices. The aim is also to research injection molding of the ceramic/polymer composites, the development of which is described in the materials section.

Summary of nanostructured support synthesis and basic properties

<table>
<thead>
<tr>
<th>Materials</th>
<th>Synthesis method</th>
<th>Post-treatment</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porous SiO$_2$</td>
<td>Galvanostatic anodization of p and p' Si wafers in aqueous HF solutions -&gt; porous Si</td>
<td>Oxidation of porous Si in air or in solution of H$_2$O$_2$/H$_2$SO$_4$ to form porous SiO$_2$</td>
<td>Randomly oriented uniform pores with tunable porosity 20 - 85 % and pore size 5 - 100 nm</td>
</tr>
<tr>
<td>Porous Al$_2$O$_3$ (AAO)</td>
<td>Potentio-static anodization of Al in either H$_2$SO$_4$, (COOH)$_2$ or H$_2$PO$_4$ solutions</td>
<td>Optional pore widening in H$_3$PO$_4$ solutions; Fabrication of freestanding membranes with selective Al etching in HgCl$_2$ solution</td>
<td>Hexagonally ordered columnar pores, with tunable porosity 10 - 30 %, pore size 5 - 200 nm and interpore spacing 50 - 400 nm</td>
</tr>
</tbody>
</table>

Laser and FIB micromachining

A method based on laser-assisted deposition of copper was utilized to complement focused ion beam (FIB) circuit editing work. Tungsten (W) patterns deposited by FIB have too high resistance, which subsequently can be reduced through applying a laser-assisted chemical vapor deposition of copper over the W pattern by scanning the beam on the top of the W line. With proper LCVD parameters, the deposition mechanism of copper is self-limiting and selective between ASIC passivation and the FIB deposited tungsten line, enabling high lateral resolution.

LCVD copper deposition on FIB deposited tungsten is shown in the figures below. This particular deposition was carried out only on the middle part of the FIB deposited tungsten wire to give better contrast between the two deposits. Before copper deposition the resistivity, as measured by the four point probe method, of the FIB deposited tungsten was found to
be of the order of 400 µΩcm. Subsequent LCVD copper deposition resulted in a significant decrease in the line resistance (from 90 Ω to 40 Ω). The resistivity of the laser deposited Cu was found to be 16 µΩcm. FIB cross sectioning showed that the original tungsten thickness was 400 nm and the copper deposition 500 nm. The line widths were 5.9 µm and 4.4 µm for tungsten and copper, respectively. The aluminum four-point probe measurement pads and fingers are also visible. The close-up image shows the small crystallites in the middle of the trace where the laser induced temperature was higher than on the edges.

Control and sensor systems using resonance and non-resonance driven piezoelectric actuators have been applied in tunable capacitive sensors having promising characteristics. The first prototype showed large displacement ~ 70 µm with a resolution of ~ 0.4 µm with a bender type piezoelectric actuator. The actuator is simple, thermally inert, tunable, and it can be integrated with various materials e.g. ceramic and printed circuit boards.

Basic research on pre-stressed piezoelectric bending actuators was conducted to profoundly characterize the physical background of their electrical and electromechanical behavior. Information on the different types of pre-stressed actuators was gathered and analyzed in order to develop special actuators (named PRESTO by us) with, to the best of our knowledge, the highest $d_{31}$ characteristics ever made. The transient computer-assisted analysis of the structures and the arbitrary driving functions for the piezoelectric actuators enable us to model pre-stressing the actuators, as well as to calculate the hysteresis of actuators. The ATILA software - with a new efficient modeling method developed in our group - has been recently employed to optimize the LTCC embedded actuator structures.

3. Components

Sensors and actuators

Electromechanically active ceramic parts, mainly for actuators, have been integrated and optimized in the LTCC environment. Due to the excellent chemical resistance of the ceramic and the temperature resilient bonding mechanism, such actuators enable operation in harsh environmental conditions. The research work is targeted at microwave driven pre-stressed piezoelectric bender actuators with wireless control. The flexible manufacturing methods we developed enable production of customized systems for consumer electronics such as alarming systems, sensors, relays, switches, valves, pumps, and mechanical controller/regulator/tuning devices. Preliminary research has been conducted for utilizing ceramic MEMS based on piezoelectric tapes.

RF and microwave components, interconnections and materials characterization

Cost-effective packaging design is becoming a precondition for the larger adaptation of multi-chip module (MCM) technologies in emerging microwave and
mm-wave communication systems. To satisfy the low cost, high performance and miniaturization demands, it is necessary to increase system integration with the use of well-established manufacturing technologies. The key solutions considered here are highly integrated modules realized with standard multilayer ceramic (LTCC) technology as well as high-density ball-grid array (BGA) and flip-chip package interconnections. Furthermore, to ensure the high reliability of module assemblies, efficient thermo-mechanical, thermal and electromagnetic (EM) co-design of module interconnections is needed.

The work done in 2005 mainly covers the design and construction of RF filter modules and interconnections, as well as some electrically tunable devices, phase shifters and dividers, for 26 GHz frequency utilizing customized ferroelectric LTCCs. Studies on the design, modeling and manufacturing of LTCC structures and modules, as well as the reliability of LTCC/BGA/PCB joint, were carried out in a series of projects funded by the Academy of Finland, Tekes, the EU and industry.

One of the main achievements was that new LTCC electroceramics for high frequency applications were synthesized with electrically tunable permittivity. Compositions with a tunability of 19 - 38 % for relative permittivities between 130 - 160 at 25 GHz, and with a dissipation close to that of pure barium-strontium-titanate compositions and a firing temperature of ~ 900 °C were obtained. These compositions were utilized in several multilayer components e.g. phase shifters, matching networks and power combiners within the EU IST MELODY project (coordinated by Ericsson in Sweden) with various partners e.g. Chalmers University of Technology in Sweden, the University of Birmingham in the UK, Filtronic Comtek in the UK and Ecole Polytechnique Federale de Lausanne in Switzerland. Research has also been started on the development of piezoelectric LTCC compositions for actuators, sensors, microphones and micropumps. Test structures with the combination of piezoelectric, ferromagnetic, ferroelectric and dielectric layers in the same modules were also prepared, aiming for future multifunctional smart packages. The results were disseminated at international conferences (PacRim, Japan, EuMW, Germany, ACES meeting, USA) and in international scientific journals (JECes, IEEE etc.).

Materials characterization (dielectric permittivity & loss tangent) of commercial LTCC ceramics was continued and performed together with the St. Petersburg Electrotechnical University (Russia) and the Institute of Physics (Czech Republic) enabling as wide a frequency range as from kHz up to THz. An important research area has also been high frequency and reliability studies of LTCC/BGA/PCB interconnections. The signal behavior in coplanar BGA, flip-chip and via structures was modeled using Sonnet and Ansoft HFSS software (in co-operation with the Technical University of Ilmenau (Germany), supported by the Academy of Finland and DAAD). A 3D integrated BGA filter module and an LNA amplifier module were designed and manufactured in co-operation with industry and VTT. The measured results showed good electrical performance for both modules and correlated very well with the EM simulations.

The results of lead-free reliability investigations on LTCC/PCB assemblies were accomplished, were novel BGA solder joints with a compliant plastic core were demonstrated for use in high-performance microwave modules. In addition, thermo-mechanical FEM analyses were performed for the developed BGA module assemblies.

Exploitation of Results

Materials, components and technologies developed by the group are already widely applied in the national electronics industry, especially in the mobile phone industry. As an important example of the present exploitation, LTCC micro modules for telecommunication...
tion applications and ceramic MEMS modules must be mentioned. In 2005, emphasis has been laid on a continuous extension of our recent scientific achievements also in the field of nanotechnology with integrated nanostructured assemblies for electronics, biotechnology/medicine, photonics, and catalyst systems.

**Future Goals**

The long term research of the EMPART research group will continue with the focus areas of electronics materials, thin films, components, packaging and reliability techniques. A growing research area of the group is LTCC micro modules for MOEMS applications in telecommunication, instrumentation and medical fields. Co-operation with other experts at the University of Oulu in the field of nanomaterials and their applications will be increased. The new Micro and Nanotechnology Centre with advanced manufacturing facilities offers excellent opportunities for multidisciplinary research. The group has a key role in the micro and nanotechnology research of the Oulu region. The EMPART group will form a high excellence research and education organization in the field of micromodule and nanotechnologies.

**Personnel**

<table>
<thead>
<tr>
<th>Category</th>
<th>Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>professors &amp; doctors</td>
<td>14</td>
</tr>
<tr>
<td>graduate students</td>
<td>23</td>
</tr>
<tr>
<td>others</td>
<td>1</td>
</tr>
<tr>
<td><strong>total</strong></td>
<td>38</td>
</tr>
<tr>
<td>person years</td>
<td>27</td>
</tr>
</tbody>
</table>

**External Funding**

<table>
<thead>
<tr>
<th>Source</th>
<th>EUR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Academy of Finland</td>
<td>118 000</td>
</tr>
<tr>
<td>Ministry of Education</td>
<td>103 000</td>
</tr>
<tr>
<td>Tekes</td>
<td>956 000</td>
</tr>
<tr>
<td>other domestic public</td>
<td>48 000</td>
</tr>
<tr>
<td>domestic private</td>
<td>134 000</td>
</tr>
<tr>
<td>EU + other international</td>
<td>36 000</td>
</tr>
<tr>
<td><strong>total</strong></td>
<td>1 395 000</td>
</tr>
</tbody>
</table>

**Doctoral Theses**


**Selected Publications**


