VTT Technical Research Centre of Finland is the largest multitechnological applied research organisation in Northern Europe. VTT provides high-end technology solutions and innovation services. From its wide knowledge base, VTT can combine different technologies, create new innovations and a substantial range of world class technologies and applied research services thus improving its clients’ competitiveness and competence. Through its international scientific and technology network, VTT can produce information, upgrade technology knowledge, create business intelligence and value added to its stakeholders. VTT is a non-profit-making research organisation.

Research and development activities in printed intelligence

2008 2009 2010 2011
Durable dynamic images by hot embossing in printing line

Read more page 20
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Towards the Commercialisation of Research Efforts

Printed intelligence technologies are enabling disruptive innovations and new business opportunities.

Both the business community and the society at large are expected to benefit from the new technological possibilities brewing in laboratories for printed electronics and organic electronics around the world. Unfortunately, the technology and products are not yet advanced enough to have provided a boost in the current economic context.

However, the tough economic times are actually having a positive impact on this field. In part, they have helped lower some of the hype surrounding the industry, and switched the focus to short-term possibilities and getting the products to market. While some have had to reduce or completely cease activities in printed electronics, others have redoubled their efforts. In the search for short to medium-term business opportunities, companies have increasingly shifted their focus to leveraging existing technical capabilities and developing feasible products based on these capabilities. In the realm of applied research, and for institutes like VTT, this is resulting in more projects serving the short-to-medium term R&D needs of companies.

We are stretching the boundaries of electronics to new types of intelligent solutions that utilise novel printed components – which may have relatively little to do with electronics as we know it. Those familiar with VTT know that we have coined the term printed intelligence to refer to this broad opportunity for disruptive innovations.

This review is a collection of extended abstracts of the most important public research and development results in printed intelligence technologies at VTT during 2008 and the first half of 2009.

EXAMPLE OF AN EMERGING DISRUPTIVE TECHNOLOGY

Printed intelligence is based on printed components and systems that:

- extend the functions of printed matter, and
- perform actions as a part of functional products or wider information systems

Printed intelligence has the potential to disrupt various industries, blur the boundaries between existing industries and create totally new markets. Let’s take, for example, the lighting industry and OLED technology.

The history of the lighting industry is full of disruptions enabled by technical innovations. As an industry, it progressed and evolved from candles and kerosene lamps to incandescent lamps, then to fluorescent lamps, and finally, to the rapid adoption of LED technology. All of these technological advances have enabled new applications...
for lighting and in turn have expanded the entire lighting market, bringing wealth to the commercial enterprises that have embraced these technologies to increase their selection of products and solutions.

The next lighting industry disruption is bubbling with OLED technology. The potential impacts of this technology are broader than one first would expect, even if OLEDs are only considered a sustaining innovation. LED lights have opened up novel possibilities for lighting design, and allowed for the application of light in places previously unheard of (e.g. LEDs are now found in stickers, greeting cards, and even retail packaging). They are an important component in most electronics devices, further blurring the line between the lighting and electronics industries.

So what more could OLEDs possibly do – than what is already being trialled in market with LEDs? And what does printed intelligence have to do with it?

With the advantages of extremely low power consumption, flexible large area surfaces and non point sources of light, OLED technology is aiming to do things even better than LED. Wide public exposure of developments with OLED in the lighting, display and even signage industries are evidence of that. Beyond this, the high volume printing of OLEDs (with disposable materials) will shed light on places where light-emitting components have never gone before, or places that may not have been viable on a larger scale with LEDs.

Eager to hear what the future will hold for such applications? We have to leave it to your imagination, for now. The first applications of printed OLEDs have entered product development.

FROM THE LABS...

Printed OLEDs is of course only one example of the printed intelligence technology that is starting to emerge from the laboratories. Many other new technological solutions can be found in this booklet.

This review covers the work and investments made within VTT’s strategic initiative, the Centre for Printed Intelligence. Over the past three years, VTT has doubled both its annual research efforts (which now exceed 100 person years) and revenues from printed intelligence.

In order to realise truly novel solutions, VTT has taken a strong multi-disciplinary approach in its printed intelligence developments. Expertise in e.g. in biotechnology, paper, electronics etc. are combined in our daily projects and researcher interactions. The diverse research backgrounds of the authors of the articles are evidence of that.

VTT has also systematically made groundbreaking investments in its printed intelligence equipment and facilities, particularly with roll-to-roll, printing and coating lines. Our larger scale investments started with the rotogravure and hot-embossing machine PICO (at near full operating capacity since 2003), the ROKO machine with 4 replaceable printing units (2007), the pilot coating line (2008), and new process equipment instalments in 2009 and 2010.

In addition, we have continuously worked to make our knowledge and experience of material process interfaces a core strength. We have developed printed components and integrated them to systems and devices.

We have actively participated in international networks, through publicly funded projects, industry associations (the Organic Electronic Association and the Plastic Electronics Foundation), as well as work with multi-party research and product development efforts. We have been particularly active within the European Commission framework program 7th research area ‘Organic and Large Area Electronics,’ where we are proud to be among the most visible contributors.

...TO MARKETS

While the bulk of printed intelligence work at VTT has been aimed at developing generic technologies, materials and processes, we have simultaneously aimed market and application development efforts at business arenas with high volume applications, namely:

- Consumer packaged goods
- Media & ICT services
- Bioactive paper & diagnostics

In all our research work the question of intellectual property, business potential and steps to commercialisation are addressed from the early stages of development. We work throughout the value chains in each of the business arenas addressing both demand and supply factors.

One example of our work in linking market needs with emerging technology supply is the recent initiation of the Interactive Packaging Affiliate Program by VTT. With the target of adding value to consumer interactions through packaging, this Affiliate Program brings together fast-moving consumer goods companies to share experiences with smart/interactive packaging technologies,

...
learn about emerging technologies, provide requirements and feedback on developments and potentially initiate joint market trials with new technologies.

As previously mentioned, an increasing share of our work goes to development projects with companies. Confidentiality is a priority in our work with our customers. In this review, we are honoured to be able to present the results of work carried with one of our key customers, Orion Diagnostica, and briefly introduce their printed diagnostics product (available on the market), the Orion Clean Card PRO.

Other notable developments in commercialisation include the research collaboration with BASF, which covers areas of printed organic electronics in the spirit of open and collaborative innovation, and new printed functionalities in high-volume packaging and diagnostics.

In 2009, VTT initiated PrintoCent (the Printed Electronics and Optical Measurements Innovation Centre), an innovation program and environment aimed at taking technologies from lab-to-fab to markets. PrintoCent creates a business, production, research and educational environment for companies to develop and manufacture prototype products, demonstrators and system solutions, and acquires a skilled workforce to enable such developments. This community includes cooperation with companies utilising resources at VTT, University of Oulu, and Oulu University of Applied Sciences. Annual R&D projects in PrintoCent will exceed 15 million euros, and within the program we are establishing a printed electronics application design environment and pilot factory, for companies to develop and manufacture prototype products and demonstrators.

PRINTED INTELLIGENCE COMMERCIALISATION

According to market forecasts, ‘printed electronics’ will generate more than 250 billion dollars by 2025 (sources: IDTechEx, Frost&Sullivan). Today we are still in the very early stages of entering the market and identifying commercial uses for the simplest technological solutions.

VTT strongly believes in the emergence of new printed intelligence markets and therefore, we continue to strongly contribute to the development of technologies, solutions and applications in this field. We are strong believers in the power of collaboration and relentlessly working to build stronger and stronger consortia both with research and industry. Ultimately the printed intelligence markets are being driven by new start-ups and spin-offs, as well as existing enterprises looking to expand their markets and add value to their products. VTT supplies services and technologies to industry leading companies.

VTT wants to also proactively participate in closing the existing gap between technology and market application and business needs, and to more actively help drive the transition from laboratories to commercial solutions. For this purpose VTT is establishing a printed intelligence commercialisation program (starting 2010). The aim of this program is to increase business development efforts aimed at commercialising new innovations and creating new businesses.

ACKNOWLEDGEMENTS

Tekes, European Commission, Nedo, other funding organisations and our industrial and research partners are highly acknowledged for their funding, collaboration and joint efforts. Without these parties, we would not be able to present the work found in this booklet.

We hope this report encourages innovative companies and people with the entrepreneurial spirit to continue to actively approach us to learn about these emerging technological possibilities and collaborate in taking them to commercial use.

We wish you inspiring readings and warmly invite you to further discuss any and all of the topics of interest to you.

October 2009
VTT Center for Printed Intelligence has established active international R&D collaboration networks. Below is a sampling of these networks.

Europe has been the major region for VTT’s international collaboration in printed intelligence – in terms of volume of activity. The European Commission (EC) coordinated funding for ‘Organic and Large area electronics’ (OLAÉ) in its 6th and 7th framework programs has opened concrete research project collaboration with several major research institutes and universities like Fraunhofer, CSEM, INM, CEA, IMEC, Acreo, Holst Centre, Joanneum, TU Dresden, University of Cambridge and many others. In this report we cover several 7th framework projects in more detail. FP7 Quadriga projects PolyNet, Opera, Prodi and Polymap are the core networks created for OLAÉ co-operation and forming the bases for the OLAÉ technology platform in Europe. EC has been actively encouraging and supporting European efforts, industry and academia joint actions towards coordinated European Strategic Research Agenda in OLAÉ for securing the development of strong European position in this new emerging enabling technologies area. We feel ourselves privileged while operating and contributing in these networks for building strong European technology backbone and business opportunities for our industries.

One example of a special effort in Europe is a project between VTT and the region of the Navarre in Spain to identify actions to generate new business for industry in the Navarre region from printed intelligence. A centre of excellence for printed intelligence is to be built in the Navarre region. VTT is delivering a roadmap study on printed intelligence to the Asociación de la Industria Navarra for this purpose. The study outlines what kind of expertise will be required for research and development in printed intelligence in the future and what kind of applications are to be expected in selected branches of industry. The new centre of excellence is expected to generate significant new business while supporting sectors in which the region is already strong, i.e. the food industry, medicine and renewable energy. The work features three future scenarios for each of the selected sectors, a listing of the technologies that best fit each of these scenarios and their feasibility for commercial use.

Institute of Industrial Science of the University of Tokyo and VTT Center for Printed Intelligence have jointly opened a technology development initiative for roll-to-roll fabricated large area flexible MEMS. The first research topic has been ‘Large area flexible MEMS-display’. A roll-to-roll fabrication process for a Fabry-Perot principle based display elements have been developed and demonstrated with a multi-color array of display pixels. We are also looking for wider application opportunities for flexible MEMS devices.

VTT and Konkuk University in South Korea have research collaboration in the roll-to-roll technology research and development for passive electrical components like resistors, capacitors and inductors and their integration as circuits for flexible electronics applications. These contents include material issues, machinery developments and characterisation for high-quality components and circuits.

Collaboration with the Canadian SENTINEL-network on ‘Bioactive paper’ and Finnish bioactive paper consortium started with discussions in 2005. A milestone event in the development of bioactive paper was the First International BioActive Paper Conference organised in June 2008 in Espoo. The event brought together approximately 80 specialists from the Canadian and Finnish networks. Since then plans for mutual projects have progressed, and first mutual studies have been started in June 2009.

VTT together with the University of Oulu and Kurchatov Institute, St. Petersburg State University and Russian Academy of Science in Russia have research collaboration in the new selective gas sensors based on printed semiconductor nanoparticles. These contents include material issues, machinery developments and characterisation for high-quality components and circuits.
As part of achieving a critical mass in organic and large area electronics, there is a subset of four FP7 EU-funded projects (PolyNet, OPERA, PRODI and PolyMap) that cover actions aimed at defining European competencies, services and industrial requirements in the field; the goal is to increase ease and foster the establishment of competitive clusters throughout Europe as well as to reinforce the European position in the area.

The four projects are often referred to as Quadriga, since there is a wide variety of joint activities organised and coordinated by the group. In practice, the coordinators of the individual projects play key roles in practical collaboration arrangements. These include review meetings, event calendars, participation in OLAE stakeholder groups organised by the EC, and program work. VTT is the coordinator of PRODI, and the co-coordinator of OPERA.

PolyNet is a Network of Excellence that aims to establish an area of organic and large area electronics in Europe, making it the world leader in science, technology and the subsequent commercial exploitation of printing and large-area electronic technologies for the hetero-integration of flexible electronics.

Industrial exploitation in this area needs a research cooperation and service base to foster the transfer from science to industry within Europe. PolyNet will support these aims with three core platforms: a research cooperation platform; a service platform and a knowledge platform.

The overall objective of the Coordination Action OPERA is to strengthen the position of Europe as a leading force in organic electronics in the world. One specific aim of OPERA is to create the conditions for establishing a number of competitive clusters in Europe. To achieve these goals, OPERA will work to develop a strategic framework that maximises synergy and cooperation in the sector; accelerate technological progress and the development of commercial organic electronic applications; create channels for exchanges of ideas and people; develop tools for stimulating entrepreneurship; accelerate the development of industry standards and enhance the visibility of the field.
The intention of the Coordination Action PRODI is to integrate European printing, coating and other advanced processing machinery manufacturers, production line integrators and process measurement and automation industry to work together to improve European excellence in roll-to-roll polymer and printed manufacturing equipment and production line business.

The objectives of PRODI involve identifying the requirements for the new manufacturing machinery, measurements and automation systems, and generating a common future vision for the industry on R2R polymer and printed electronics manufacturing equipment and production lines and systems.

PolyMap is a Support Action that aims to strengthen Europe’s position as the leading force in organic electronics.

For that purpose, PolyMap will map public funding in organic and large area electronics, set up an ERA-NET, create a Wikipedia-type database especially aimed at materials and new applications, and support SMEs in this area.
Orion Clean Card PRO, Roll-to-roll Manufactured Test for Hygiene Control

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Roll-to-roll manufacturing provides a cost-effective and high-volume method for producing disposable and easy-to-use environmental monitoring tests. The first series to commercialise roll-to-roll-test for rapid environmental monitoring, Orion Clean Card PRO, provides a user-friendly, accurate test for hygiene control.

INTRODUCTION
Environmental diagnostic tests contain a number of different methodologies to monitor chemical substances and microbiological species. The general requirements for these types of tests include simplicity, ease-of-use, fast detection and a low price. Conventional tests are typically filter-paper analysis involving a number of different chemical substances that generally make the test expensive, hard to perform by non-professional users, and too complex for continuous monitoring.

The roll-to-roll fabrication of wiping type rapid environmental diagnostic tests has a number of advantages over traditional environmental monitoring test kits. First of all, roll-to-roll manufacturing makes it possible to produce tests cost-effectively and in the volume required by continuous monitoring. Secondly, wiping tests are relatively simple, making it possible and easy to be used by non-professional users as well.

TECHNOLOGY
In the Orion Clean Card PRO test, proteins are detected in a tissue upon which required chemicals are printed. Therefore, test development started by transferring and optimising existing test chemistry from in vitro to tissue and resolving sensitivity, stability and production problems. After successful chemistry optimisation, technologies were transferred and optimised to roll-to-roll production. In this phase, VTT’s research and pilot production scale equipment were widely used. The last phase of technology development was the technology transfer from VTT to subcontractors.

Production scale fabrication of the Orion Clean Card PRO hygiene monitoring test involves a number of different roll-to-roll production technologies. Printing of indication reagents on fabric can be done either by gravure- or flexographic printing. Building up a test that is ready for use also requires other techniques such as lamination and die-cutting.

ORION CLEAN CARD PRO
Orion Clean Card PRO is the first commercialised roll-to-roll manufactured test in a series of rapid environmental diagnostic tests. The Orion Clean Card PRO protein test performs similarly to the conventional protein test. Thus Orion Clean Card PRO provides a user-friendly accurate test for hygiene control.

For several years, VTT has been developing roll-to-roll methodologies for the manufacture of swapping type tests for environmental diagnostics for Orion Diagnostica.
The BioFace project aims to develop new tailored sensing surfaces to be used in printable biosensors. Engineered, extremely stable avidins are the ideal biomolecules to obtain sensing material that is thermally and chemically resistant and apt for applications in the biosensing field. Through covalent linkage or by acting as intermediates, functionalised polymers allow efficient immobilisation of the tailored avidins, which are printed on the sensor chip substrate. The developed materials can be used in bioanalytical devices in the field of diagnostics, drug research and life science research.

INTRODUCTION

One of the future key areas in the field of point-of-care diagnostics is the use of mass production methods for low-cost, disposable biosensor platforms. The sensing layer of the sensor must fulfil the requirements of reproducibility, stability, sensitivity and selectivity. To meet the demands for bioactive sensing layers, new materials have to be developed which allow high-volume, quantitative, multi-analyte point-of-care test platforms to be manufactured. The BioFace project aims to produce new solutions for tailored sensing materials that can be integrated in the manufacturing process of printable sensors. The project has three research parties: the University of Tampere (Institute of Medical Technology, IMT), the University of Oulu (Department of Chemistry, UO) and VTT. UO and VTT develop functionalised materials and IMT produces the biomolecules.

MATERIALS AND METHODS

Novel polyalcohol-modified silane precursors are synthesised from alkoxysilanes and polyalcohols using standard synthetic methods. The generated compounds are characterised with chromatographic and spectroscopic methods. The attained precursors are then reacted to form sol-gels. Mild reaction conditions are used, adding only water in the mixture of the precursors and propagating the reaction at room temperature. The manufactured sol-gels are characterised with spectroscopic and chromatographic methods including NMR, ATR-IR and GPC methods. The sol-gels obtained are then used as intermediates for biomolecules.

The biomolecules used in this project are stabilised chimeric avidins (Figure 1). The biomolecules are doped into the sol-gel in order to form a protective sol-gel layer around the biomolecules.

The printability of the sol-gel with doped chimeric avidin is tested in the lab scale. Primarily, the liquid is applied with a control coater applicator on plastic substrate (mostly PMMA polymethyl methacrylate) and the coating is cured in an oven.

Another way to immobilise the chimeric avidin on the sensor surface is to covalently link activated carboxylic acid molecules of the polymeric substrate with amino groups of the biomolecules. Carboxylic acid molecules are activated by EDC/NHS (1-ethyl-3-(3-dimethylaminopropyl) carbodiimide/N-hydroxysuccinimide) linking chemistry. Reactive COOH groups are derived from the co-polymer PMMA-co-PMAA (polymethyl methacrylate-co-polymer acrylic acid) and NH2 groups from chimeric avidin. The co-polymer is gravure printed in lab scale on the plastic substrate and the layer is cured. The chimeric avidin is applied on the surface. After some time, the reaction is quenched with ethanolamine and the substrate surface washed with buffer. The optimum amount of linker and chimeric avidin is examined along with the ratio of linker, avidin and the reactive groups of the substrate.

The bioactivity of the immobilised biomolecules is verified with the fluorescence immunoassay method. Biotinylated anti-CRP is attached in various concentrations to the chimeric avidin and detected with Alexa 488 goat anti mouse IgG. Chimeric avidin molecules which are passively coated on plastic substrate are used as control.

RESULTS

In the development of biocompatible materials, building blocks have to be adjusted in terms of both chemistry and biochemistry. Biocompatibility of the developed material is a major factor. Another important consideration is the repeatability of the manufacturing process of the chemical material. Other necessary properties include stability and easy handling of the material. With sol-gel materials,
the polymerisation degree of the sol-gel plays an important role. This means assessing the timescale for the insertion of the biomolecules in the forming Si-O-Si cage, including the so-called aging time of the sol-gel as well. For printing purposes, wetting properties are considered, including the pre-treatment of the polymeric substrate and additives in the ink. Proper adhesion of the homogeneously spread bioactive layer on the plastic substrate is essential to ensure that results are accurate.

In the BioFace project, it was verified that polyalcohol modified silane precursors (Figure 2) can be synthesised with a repeatable process. In parallel synthesis, similar materials were attained according to NMR spectral and TL chromatographic analysis. Sol-gels manufactured from each of the precursor batches behaved in the same way with one other; these were used for the entrapment of chimeric avidin.

In terms of the printability of the manufactured sol-gels, water-based inks were noted to smoothly coat plastic substrates with the help of additives. Proper adhesion to the substrate can also be attained with oxygen plasma etching.

One challenge involves modifying the porosity of the sol-gel to make it ideal for the entrapment of chimeric avidin. This allows the sol-gel to protect the biomolecules from environmental strain but leaves the active parts of the chimeric avidin molecules available for selective recognition and detection.

In the other approach, chimeric avidin is immobilised by covalent linking with the activated substrate polymer. The level of immobilisation of chimeric avidin is the same when both covalently linked and passively coated on plastic substrate. Shelf life tests will show if covalent linking is superior to passive coating.

SUMMARY
The BioFace project is developing generic printable biosensor surface materials which are able to fulfil the requirements of reproducibility, sensitivity and stability of POC test platforms. The developed materials will be used for an industrialised, simple and cost-effective production method for generic bioactive surfaces that are suitable for use in different biosensing applications.

BUSINESS POTENTIAL
The methodology for printable diagnostic tools will be applied to various diagnostic tasks. Detecting targets range from small molecules (drugs, hormones) to microbes (bacteria, viruses). The potential of the developed methodology is related to the versatility of the platform, which makes it possible to employ the avidin-functionalised material for a broad range of targets.

REFERENCES

ACKNOWLEDGEMENTS
BioFace is funded by the Tekes Functional Materials program, VTT, University of Tampere, University of Oulu and the companies Orion Diagnostica Oy, BASF (Ciba Finland Oy), Oy Medix Biochemica Ab and Next Biomed Technologies NBT Oy.
Introducing polymer-based microfluidic chips which can be mass-manufactured by roll-to-roll (R2R) printing methods. Hot-embossed microfluidic channels with variable shapes and dimensions are suitable for use in different types of diagnostic applications. In the Finnish collaboration project Welfare2, chips and methods for immunoassay detection have been developed. Capillary electrophoresis chip (CE) for transcriptional analysis was developed in collaboration with University of California Berkeley in QB3-project.

Introduction
Low cost, miniaturised and mass-manufactured point-of-care solutions are of great interest for diagnostic research and industry. VTT is rising to this challenge by means of roll-to-roll print technology. Microfluidic biosensors enable rapid assay performance in many application fields and thus offer advantages over many traditional methods. We have put together special expertise in the area of microfabrication, sensing methodology, materials, optics and modelling to develop new analytical platforms.

In the Welfare2 project, we have concentrated on the development of a microfluidic biosensor platform suitable for use in the measurement of fluorescence-based immunoassays directed towards the point-of-care diagnostic field.

The QB3-project aims to transfer microfluidic chips made by etching on glass to polymer devices fabricated by roll-to-roll manufacturing methods. The transfer from one manufacturing method to another raises questions for the development of roll-to-roll fabrication, such as how to create and align multilayered structures with features in the micrometer range. The differences of glass and polymer materials also bring challenges to the project work: dissimilar surface chemistry and the optical quality of the plastics require assay development to adapt the biochemistry to the novel analytical platform.

Materials and Methods
Microfluidic features were added to plastic materials by hot-embossing and lamination technologies. Both flat-bed and roll-to-roll hot-embossing have been studied. Inkjet-printing has been used to print capture antibodies on the surface of microfluidic channels. CRP (C-Reactive Protein) has been used as a model analyte in immunoassay development.

Results
Immunoassays:
Microfluidic channels (mould dimensions: 700 µm wide, 40 µm deep and 3 cm long) were obtained with flat bed or roll-to-roll hot-embossing. CRP-antigen doped in bovine serum in the range of 0.5-2 µg/mL (the reference value for sensitive CRP is < 2.6 µg/mL) was detected with a CCD-camera based fluorometer, which was construct-

Figure 1. A capillary electrophoresis chip with hot-embossed microfluidic features.
ed in the Welfare2 project. Fluorescence intensities were calculated with a Fluorescence Intensity calculation program in the Matlab environment.

**Capillary electrophoresis:**
The R2R hot-embossed CE channels were 150µm wide, 30µm deep and 16cm long. 40µm deep channels could be achieved by flat bed hot-embossing; the stamp height was 40µm in both methods. Both the optics and surface quality of the polymer chips were good. The TRAC (transcriptional analysis with the aid of affinity capture) assay is being transferred to chip format in close collaboration between UC Berkeley and VTT. Current research activities within the project are focused on developing a printing environment for multilayer alignment and lamination, and on matching the sensitivity of the microfluidic assay with the level of the analyser currently in use.

**SUMMARY**
In the Welfare2 and QB3 projects, methods for manufacturing diagnostic polymer microfluidic chips by roll-to-roll methods were developed. This method enables the low-cost production of highly versatile chips which can be used in a broad range of diagnostic applications.

**BUSINESS POTENTIAL**
The roll-to-roll printing technology is a strategic research investment from VTT’s side as it drastically reduces the diagnostic chip price and manufacturing times. These are both necessary for the large scale utilisation of biochips in different analytical systems (e.g. diagnostics, environment, food safety testing). The pilot printing facility at VTT is able to produce thousands of microfluidic features per day, which is significantly more than that produced by etching onto glass or silicon. The reduction in cost of a microfluidic chip enables disposability, which is one of the key elements in point-of-care diagnostics.

**ACKNOWLEDGEMENTS**
The Welfare2 project has been jointly funded by the TEKES FinnWell program, VTT and industrial partners (Orion Diagnostica Oy, Nokia Oyj, Magnasense Oy, Braggone Oy, Comsol Oy). It has been performed in collaboration with University of Oulu.

The QB3 project is jointly funded by TEKES, VTT and industrial partners (Orion Diagnostica Oy, Medifju Health-care Oy, Ciba Finland Oy, Lahmaster Oy, Oy Panimolaboratorio, Mobidiag Oy, Glykos Finland Oy, Zora Biosciences Oy, PlexPress Oy, KKI Kaartinen Tutkimus Oy). It is performed in collaboration with University of California Berkeley.
Printed Enzymatic Power Supply with Integrated Capacitor

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Printed electronics with integrated power sources have remarkable potential in several mass-marketed consumer products e.g. as package integrated functionalities (sensors, displays, entertaining features, etc.). One of the main requirements is that the power and its package must be recyclable without special treatment. The main goal of our research has been to meet these demands in a printable fully enzymatic biofuel cell that is a suitable power source for e.g. an active RFID tag.

INTRODUCTION
Printed electronics will be integrated to many mass-marketed consumer products e.g. as package-integrated functionalities. The power source and package should be recyclable without special treatment; production costs should also be reasonable. As an alternative power source, the miniaturized biological fuel cell has the potential to meet these demands. The low peak current capacity of enzymatic fuel cells can be improved by integrating the cell to a printed capacitor. The main goal of our research is to develop a printable, fully enzymatic biofuel cell that utilises enzymes as the catalyst on both cathode and anode electrodes. New printable functional materials can be used in several application areas like displays, sensors, power sources and printed RFIDs. The aim of developing a power supply of this kind is to meet the demands of applications such as active RFID tags.

MATERIALS AND METHODS
Biofuel cells are devices capable of directly transforming the energy within chemicals to electrical via enzymatic catalysis [1, 2]. At the bioanode the fuel, such as sugar or alcohol, is oxidised with the help of a suitable oxidoreductase enzyme and the electrons are transferred to the anodic electrode. At the biocathode, the electrons are then transferred to the electron acceptor, typically dioxygen or peroxide, through an enzymatic reaction. The work carried out at VTT focuses especially on the construction of printable enzyme electrodes. The cathode electrode uses fungal laccases as biocatalysts. Bacterial dehydrogenases and oxidases are applied as biocatalysts for the anode half cell, where two feasible enzyme/mediator combinations have been identified.

The first challenge encountered with the enzymatic electrodes related to maintaining enzymatic activity in the printable, conductive ink. Most conductive inks are based on various solvents, which are often harmful for the stability and catalytic activity of enzymes. Suitable water-soluble inks from commercial sources were thus screened and further optimised or experimentally developed in order to obtain printed enzyme electrodes with optimal performance as well as satisfactory electrochemical properties.
RESULTS
Enzymatic activity can be maintained for up to months in different conductive inks depending on the storage conditions. The cell can be activated by adding moisture (electrolyte) [3]. A film that is both moisture impermeable and oxygen permeable is capable of sealing the laccase-containing fuel cell. A sealed fuel cell is able to generate power for several days [4]. It was also successfully demonstrated that biofuel cells can be manufactured at an industrial scale by utilising silk-screen printing to produce the enzymatically active layers. The other functional parts of the fuel cell, like current collectors and separators, could also be produced with the processes used in paper converting and paper manufacturing. The results obtained with the printed fuel cells were comparable to those obtained with hand-made prototypes in both current producing capability and in the uniformity of the quality of the produced cells. The cells have also expressed a stable performance in the tests with the RFID simulator. Three serially connected cells are capable of powering a tag for 3-4 days.

SUMMARY
Printable electrodes based on a biocatalyst could offer an inexpensive way to mass-produce disposable devices such as biosensors and power sources based on biofuel cells. The non-toxicity of materials is also important in the printed components. By using suitable conductive inks, enzymatic activity can be maintained in the printed layer. It was also demonstrated that biofuel cells can be manufactured at an industrial scale by utilizing silk screen printing. The low peak current capacity of enzymatic fuel cells can be improved by integrating the cell to a printed capacitor. Efforts are currently being made to improve the selection of materials and redesign the configuration to further develop printed capacitors.

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ACKNOWLEDGEMENTS

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Under VTT’s direction, new methods are being developed for the economical mass production of bioactive paper products, among others based on printing technology. Publicly funded projects started in 2007 and will continue through 2011. The goal is to create basic concepts and generic technological know-how for developing various bioactive paper product applications, such as test paper slips that reveal allergens in swimming or drinking water.

INTRODUCTION

Bioactive paper is a product that includes functionalities based on the selective reactions of biomolecules, such as enzymes or antibodies. The application possibilities are extremely broad, and include indicators or sensors attached to filters, food product packaging or personal health diagnostics, all of which would be cheaper than current products. In printed intelligence applications, the paper’s competitiveness lies in the fact that it is biodegradable, which is important in terms of sustainable development.

A research project started in 2007 in order to gather basic knowledge and create technologies that enable the production of intelligent fibre-based products in a cost-efficient way. In this project, more than 250 application concept ideas were visualised by industrial design students at the University of Lapland. Laboratory scale demonstrators were developed for selected applications. Developing the demonstrators required:

• A paper network with controlled flow characteristics
• Methods to link biomolecules on fibres
• Biomolecule-compatible printing inks
• Biomolecule-compatible paper coating recipes
• A preliminary outline for the electrical detection of biochemical reaction

For 2009-2011, the targets for development include generic technological know-how for various bioactive paper product applications. This includes processes for the laboratory scale methods used in the above mentioned demonstrators –like manufacturing methods. Another target is to develop systems which allow multiple reactions from one sample to be gauged with a single test. The development of quantitative systems with electrical detection will continue. The feasibility of the developed product concepts will be tested in “real life”, and the possibilities to build services in connection with the products will be clarified. Market acceptance and marketing methods for completely new types of products will also be developed.

The work utilises forest and bioindustrial knowledge, and it creates potential for new products in both industrial areas. The goal is to use and develop paper’s strength as a material, as well as to create new business for the paper industry and consolidate existing business.

The project led by VTT involves a network of research partners, including Åbo Akademi, TKK and the University of Lapland. It has been funded by Tekes, VTT, research partners and eight industrial companies (UPM-Kymmene Oyj, Tervakoski Oy, Ciba Specialty Chemicals Oy, Hansaprint Oy, Oy Medix Biochemica Ab, Orion Diagnostica Oy, Eagle Filters Oy, Starcke Oy Securities).
Hot embossing is a general purpose production technology that has several application areas, including optical, mechanical and even electrical structures. In this work, one specific optical application area – dynamic graphics on packaging materials – was investigated. Hot embossing was proven to produce environmentally friendly and dynamic decorative patterns on packaging materials and it can be integrated into a printing machine environment.

INTRODUCTION

Hot embossing is a general production technology that can be used for many different end uses, including to produce optical effects on a nanoscale, to make channels for microfluidistics on a microscale and to make surface forms on packaging on a macroscale.

Nanoscale hot embossing is similar to nanoimprinting technologies. Both technologies use a tool that has a nanoscale patterned surface and the tool is pressed on a substrate to copy the pattern on the tool to the substrate. The difference is that embossing is done on surfaces of several square meters, while nanoimprint is typically applied to areas measuring a few square millimetres. The impact time of roll-to-roll embossing is a few milliseconds when the speed of the web is hundreds of meters per minute. The general impact time of nanoimprint is a few minutes. Nanoimprint aims to make top-quality nanoscale electronic structures. Roll-to-roll embossing is used in applications where larger surfaces with structures of a lower quality are acceptable.

MATERIALS AND METHODS

In this work of embossing dynamic optical effects for packages, both paper and plastic packaging materials were used. The special focus was on VTT-developed starch coatings that aim to reduce mineral coatings on papers. VTT has several patents in this area. In addition to coatings, VTT has formulated inks based on starch.

The embossing properties of these new materials were studied. Another focus area involved formulating protective coatings for nanoscale patterns.

Dynamic optical effects were chosen from a library of existing designs. The embossing plate was produced using a normal electroplating method and formed as a sleeve by laser welding.

Embossing tests were initially done in the laboratory with a flat bed machine and then with a pilot machine containing two printing units and an embossing unit. The web width is 200 mm and the maximum speed of the machine is 100 m/min. The embossing unit has two cylinders: a heated embossing cylinder and a backing cylinder with a very smooth surface. The sleeve is installed on the embossing cylinder.

The embossing cylinder is heated to over 100°C and the two cylinders are tightly pressed together. The substrate (paper, plastics) goes through this nip and the nano patterns are copied from the sleeve to the surface of the substrate.

Because the nanoscale optical structures are sensitive, a special protective coating is required. Several variations of the basic coating formulation were prepared and tested. In addition, two coating principles were tested: the coating was applied either before embossing or after embossing.
To control the embossing process, some measurements are required. The embossing pressure is measured on both ends of the cylinder and the temperature is measured near the surface of the cylinder. The quality of the embossing is analyzed by using test gratings on the edges of the web. These are measured by a laser device developed at VTT.

RESULTS
Dynamic optical effects were produced in high quantities on different paper and plastic materials. These optical effects produce dynamic colour changes when the surface is viewed from different angles. The quality of the optical effects is high enough to be viewed by the naked eye even without metal coatings.

The clear optical effects are destroyed if rubbed with a finger. When the protective coating was applied, the surface withstood rubbing. The protective coating can be applied before or after embossing. Both methods provide a working solution, depending on the specific application. The protective coating can require tuning depending on the base material.

The embossing of mineral coated papers produces poor results. In this work, the embossing of biodegradable starch-based coatings and inks produced good optical effects. To get the right formulation of the starch coating for a specific base material, tuning is required. Now it is possible to obtain nice decorative surfaces and be environmentally conscious at the same time. In this area, VTT has patents pending.

In this work, existing holographic technologies were combined with proprietary developments. A lot of technologies are already in use in hologram production: nickel plates are made by electroplating and narrow slow speed embossing units are used. Normally vacuum coaters are used to make metal or highly refractive index coatings. VTT wanted to show that the embossing technology can be integrated into a printing machine. Three items are important in reaching this goal: solution-based protective coating, embossing sleeve and process control. When the protective coating can be applied in the printing machine, there is no need for vacuum coating. The embossing sleeve technology permits high speed production. Process control is important to reach top quality in high speed production.

SUMMARY
In this work, an approach for making dynamic graphics on packages was studied. This approach is possible for normal packaging and printing materials. There is no need for holographic labels or foils. Both transparent and non-transparent substrates can be used. The approach is environmentally friendly. No metal coatings are used. Embossing can be integrated into a printing line so that high volume low-cost production is possible. There is no need for extra production phases like vacuum coating.

BUSINESS POTENTIAL
Hot embossing of dynamic graphics is targeted to consumer brands and their communication and authentication supply network covering packaging converters, printing houses and brand design agencies.

VTT provides technology transfer services. Dynamic graphics help consumer brands to differentiate their packaging from those of the competitors and from counterfeit products. Brand design agencies learn how to enhance static printed graphics with dynamic optical effects. Packaging converters and printing houses can get consultations on incorporating hot embossing technology to their printing lines to make all of this possible.

Hot embossing is a general production technology that can be used in many application areas apart from dynamic graphics. When converters and printing houses start from dynamic graphics, they have the production capability in place to continue to more advanced hot embossing applications like indicators.

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This work was funded internally by VTT.
Low-cost printed indicator devices can benefit the food, cosmetics and medical industries by improving quality control, product safety and traceability. In this project, printing techniques were utilised to produce disposable quality indicators reactive to oxygen. Leakage indicators for the food and medical industries were the primary applications. The indicators were activated by heating e.g. when the product is sterilised, or by applying a volatile reducing agent before printing.

INTRODUCTION
Many foods and medical products are packed in protective oxygen-free atmospheres. Oxygen-sensitive sensors added to the package interior can be used to show whether the package has been damaged. The most important requirement for product quality indicators is the correlation of the sensor indication with the product quality. Various promising substances presenting colour change in redox-reactions are available. In any case, the colour change or the reading must be irreversible and easy to interpret. Printing techniques place high demands on the quality of inks: ink-jet inks must be low in viscosity and must not dry out, and the ingredients must not flocculate as the fine nozzles will become blocked. Further, the inks must interact with the substrate to spread and adhere in the desired manner. The active substances must retain their reactivity, and the final printed surfaces must withstand the conditions for which they are destined. VTT has developed and patented special low-cost inkjet printable indicator systems and allowing on-demand, customised indicator (1).

MATERIALS AND METHODS
The project generated knowledge on the following: successful formulation of the ink, the issues related to the reactive substances contained in the ink, the serviceability of the ink in the printing process, and the compatibility of ink and printing substrates (plastics, fibre based materials). Printing techniques included flexography and inkjet printing.

The colour change reaction of the printed and heat-activated indicator systems was studied earlier by VTT. In this project, the system was further modified and formulated into printing inks. In the project, a water-based ink for fibre-based substrates and a solvent-based ink for plastic substrates were developed.

The reactive substance in the indicator is water soluble. In order to prepare a solvent based ink, a derivative of the molecule was prepared. The indicator is designed for use on the inner surface of any package. Therefore, food additives or other elements suitable for contact with food were used.

Normally non-porous materials (plastics) are printed using solvent based inks, while aqueous inks are suitable for highly porous materials (paper). The disadvantages of aqueous-based inks are related to their behaviour on non-absorbent material, their drying times, the solubility of active substances and the binder, and the wet fastness. In the case of solvent-based inks on nonporous substrate, no absorption or penetration occurs; as a result, the printed image relies on the quick evaporation of the

**Figure 1. Demonstration of indicator colour change in packages.**
ink solvent to be fixed on the substrate. In the development of solvent-based inks, new binder systems were identified to be printable using a variety of solvents. The wetting and adhesion of the solvent-based binder systems on plastics is remarkably better than that of the water-based since the surface tension of the solvents is much more compatible.

The most crucial part of printing technology is the ink and its physical properties:

- Ink: viscosity, surface tension, non-foaming, non-corrosive, stable (shelf-life), non-toxic, no bacterial growth
- Image: good adherence, quick drying, high colour density, light and moisture resistant, smear resistant
- Indicator performance: reliable colour change, sensitivity, stability during ageing, absence of interfering reactions, reliable operation in various working conditions, humidity and temperatures, irreversibility

RESULTS

The developed package leakage indicator systems have following features (2, 3):

- activated by heating (e.g. 121°C) or by a volatile reducing agent
- all ingredients are food additives or suitable for direct contact with food
- indicator inks are printable directly on the inner surface of the package, on stickers, and on oxygen absorber pouch
- three ink products have been developed; water-based for paper substrates, water-based for plastics, and solvent-based for plastics
- good adherence to plastics and paper
- printable in text or code form
- use with oxygen absorber
- easy to store and use, one product, applicable in a single step (no specific requirements on storage conditions before or after applying indicator in package)
- adjustable speed of reaction
- good sensitivity against visible light
- clear and irreversible colour change

SUMMARY

VTT has developed various low-cost indicator technologies for consumer packages. Printing inks containing certain reactive substances indicating oxygen, and suitable for printing on both fibre as well as plastic materials have been produced. In this project, the formulation and design of an easy-to-use heat or volatile reagent activated indicator was developed. The reactivity of this kind of indicator can be tailored to signal package leakage in seconds or in days/weeks, and can be used e.g. for sterilised medical products and perishable foods in modified atmosphere packages.

BUSINESS POTENTIAL

Potential future applications:

The indicators developed can be optimised for use in various food, drug and medical product packages, including:

1) product quality indicators for manufacturer, wholesaler, brand-owner and consumer (online seal quality control devices in production plants; quality indicators/anti-tampering devices in the supply chain), 2) use-by-indicators on opened packages, 3) indicators integrated in printed codes.

Benefits:

Low cost indicator systems provides extra merchandising and differentiation features for brand-owners and adds value for consumers — either giving visual signal or integrated in codes or pictures and read e.g. with mobile camera phone.

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The indicator concept is based on the hot embossing of indicator surfaces, the composition of indicator materials and the reactions of the substances used in various conditions. Hot embossed gratings can be added to materials that can react to certain stimuli of the surroundings. The grating pattern is destroyed when the dimensions of the structure are changed. The first application is as a humidity indicator.

INTRODUCTION

Indicators on conditions such as temperature, relative humidity and the atmospheric conditions in which products are kept can provide information related to product quality. Only a few significant commercial active and intelligent packaging systems are on the market but these are expected to become common on retail packages in the near future.

This project aimed to develop an indicator for food and drug packages. The work is based on strong knowhow in hot embossing technology and on the formulation of indicator materials. The primary application is the humidity indicator. The indicator concept can be used for establishing the necessary conditions for packed products, product authentication and tamper-proofing.

MATERIALS AND METHODS

The indicator is composed of an active substance combination, which can be attached (preferably, through printing) on a package or on a label. The indicator is made of materials that swell (or shrink) due to the expected conditions. The humidity indicator consists of hygroscopic substances. The packaging or other substrate material is a polymer film or fibrous material. Indicator substances were formulated as solutions. The first trials were carried out using hand coating techniques. Hot embossing was carried out using a Madag P2000 flat bed embosser. The hot embossed indicators need to be stored in dry conditions. Tests on the indicating reactions were carried out in controlled humidity chambers. The indication reaction becomes visible as the glittry grating pattern disappears.

RESULTS

It was possible to produce hot embossed gratings on certain materials that can react to certain stimuli in the surroundings. The starting substances were combined into working compositions. The various indicators were tested in order to measure the reaction rates as a function of the composition. Indicators reacting to humidity were found to work well. The reaction rate can be adjusted by formulation (grating disappears in a time period varying from 30 seconds to a few days). The composition was then optimised. Various dyes were included in the formulations in order to improve the visibility and appearance of the indicators. In addition, various coloured packaging materials used as substrates for the indicators resulted in distinct optical patterns and indication reactions.

SUMMARY

Hot embossed gratings can be produced on certain materials that can react to certain stimuli in the surroundings. Various formulations have been found to react to humidity. The reaction rate was modified by optimising the composition of the active material. Dyes and coloured sub-

Figure 1. Indicators produced on coloured substrates or containing various dyes.
strates were utilised for producing clear and readily visible patterns and indication reactions.

**BUSINESS POTENTIAL**
Humidity indicators can be used in a number of different consumer packed good applications, e.g. in food, cosmetics, pharmaceuticals and electronics. The humidity indicator can secure the conditions of products that require dry storage. Irreversible indicators could be utilised either to indicate the moisture entrapped in the package during packaging procedure or to indicate the storage of the opened package in the presence of high relative humidity.

Application examples include: low cost, attractive tamper evidence systems, anticounterfeiting labels; brand promotion features with changing images

**REFERENCES**
Patent application FI20085611

*Figures 2-3. Indicator as tamper evidence or brand enhancement applications.*
**INTRODUCTION**

Optical indicators are based on active compounds, which undergo a definite colour change depending on changes in exposure conditions. VTT has developed and patented special low-cost inkjet printable indicator systems. In addition, VTT has created indicator monitoring systems based on camera phone technology. This article describes the technologies and the operational environment in which the new camera phone based indicator applications are developed.

One application area of mobile phone readable optical indicators involves ensuring the freshness of food products. When the information included in a two-dimensional bar code is decoded, the colour of the symbol can also be detected. One application for colour detection is printed food quality indicators, which enable the freshness information and other useful information to be combined in one symbol.

Detection of colour change in the food quality indicator involves detecting the colour coordinates of a printed area whose colour changes based on the state of the packaged product. The colour detection device can reveal if the product is fresh based on information given by the two-dimensional bar code. If the values detected are out of the range values, the software tells the user not to use the product.

Applications for colour codes read by a camera phone include the reliable detection of quality indicators to ensure that spoiled groceries are not sold or consumed. With camera phones, the product freshness can be easily checked even when the products are already on shelves or at the cash register.

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**Figure 1. The principle of a quantitative indicator.**
RESULTS
The aim of the project was to build an illustrative and easy-to-use demonstrator, which clarifies the basic principles of quantitative indicators. To carry this out, a special, reversible heat indicator was developed based on thermochromic inks. Camera phone software was also created to interpret the indicator.

The principle of a quantitative indicator is described in Figure 1. The data included in a 2-D code is used to interpret the colour change in the indicator and based on these numeric values a mobile phone equipped with special software will give information, instructions or warnings to the user. There is also a colour calibration field in the indicator so that the effects of varying illumination in different places can be taken into account.

The final indicator mobile phone system can be seen in Figure 2. There is an indicator field on the business card which changes colour when heated. The temperature area of this indicator is 23-45°C. At room temperature, the colour of the indicator is purple, but it becomes more transparent when heated. The indicator is totally transparent at a temperature of 45°C.

The action of the demonstrator can be shown by pushing it against a hot coffee mug. When heated, the indicator becomes transparent. When the indicator field starts to cool down, frames can be taken with a camera phone and special mobile phone software translates the colour values into a temperature, which can be seen on the display of the mobile phone.

SUMMARY
In this article, some areas of VTT’s indicator and mobile phone research were covered. But the applications of coding and camera phone technologies are practically unlimited. In other words, this is only the beginning.

BUSINESS POTENTIAL
Research and experimental studies for coding and optical and electrical detection systems were carried out to outline the possibilities of camera phone readable inkjet printed indicators. The indicator can in principle monitor almost any changes in its surroundings like temperature, relative humidity, UV radiation etc. It can also be used for logistical as well as anti-counterfeit systems. So there are vast amount of application areas for the system. For this reason, all new business can be built based on the technology.

REFERENCES

Figure 2. The final system, which consists of an indicator and a mobile phone with a special interpretation software.
The primary objective of the EU FRESHLABEL (COLL-CT-2005-012371) project was to develop tailor-made time-temperature indicators (TTIs) for specific fish and meat products in the European fish and meat industries. The indicators were tailored according to the shelf life and optimum storage conditions of the products they are designed to monitor. The project was carried out as a collaboration between industrial associations, small and medium-size enterprises and research organisations (TTZ-Bremerhaven, VTT, the University of Bonn and the Technical University of Athens). TTZ-Bremerhaven was the coordinator of the project.

INTRODUCTION
The consumption of fresh and chilled meat and fish products is on the rise within the EC, and food safety and control are a major concern for all consumers. The state of frozen/fresh fish and meat products is often related to the temperature conditions during transport and storage. Elevated temperatures are the most common cause for spoilage along the supply chain. It is therefore of the utmost importance to ensure the continuity of the cold chain. In FRESHLABEL, the aim was to develop tailor-made time-temperature indicators (TTIs) for specific fish and meat products. At VTT the feasibility of OnVu™ time-temperature indicators for the quality control of marinated salmon trout slices and cold-smoked salmon was the main issue examined.

Depending on the season and the prices of the raw material, marinated salmon trout for commercial distribution can be produced using either fresh or frozen fish obtained from different sources. The quality and freshness of the raw material is likely to influence the shelf life of the end product and the potential variation in the raw material was considered a major challenge in the implementation of the time-temperature indicators for this particular product. The main aim of our final storage tests with marinated salmon trout cuts was to investigate the effect of different raw materials on the shelf life of marinated product and to validate the performance of the time-temperature indicator (TTI) on the products made from different raw materials.

On the basis of our previous test runs with cold smoked salmon slices, the product was found to remain very stable during its commercial shelf life. However, if the cold chain is not constant, quality and safety problems are likely to increase. In the pilot tests of FRESHLABEL, the focus was on confirming the capability of the time-temperature indicators to detect problems in the cold chain and to assess the possibility of establishing a link between colour change and deterioration in product quality.

MATERIALS AND METHODS
Samples of marinated salmon trout (Oncorhynchus mykiss) cuts and cold-smoked salmon (Salmo salar) slices (commercial packages) were produced and packaged by the manufacturers Kuopion Kalatuote and Myrskylän Savustamo. Several OnVu™ indicators activated using different UV-doses were attached to the packages.

Samples of marinated trout were stored at two different temperatures representing the optimal and abused storage temperatures in the cold-chain. In the case of the cold-smoked salmon, some of the sample packages were placed in a real cold chain (refrigerated lorry and cold storages in distribution chain) immediately after packaging, and the rest of the samples from the other tests were stored in controlled conditions in different temperature schemes (Figure 1).

The microbiological quality (psychrophilic aerobic bacteria, lactic acid bacteria, Enterobacteriaceae, Listeria monocytogenes) and sensory quality were evaluated by a trained panel to characterise the spoilage process of the two fish products studied. For time-temperature
CONSUMER PACKAGED GOODS

indicators, colour measurements were taken with an Eye-One Pro colorimeter (X-Rite) and a sensory evaluation was done to determine the darkness of time-temperature indicators. The shelf life of the fish products was estimated at different storage temperatures with regard to microbiological and sensory quality by determining the cut-off time, i.e. the time required until the product quality deteriorated to a level considered unacceptable.

To compare the suitability of the time-temperature indicators and the product deterioration rate, the correlations between different analysis results were evaluated. The correlation results were also used to compare the visual and instrumental TTI colour change evaluations.

RESULTS
Marinated salmon trout
It was confirmed that marinated salmon trout cuts are microbiologically sensitive products and that maintaining a low storage temperature is very important for maintaining the product quality. The type of raw material used (fresh vs. frozen) appeared to have a considerable effect on quality maintenance. The microbiological quality, especially the number of Enterobactericeae seemed to be the restricting factor in terms of quality maintenance.

Since the indicators are most likely evaluated through solely a visual inspection in real distribution chains, the colour of the indicators was also evaluated visually by the sensory panel as well. There was a close correlation found between the sensory evaluation of the indicator darkness and the instrumental measurement.

It could also be seen that the rate of quality deterioration of the marinated salmon trout cuts – particularly, the microbiological quality of the product – closely correlated to the colour change rate of the indicators. The rate of the indicator colour change seemed to remain steady regardless of the origin of the raw product. However, as the shelf life of different types of raw products varied considerably, it was determined that the indicator end-point should be optimised separately for each type of raw product. In this trial, there was a strong correlation between the shelf life of the fresh domestic product and the end-point of an indicator activated for 2 s (Figure 2).
Cold-smoked salmon

It was confirmed that cold smoked salmon is a very stable product and despite the fact that extreme temperatures were artificially introduced while the product was stored, neither sensory nor microbiological spoilage occurred during the commercial shelf-life. In the real distribution chain studied in this work, the temperature remained at an acceptable level throughout the storage.

However, as cold smoked salmon is a product prone to contamination by pathogenic *Listeria* spp., the maintenance of cold-chain is extremely important to maintain the safety of the product. In the instrumental measurement and visual evaluation of the colour change, the different temperature schemes clearly affected the indicator colour. Additionally, it could be confirmed that the visual colour interpretation closely correlated to the instrumental colour measurement when the end-point of the indicator was defined as earlier in the Freshlabel project ($L^* = 69$, $a^* = -7$, $b^* = -5$).

Moreover, there was a reasonable correlation between the indicator colour change rate and deterioration rate of the product quality. The indicators activated with feasible activation doses (3s and 5s) seemed to reach the end-point before quality began to deteriorate. However, due to the potential risk of pathogen growth in these circumstances, it is justified to choose an indicator that reacts to temperature abuse before quality deterioration commences – provided that the indicator does not reach the end point in optimal storage conditions.

SUMMARY

To summarise the results obtained in the final tests with OnVu™ time-temperature indicators, the indicators seemed suitable for evaluating the quality of marinated salmon trout cuts. Moreover, cold-smoked salmon was found to be a very stable product: despite the extreme temperatures artificially introduced during product storage, neither sensory nor microbiological spoilage took place during the commercial shelf life. However, as cold-smoked salmon is a product prone to contamination by pathogenic *Listeria* spp., the maintenance of cold-chain is important and the different temperature schemes had a clear effect on the indicator colour. TTIs are hence a suitable tool for safety enhancement of products being microbiologically stable but still prone to pathogenic contamination.
Moreover, a visual colour assessment of the indicator was found to correlate closely to the instrumental colour measurement.

BUSINESS POTENTIAL
Increasing consumer confidence in meat and fish products, especially regarding cold-chain aspects, is of high priority for all those involved in food manufacturing, trade, logistic and distribution.

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NAFISPACK - Natural Antimicrobials for Innovative and Safe Packaging

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NAFISPACK is a three-year European project under the EU 7th framework. VTT together with 16 partners from Sweden, Norway, Denmark, Spain, Italy and Germany are developing packaging technologies that will improve the shelf life of fresh food. VTT participates in the development of antimicrobial packaging material and printable freshness indicators.

The project has two main objectives:
* To develop innovative and safe packaging systems in order to increase fresh product shelf life by using two novel packaging technologies: antimicrobial active packaging and intelligent packaging for fresh fish, chicken, and minimally processed vegetables.
* To design a safety assessment methodology for antimicrobial active and intelligent packaging using chemical, toxicological, microbiological and sensory analyses methods.

This project aims at assuring the safety and quality of foodstuffs through the supply chain.

INTRODUCTION
The delivery of safe food from the producer to the consumer is a key priority for industry and authorities. It requires meticulous monitoring at every stage in the supply chain “from farm to fork”. Packaging plays a crucial role since its most important functions are the preservation and protection of food. The industry needs effective packaging systems to preserve food safely and maintain the food quality during distribution and storage while, while addressing increasing consumer demands for fresher, minimally processed, more convenient and safer foods. This leads to the need for developing innovative and safe modern packaging that have never been used and are produced using new processes. So there is the need to ensure the safety and benefits that such food packaging solutions can bring.

ACTIVE AND INTELLIGENT PACKAGING
The role of packaging is, in most cases, a rather passive and inert one but during last decades, the idea of active and intelligent packaging has got more attention and many commercial products have been introduced and used in the food area.

Active food contact materials are intended to extend the shelf life or to maintain or improve the condition of packaged food. They are designed to deliberately incorporate components that would release or absorb substances into or from the packaged food or the environment surrounding the food. On the other hand, the goal of intelligent food contact materials is to monitor the condition of packaged food or the environment surrounding the food.

Polymers are appropriate materials for the development of active structures thanks to their mass transport characteristics – permeation, sorption and migration. The active components can be incorporated into the package walls by diverse procedures which are included as solutes in polymer solutions or dispersions for coatings, as constituents which are melt-blended during plastic extrusion or through the functionalisation of the package surface. From there, the active agent can be released into the food or headspace to make their action beneficial; it can also remove food or headspace components which are sorbed into the polymer matrix or act upon contact with the food.

Intelligent packaging systems can provide rapid, low-cost, package integrated ways to determine the quality of food in consumer packages throughout the logistics chain from producer to consumer. For instance, volatile basic nitrogen compounds have been recognised as indicators of seafood spoilage. Non-amine volatiles, mainly short chain alcohols and oxidation products of fat are also potential compounds by which food freshness can be gauged.
The combination of natural antimicrobial (active) and intelligent functions in packaging appears to be a really innovative and safe solution to ensure the proper preservation of fresh products and to prolong their short shelf life and improve its quality. NAFISPACK will develop novel packaging solutions by addressing all packaging aspects within the supply chain (Figure 1).

KEY AREAS OF RESEARCH

In order to fulfil the objectives, the following detail-specific areas of research will be covered:

In the project evaluation of the suitability/feasibility of Natural Antimicrobials for food packaging applications will be carried out. Certain natural antimicrobial agents with the highest possibilities for incorporation in packaging materials and which are effective against the most common and dangerous target microorganisms which may be present in the target foods initially chosen (fresh fish, chicken and minimally processed vegetables) are identified.

In the project effective antimicrobial food materials for package design are developed. Polymeric materials which include natural antimicrobials using different processing strategies such as coating, extrusion and material functionalisation are developed. Their mode of action will be characterised and their efficiency for the target food will be assessed. A controlled release of active agents is tailored through the use of novel technologies such as encapsulation and nanotechnologies. Design and up-scaling in pilot plants will be carried out in order to obtain real food packaging systems of the materials developed.

An intelligent food packaging solution will be presented. Tailored indicator materials that react to the presence of the quality indicating metabolites, hence providing rapid, low cost, package integrated ways to determine the quality and safety of the target food in consumer packages through the logistics chain from producer to consumer are developed.

Risk assessments and new risk assessment models to develop new active and intelligent packaging solutions (microbiology, migration and toxicology) are introduced. The effectiveness of the developed packaging solutions to extend shelf life by maintaining the chemical, physical and sensorial quality of the foods is evaluated. The recyclability of the new packaging solution will be assured by new tools, and risk versus benefit for the materials to be used as recycled materials will be compared.

NAFISPACK will contribute to the EU Framework Regulation regarding the safety assessment and will help to fulfil the regulatory gap regarding active and intelligent packaging. The purpose is to supply proposals for advice to concerned parties, such as legislators, food inspectors and industry.

ACKNOWLEDGEMENTS

Financial support from the European 7th Framework Programme for the NAFISPACK project is gratefully acknowledged.
Antenna and sensor printing processes were developed. Several sensors can be manufactured for physical sensing or user interfaces. Sensors integrated to silicon-based readout, communication electronics and printed antennas can form an extensive sensor platform for various applications.

INTRODUCTION
Printed electronics offer a better cost-benefit ratio than printed circuit boards when the print area is very large. A large production area is obviously needed if quantities are extremely high. Typically, however, such production requires lengthy development and markets cannot generally be created in a short time. On the other hand, printed electronics can be used in applications in which the product requires a specific size. This is the case of user interfaces of limited size: they may make use of a human finger on keyboards or the human eye’s ability to see written text on displays. The second requirement for the components of a large production area is antennas, the size of which is constrained by the wavelength of electromagnetic radiation. Finally, the third requisite is large area sensing, which is often needed when the whole structure must be observed rather than sampled. All these applications could make use of printed electronics components.

However, applications that combine some or all of these components can provide even more benefits; one example is communicating sensor systems, potentially even with an integrated user interface. It may be possible to integrate the manufacture of these by using the same materials and processes, thus producing printed sensor system platforms in one or a few print runs. However, since silicon-based microelectronics is currently the only technology that performs well enough for communication and signal processing, silicon chip or electronic modules are needed to act as the “brains” of the system. Silicon can be directly attached to a printed sensor system with flip chip bonding, using a carrier substrate or by a connector with a separate electronics module.

MATERIALS AND METHODS
The main component studied in this work are:
- antennas and interconnections, thus simple conductive structures; and
- sensors, especially strain and pressure sensors.

ANTENNA PRINTING
Antennas often limit the scope of a communication system, but demand for conductivity is high, especially in frequencies below the UHF – band. With today’s print-
ed electronics, this typically leads to the application of a thick layer of metal-based ink. Therefore, the rotary screen printing of antennas was studied here in pilot scale trials. The test structures consisted of HF and UHF antennas for RFID tags, an antenna structure for inductive energy transfer and structures for measuring and analysing print quality. PEN and Polyimide substrates were used with two silver PTF – (Polymer Thick Film) inks and one silver nanoparticle ink. Additionally, the printing screen mesh size was varied in test runs to study its effects on e.g. layer thickness. A hot air drying oven length of 4 m was used. Printing trials were run using VTT’s ROKO pilot line.

SENSOR PRINTING
A pressure sensor can be obtained with a three-layer structure consisting of two electrodes and a layer of material with piezo effect in between. Silver prints or deposited aluminium can be used for electrodes. Materials that provide the piezo effect include, for example, several titanates. Additionally, resistive sensors (strain gauges) were printed with both gravure and screen printing.

WIRELESS SENSOR MEASUREMENT PLATFORM
For the purpose of the demonstration, a VTT wireless sensor platform module was used. A screen-printed resistive sensor and a wireless measurement platform are shown in Figure 2.

RESULTS
Antenna printing trials
The performance of rotary screen-printed antennas depends on several parameters such as ink type, viscosity, screen mesh size, printing speed, substrate etc. With the correct parameters, rotary screen-printed antennas perform adequately. The line thickness is typically 10 - 15 µm and sheet resistance 50 mΩ / square. The best print quality was obtained using a printing speed of 2 m / min. It was obtained that with further oven drying sheet resistance could be decreased to 30 mΩ / square. This means that optimal drying result could not be achieved in this pilot trial.

Piezo sensor
A piezo sensor is based on a three-layer structure consisting of a bottom electrode, a piezo layer and a top electrode. The bottom electrode can be printed using inkjet, screen or gravure printing, or patterned by etching or lift-off from a deposited metal layer. For a large area, piezo layer screen printing offers the highest reliability, although gravure printing has also been tested successfully. The piezo material must be poled with DC voltage to get a response. This technology can be used to produce a real printed pressure sensors. Several print compositions have been tried, all with a piezo response. Static electricity shielding may be required for reliable pressure sensing.

The screen-printed large area resistive sensor was used to demonstrate wireless entrance monitoring. The printed sensor was embedded in a rubber carpet and connected to wireless measurement node (shown in Figure 2). A person entering can be detected by a computer with wireless transceiver and signal processing application. The application can be used e.g. for safety controls in industrial environments.

Additionally, printed strain gauges were compared to commercially available strain gauges. With gravure printed silver conductors on PET foil, a significantly higher response compared to response of compensated commercial strain gauge was obtained. However, since the material selection was not optimal, the substrate’s elastic properties caused a slow response time, low repeatability and strong temperature dependency. However, for applications like entrance monitoring, only changes are required (as opposed to slow resistance changes or accurate strain value); as a result, printed strain gauges can be used for monitoring applications. For more accu-
rate measurements, material selection must improve and compensation needs to be further developed.

SUMMARY
The production methods for large-volume printed electronics have been studied for a large area sensor and antenna implementation. Antennas can be produced by rotary screen printing, with resulting resistance levels of approximately 50 mΩ/square. Additionally, the printing of strain gauges and pressure sensors have been studied. The use of a printed strain gauge wireless readout system has been demonstrated.

BUSINESS POTENTIAL
Antennas and sensors are typically components limited by area. As a result, the miniaturisation benefits are very limited, giving printed electronics high potential. Nonetheless, silicon-based electronics will continue to be used in the communication and signal processing of sensor data. Applications for sensor systems are used for large structure monitoring in buildings, machines, etc. Later on, printed sensors combined with silicon-based electronics will also be used for cost-effective applications in ubiquitous sensing or smart packaging.

ACKNOWLEDGEMENTS
This work has been performed in several VTT and jointly funded projects. We would like to thank all our supporters and collaborators.
In this work, VTT has further developed an add-on microscope module. The new universal module can be connected to different mobile phones and thus the advanced camera technology and image processing software of the latest phone models can be utilised. Surface measurement providing 3-D images were added to the system. Complex analysis can be made combining the capabilities of mobile phones and servers that can be accessed using the versatile communication methods of the phones. Application areas cover security and brand protection, consumer-brand communication, surface analysis and pharmaceutical studies.

INTRODUCTION
A microscope add-on module to a 1.3 megapixel camera phone was developed some years ago in the AKTIVA-project [1]. The core component in the module is a special plastic lens that could be produced using the cost effective injection moulding method. The lens serves two purposes: illumination and imaging.

There are LEDs on the electronic circuit board embedded in the lens structure. The lens collects and guides the LED light to the surface to be imaged. The imaging part of the lens is designed to be in front of the camera optics and to guide light to the CMOS image sensor with very tiny pixels (< 5um). The self-contained add-on optical module contains macro lenses, light sources, printed circuit board, battery, switches for lights and a casing. It can be connected with a bayonet mount to the Nokia 6630 camera phone.

MATERIALS AND METHODS
In this work the functionality of the microscope module was expanded. The fact that connection was limited exclusively to the Nokia 6630 was considered too restrictive, and as a result, a more general purpose connection concept was developed.

The casing was designed so that it can be attached to the mobile phones that have a sufficiently flat area around the camera optics. In temporary connections, the method can be double-sided tape that is targeted for easy removal and for remounting, stickertape or magnets. If a special instrument is made using the microscope module and a mobile phone, then a fixed connection (using glue or screws) can be used. For quick temporary usage, even two-hand operation is possible: one hand guides the module and the other hand, the mobile phone.

A system was developed to take 3-D images of the surface. The system contains the microscope module with electronics, a mobile phone with picture-taking software and a server or a PC with analysis software. The mobile phone software commands the microscope module using sound signals sent via speaker output to the module. The module takes the signals and light LEDs according to the

Figure 1. Mobile microscope to study print quality.
commands. The software can light the LED on one side, take a picture and then light the LED from another side and take another picture. These pictures are sent to the PC using a Bluetooth connection. The analysis software in the PC makes a 3-D image of the surface utilising these two pictures and sends it back to the mobile phone.

RESULTS
The universal connection of the microscope module provides the possibility for using the newest mobile phones with their advanced camera systems. For example, with 5 megapixel cameras, the resolution of the microscope system drops below 10 micrometers.

One application area of the mobile phone microscope is security codes. These codes are like normal 2-D bar codes but very small, for example, 3 mm by 3 mm. Difficult to copy high-accuracy printing methods can be used and the microscope does not set any limitations because of the high resolution. Even laser engraving can be used to form very small pixels for the codes. With a laser, the security code can be engraved onto the package and even onto the product itself. The same software that is used to decode normal size 2-D codes can be used to decode the micro codes when the code is enlarged by the microscope.

Another application of micro codes is for consumer-brand communication. A lot of information can be printed on product packaging without disturbing the graphical layout of the package. Micro-code readers can be installed in shopping centres so consumers can take a package to the reader and access recipes, for example; in other cases, bottles marked with micro codes can be used with game consoles. Because the microscope module can be produced using cost-effective high volume production methods, it can be distributed to the consumers as part of a marketing campaign.

The possibility to take 3-D surface pictures opens a wide range of applications from security to surface analysis. In document or packaging security applications, pictures can be taken of paper surfaces. Because of the stochastic nature of the paper production process, the surface patterns are unique. Therefore, inspectors can take pictures of packages in the field and compare the results with the database in order to find out if the packages are original. In many business fields, experts need to analyse surface smoothness or particle size, like the quality of painted surfaces or printed patterns. A pocket size microscope with communication capabilities is ideal for these users.

SUMMARY
VTT has developed an add-on microscope module that can be attached to mobile phones or other camera systems. The module can be customised to a specific application that takes into account requirements like the image area and resolution. Lighting can be normal visible light; in others cases, UV- or IR-lights can be used. The

Figure 2. Example of a 3-D surface image.
microscope module can be produced in different volumes. Even high volume low-cost production is possible thanks to injection moulding lens production.

VTT has further developed the functionality of the mobile phone microscope to include 3-D surface imaging. This provides the functionality of a surface analysis device at a fraction of a cost because a mobile phone is used as the core component of the system. By connecting this pocket size analyzer via wireless Internet to servers containing surface data or complex analysis software, very powerful systems can be offered to experts doing research in the field.

BUSINESS POTENTIAL

The mobile microscope has a wide range of potential applications. Document security, high security and brand protection applications are a few important areas. The microscope module can be an enabler in consumer-brand communication applications. Surface analysis capabilities provide great benefits for field experts in many business segments.

A microscope is a traditional measurement device for pharmaceutical work. The mobile phone microscope could provide a pocket size microscope with excellent communication capabilities for to analyse biological samples in field work. Due to versatile lighting possibilities, the mobile phone microscope has the potential to become a reading device for new disposable biosensors.

REFERENCES


ACKNOWLEDGEMENTS

This work was funded by VTT.
In these studies, the aim has been on finding new business applications that utilise simple intelligence, such as 2-D codes that can be added to substrates with normal printing technologies. The first applications were weather sensors, UV-fluorescent security elements and UV-indicators. In a further study, the complex value chain of packaging was examined to determine the business potential for applications and new roles for the players.

INTRODUCTION
Printed functionality refers to adding new functionalities to flexible substrates like paper or plastic through print technology. In these studies, we have concentrated on finding new innovative applications based on existing technologies that are suitable for traditional printing plants. In the first part of the study, applications for sheet-fed printers were developed. In the second project, we created new applications in package printing. The aim is to determine the communication requirements among the different players of the value chain, and to develop new business potentials and roles for the players.

MATERIALS AND METHODS
The first part of the study concentrated on the production chains needed for printed functionality. The technical requirements for the production were compared to the performance of traditional printers. New applications were then developed and their economic potential was estimated.

In the second part of the study, new applications of hybrid media were developed for the packaging industry. A survey of optically readable technologies for mobile camera phones was done. A great number of players representing the entire value chain of packaging were interviewed about their applications of hybrid media, their strategic intentions, their need for online information and their future roles in the value chain.

New applications of service forms for packaging are innovated and developed in some pilot projects. New business models are developed for the applications and the consumers’ willingness to pay for new services is assessed. The usability of the applications is tested and the solutions are further developed into commercial products.

RESULTS
Optically readable codes are the simplest form of intelligent elements for printed functionality and can usually be printed in conventional presses using ordinary materials. New service forms can be built on the codes, but the critical factors are the readability of the codes, the need for a reading technology and the penetration and standardisation of this technology. Other intelligent printed elements are sensors and indicators.

In the first part of this study more than sixty innovation embryos of new services were created. Weather sensors for newspapers, UV-fluorescent security elements, UV-indicators and conductive codes were piloted on a labora-
tory scale. In addition printed UV-indicators and UV-fluorescent security elements were also successfully made in full scale sheet-fed printing.

In the second part of the study applications of hybrid media in packaging is studied. Hybrid media enables new on-line information flows both upstream and downstream in the value chain. The role of each player now and in the future has been mapped. In particular, the brand owner needs to receive current data about consumer behaviour via the new applications. The retailer seems to be in a key position with regards to the application of the new technologies.

In the further study, a number of new service applications will be piloted involving several partners from the value chain. The consumers’ willingness to pay for new services will also be estimated together with the usability of the applications.

BUSINESS POTENTIAL

The 2-D bar code system developed at VTT has been successfully exploited by UPCode Ltd and Mobicode Oy and is now commercialised in 48 countries.

Several of the pilots tested in the first part of the study, including the UV-fluorescent security elements and the UV-indicators, are commercially exploited by participating companies.

The pilots created in the second part of the study will engage brand owners, materials manufacturers and system vendors and are expected to result in commercial exploitation of the new services for the packaging industry.

SUMMARY

The value chains of printed functionality offer new roles for the players, among them the printer. In packaging, they give the brand owner new possibilities to communicate with the consumer. The retailer has a central role in the exploitation.

REFERENCES


ACKNOWLEDGEMENTS

The study was partly financed by the Finnish Funding Agency for Technology and Innovation (TEKES) and by the Graphic Industry Research Foundation of Finland (GTTS).

In the first part of the study, the following companies participated: Hansaprint Oy, Ikistoori Oy, Joutsenpaino Oy, Lomakevaihtoehto Oy, M-real Oyj, Sanomapaino Oy.

The second part of the study was performed by VTT, the Finnish Pulp and Paper Institute KCL and the Helsinki School of Economics HSE in co-operation. The following companies are participating: Association of Packaging Technology and Research – PTR, Oy Gustav Paulig Ab, Raisio Oyj, StoraEnso Oyj, UPCode Ltd.
When using printing techniques to manufacture OLEDs, silver ink has been the only conductive ink commercially available. However, the work function of silver is high, resulting in reduced device performance. Hence, there is a need for developing suitable low work function metal inks. Low work function metals tend to oxidise easily, which makes the use of these inks challenging.

INTRODUCTION

Low work function metals oxidise easily and the oxidised metal surface prevents ohmic contact between metal particles in the ink, resulting in poor conductivity. Oxidation has to be prevented during the manufacture of the ink and the printing of the device. In addition, these metals are reactive with many materials, thus limiting the choice of solvents, binder materials and additives.

Good contact between the active layer and the cathode is crucial. Therefore, the solvent chosen must not dissolve the underlying layer. In addition, the curing temperature has to be lower than the glass transition temperature of the organic materials and the substrate.

RESULTS

Metal powders were ball milled in hexane prior to the ink preparation, and hexane was removed after milling. Ball milling of Al and Mg yields a smaller particle size and reveals a pure conductive metal surface. A mixture of Al – Mg 10% was also prepared by ball milling as part of the study.

THF and toluene were used as solvents and polystyrene and PMMA were employed as binders.

Flat screen printing tests were done in a glove box in an N2 atmosphere. The screen-printed Al layer was fairly smooth, although some pinholes still appeared. The thickness of the aluminium layer was 6 -15 µm. (Figure 2).
Pilot printing tests were done with an ROKO pilot printing machine using a rotary screen printing unit. Because the device lifetime without encapsulation is less than 1 min, protection must be applied before adhesive printing and lamination. This was done by printing a polystyrene layer on top of the cathode layer with rotary screen printing unit 2. Therefore, two inert gas printing units were built to enable both the cathode and the protective layers to be printed at the same time (Figure 3).

The volume of inert gas units reached approximately 2 m³ and a high volume of N₂ flowed into the units from the liquid nitrogen tank equipped with a condenser (Figure 4). This system produced an O₂ level of 0.1-0.2%.

When testing the same Al ink formulation as that used with the flat screen tests, problems arose: pinholes appeared, drying occurred too fast and the screen was blocked during printing. Different amounts of tetralin and mesitylene were tested to prevent drying from occurring too fast. Al powder was sieved prior to the ink preparation, which yielded homogeneous ink without large particles or lumps. The best printability was achieved with a formulation of 43.2wt% of Al, 7.1wt% of PS, 5.7wt% mesitylene and 44wt% of toluene.

Printing was done with a Gallus EP screen (110 mesh, ink laydown 26 µm, mesh width 150 µm) at a speed of 2 m/min and drying at 140°C.

The printing of the cathode ink was fair and the layer thickness was 10 µm (Figure 1). However, the pilot printed Al layer did not show electrical conductivity. The oxygen and moisture levels were not low enough to prevent the oxidation of the aluminium.

**SUMMARY**

This research revealed the possibility of manufacturing the low work function conductive ink and printing the cathode layer for all-printed flexible OLED devices. The aluminium ink was successfully printed at the pilot scale using a rotary screen printing technique (Figure 1). The functionality of the ink was lost due to an insufficient inert atmosphere. A new inert gas pilot line that enables the printing of the cathode layer will be installed in early 2010.

**BUSINESS POTENTIAL**

Low work function and highly conductive metal inks can play a key role in producing printed active optoelectrical devices like organic light emitting diodes (OLED) and organic solar cells (OSC).

**ACKNOWLEDGEMENTS**

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In the MAGIA project, production techniques for tailored magnetic nanoparticles have been developed. Particles are utilized to produce magnetic inks and polymer substrate materials. The properties of the produced composites and structures are studied both experimentally and theoretically in order to apply them especially in small antenna applications.

INTRODUCTION

The MAGIA project focuses on synthesising nanoparticles using scalable techniques with controlled characteristics (size, composition, crystal structure and coating) and then utilising the nanoparticles for material integration and ink formulation.

Furthermore, the magnetic properties of fabricated components and composites are evaluated based on existing theories on magnetic nanocomposites. In applications, the objective is to improve the performance of small antenna.

MATERIALS AND METHODS

Cobalt nanoparticles were produced using hydrogen reduction of CoCl₂ vapour at a temperature of 950ºC [1]. FTIR was used to monitor the concentration of the HCl reaction product.

Metal-polymer composites of Co nanoparticles in a thermoplastic polymer ER182 matrix were compounded using a mixing extruder. The homogeneity of the Co distribution was examined by FESEM. The magnetic properties of the composite were analyzed using Agilent E4991A RF Impedance/Material analyzer at 1 GHz.

Research teams participating in this project have extensive experience in scalable nanoparticle production, ink formulation and printing and are highly competent in printed electronics and the modification of substrates with nanoparticles. In MAGIA, these skills are combined to develop novel inks and other materials such as paper, plastics, composites and electronics substrates with tailored magnetic properties.

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Figure 1. A TEM image of chainlike cobalt agglomerates.

Figure 2. A TEM image of Co nanoparticle coated with four layers of graphene.
A simple model (denoted as the reluctance theory) was developed for the effective relative permeability $\mu_{\text{eff}}$ of the composite substrate. The model is based on equivalence of magnetic (reluctance, $R$) and electric (resistance) circuits, assuming periodically distributed cubic non-interacting nanoparticles [2].

**RESULTS**

A total of 50 g of cobalt nanoparticles was produced. Mass concentration of particles in the flow reactor was 17 g/m$^3$. The size distribution of primary particles was narrow, with a mean particle diameter of 76 nm and a standard deviation of 20 nm.

In order to prevent the oxidation and sintering of particles during handling, transport and storage of the powder, a technique for in-situ coating of the metal particles was developed. Graphene was also seen to reduce the particle size as coating took place during the formation and growth of the particles.

The particles were embedded with different filler loadings in thermoplastic substrate to increase the permeability of metal-polymer composite. The microstructure analysis showed that although the majority of the particles were separated, the particle chains and agglomerates could also be observed.

The preliminary results indicate that there is a significant improvement in permittivity ($\varepsilon_r$ from 2.41 up to 22.2) and permeability ($\mu_r$ from 1.06 up to 3.07) obtained with relatively small amount of cobalt nanoparticles.

The reluctance theory was compared with the well-known Bruggeman effective medium theory [3,4]. At the low loading levels, the predictions of the $\mu_{\text{eff}}$ were close to one another. However in the case of larger loadings, the models differ considerably. Models will be generalized to take into account losses as well as the demagnetization effects, which are influenced by non-uniform particle distributions. The preliminary simulation results on the radiation efficiency and the quality factor of small loop antenna as a function of lossless magneto-electric loading have been very promising.

**SUMMARY**

Cobalt nanoparticles look promising for use in magnetic dielectric composites in future applications. Reluctance modelling results also show potential for antenna applications such as WLAN. The goal is to print with conductive ink on substrate with enhanced magnetic properties.

**BUSINESS POTENTIAL**

High throughput printing techniques enable direct patterning and reduced material and processing costs compared to silicon technology. RFID and security codes are expected to be among the first commercially viable applications of printed electronics. In these applications, magnetic inks and thermoplastic magnetic substrates can be utilized for the critical enhancement of device characteristics in performance, cost and physical size to meet the application requirements.

**REFERENCES**


**ACKNOWLEDGEMENTS**

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New camera systems for the monitoring of printing quality and register accuracy were designed and installed in the ROKO pilot printing machine to create a pilot equipment for different R&D projects with challenging R2R demands. A load cell system was also installed to monitor the web tension between printing units.

INTRODUCTION

A ROKO pilot machine was installed at 2007. It is equipped with four replaceable printing units and gravure, flexography and rotary screen printing techniques are employed. When printed electronics and optics are roll-to-roll manufactured in different R&D projects, it is essential to perform online measurements to see how different printing parameters affect print quality, registration accuracy and web line control.

When installed, the ROKO pilot machine was provided with pre-registration equipment. During 2008-2009, the pilot facility was first upgraded to include load cells and punching control measurement (web control) and the second important upgrade was a registration/printing quality measurement system.

WEB LINE CONTROL

A web line control system was created to measure substrate tension from both sides of the web during printing trials. The web tension effects on the web width and the changes in the web width effects to the printing register accuracy. So web tension should clearly be under control when the aim is to attain accurate multilayer printed electronic components.

A ROKO pilot machine (Figure 1) has a total of eight load cells for web tension monitoring. One load cell is required before and one after punching tool (Figure 2) to ensure that punching is accurate for both edges of the substrate. The accuracy of the punching tool i.e. the hole position from the web edge and their distance from one another is monitored on the touch panel (Figure 3). The accurate punching is essential for the usage of tractor rolls, which guide the web material in the right position through the pilot machine. Furthermore, there is
one load cell before every printing unit (a total of four), one before rewind S-WRAP (mechanical web tension adjustment) and one load cell before web rewinder. Every load cell measures the strain on both edges of the substrate material; the machine side (MS) and the user side (US). The data from these cells are collected in real time and displayed on the touch panel monitors (Figure 4). In Figure 5, the uneven tension of the substrate material is shown. The white and red signals are separated before printing unit 1 (PU1) and the tension of the substrate material is corrected before printing unit 2 (PU2). The two signals are displayed on top of one another in PU2.

PRINTING QUALITY AND REGISTRATION ACCURACY MEASUREMENTS

To measure the print quality and the registration accuracy of printed ink materials, a camera system was developed and installed in the ROKO pilot machine. There are four cameras (two double camera systems), which provide online measurements of either both edges of the substrate (register marks) or the print quality from the any desired position of the web (Figure 6). These camera systems are portable and can be transfer to the other printing units if needed. In Figure 7, the register marks are shown. The line width of the cross mark is 500 µm and the size of the squares around the cross are
2x2 mm². The camera system is equipped with adjustable white, green and red led lights and this feature enables the lighting of different printing materials like transparent or reflecting electronic inks printed on plastics.

SUMMARY
The web control and printing quality systems were designed and manufactured for the ROKO pilot printing machine. These systems clearly save time, because printing process monitoring can be done online. Furthermore, the monitoring of the register accuracy of multilayer structures can be done and the web tension monitoring system prevents register problems due to unwanted substrate lengths between printing units.

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Coating Line for Semi-pilot Testing of Functional Coatings

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VTT offers roll-to-roll semi-pilot services especially for coating of fibre products with functional coating materials. The coating line consists of various modules which can be connected in the desired order. The modules include film coating, spray coating, corona treatment and a varying number of drying units.

INTRODUCTION
A new coating line for functional fibre products was installed at VTT Espoo in 2008. The coating line represents a new revolutionary coating concept for pilot testing, where the idea of a fixed process is completely discarded. The coating line consists of multiple modules, which can be connected to a complete line in the desired order.

With spray coating, very low amounts of coating can be applied on the web; by selecting an anilox roll with large cups, in contrast, a fairly high coat weight can be achieved. These extreme cases have completely different requirements in terms of drying, and the drying units and capacity can be selected accordingly.

With an adjustable coating width, a fairly small amount of coating suspension is required to manufacture roll-to-roll coated samples. This is especially important when new materials are being developed for coating applications.

The drying fumes are filtered, which enables various solvents to be used in coating suspensions.

MATERIALS AND METHODS
The coating line consists of multiple modules, which can be connected to a complete line as desired.

The units available are:
- Roll coating with anilox roll
- Spray coating
- Un/rewind, and winder
- Air drying units 5 pieces, IR-dryer
- UV-unit
- Edge guidance
- Spreading roller
- Corona treatment
- Offline calendar

The common features of the coating line are:
- Speed range 3... 90 m/min
- Electric driven, controlled via PLC
- Substrate grammage 40... 310 g/m²
- Coating weight 0.1... 40 g/m²
- Viscosities min. 1... 15 000 mPas
- Online moisture and coating amount monitoring
- Online web surface temperature monitoring
- Coatings can be water, alcohol, acetone and toluene based, pH 4... 10
- Web tension range 45... 450 N/m
- Coating width 500 mm (adjustable between 200... 500 mm)
- Max. width of roll 600 mm
- Max diameter of roll 500 mm

Figure 1. The coating line’s modules can be connected to a complete line in the desired order.
Roll-to-roll printing itself is not always sufficient for the production of printed electronics components and systems. A suitable counterpart for the additive printing process would be a high-precision, digitally controlled material removing process. Laser ablation has been used for this.

Additionally drying and sintering are the bottleneck processes in metal particle based conductor printing. Lasers show high potential for the curing process, especially in the case of nanoparticulate inks. This is due to the fact that the typical sintering temperatures for nanoparticles (100–300°C) are only a fraction of the macroscopic melting point of the corresponding materials. This allows paper or plastic substrates to be used.

INTRODUCTION

This work was performed at VTT in Lappeenranta using the test facilities built in 2007. Equipment includes a Lumera Rapid laser, an optical table and other necessary equipment with winding and unwinding machines for roll-to-roll testing. The facilities were developed to stabilise film vibrations and to improve the registration of print and laser processing. Ablation tests were done using different materials and wavelengths. During the project, the sintering of printed nanoparticle structures using laser treatment was also investigated at VTT.

MATERIALS AND METHODS

A major part of the work was used to improve the accuracy and web control of the R2R system. Improved accuracy was gained by replacing the triggering unit with a new version, positioned closer to the actual processing point.

In the first trials, the triggering accuracy was far too low for roll-to-roll production (up to 1.5 mm). To improve triggering accuracy, a laser beam was aimed at the triggering mark on the moving film using a focusing lens. The registration accuracy could be improved to 50 µm. This also required a change to the triggering setup in the scanner programme. After these improvements, the registration accuracy was limited by the edge roughness of printed registration marks, which were measured to be in the range of 50 µm. No correction system was used to compensate for the sideward mistakes of the roll-to-roll system.

Another web-related issue was the vibration on the processing area. According to preliminary tests, some vibration of the moving plastic film occurred in the working zone when the film was moving in the air between rolls. A supporting plastic plate was added to the working area, which substantially decreased vibrations.

ABLATION RESULTS

Ablation tests were made with two wavelengths (355 and 532 nm) with picosecond LUMERA RAPID 2 W laser and with four different materials (Al-PET, Cu-PET, ITO-PET and PEDOT-PSS). Different power levels were used on a pulse repetition rate of 100 kHz and with different scanning speeds of up to 1000 mm/s.

Figure 2 shows the laser interferometer measurement of an ITO-PET track done with 1000 mm/s and 100 kHz. The roughness is in the range of tens of nanometers.

Figure 1. VTT’s roll-to-roll test facilities in Lappeenranta.
When parameter settings are incorrect, spikes on the edges can be up to a couple microns in height. This is problematic in the production of thin film components, like OLEDs or TFTs (Thin Film Transistors), since roughness causes short circuiting between layers. This problem has to be solved in the future.

**SINTERING RESULTS**

The sintering of printed nanoparticle structures using laser treatment has been investigated at VTT. Laser sintering can be utilised in the manufacturing of printed conductor structures such as antennas, circuits and sensors [1, 2]. A drop-on demand printer was used to print patterns with metal-organic silver nanoparticles on a flexible polyimide substrate. Laser sintering was done with a 940 nm CW fibre-coupled diode laser. The process was optimised using different scanning speeds, laser power levels, line separation and repetition rounds.

In sintering tests, three different line thicknesses were printed to gauge the effect of line width. Sintering tests were done with Ink 1 using the hatch technique. The laser speed was 1000 mm/s and the line-to-line distance was 0.2 mm. The beam size was 1 mm in diameter. After a series of pre-tests, the optimal range of average power was estimated to be between 20–50 W. Table 1 shows the effect of average power on sheet resistance.

<table>
<thead>
<tr>
<th>Line Thickness (µm)</th>
<th>50 W</th>
<th>40 W</th>
<th>30 W</th>
<th>20 W</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 µm</td>
<td>0.16</td>
<td>0.18</td>
<td>0.29</td>
<td>0.52</td>
</tr>
<tr>
<td>200 µm</td>
<td>0.17</td>
<td>0.27</td>
<td>0.31</td>
<td>0.59</td>
</tr>
<tr>
<td>350 µm</td>
<td>0.16</td>
<td>0.19</td>
<td>0.28</td>
<td>0.38</td>
</tr>
</tbody>
</table>

**SUMMARY**

During the project, web control and ablation accuracy was significantly improved. A lot remains to be done in the coming years to be able to meet the demanding application needs. Laser ablation seems to be fastest and most flexible method for patterning layers on a moving web. Nonetheless, the edges of evaporated lines constitute a major challenge for researchers.

Laser sintering of nanoparticle inks seems to be promising curing technique for R2R sintering. Especially in cases where only part of the substrate need to be cured, laser has the potential for reaching a high processing speed. Additionally, it can allow low-cost low melting point substrates to be used since heating is well-targeted to inks. Choosing the right ink for the process and keeping the substrate clean are key factors for successful operation. Sheet resistance values only slightly exceed the values obtained by heat sintering (by about 20-30%). This result was obtained in preliminary test and can be further optimised.

**BUSINESS POTENTIAL**

Laser ablation might be a choice in the future for the online patterning of printed layers or evaporated layers. It also offers a unique chance for unit level personalisation in manufacturing. Additionally, lasers can be utilised as a tuning method to improve the accuracy of printed components. Laser sintering has a high potential for curing metal particle inks on flexible substrates. It offers a fast processing speed and low temperature processing, and therefore, it often represents an improvement over oven sintering.

**REFERENCES**


**ACKNOWLEDGEMENTS**

Laser sintering work was done in a TEKES funded PESEP-project. The ink jet printing for laser sintering trials was done in this project by Eerik Halonen from the Tampere University of Technology.
Organic light-emitting diodes have been one of the key optoelectronic components processed at VTT using roll-to-roll techniques. The aim was to develop a printing method with suitable polymer inks to realise organic light sources for signage applications.

INTRODUCTION TO THE OLLA PROJECT
An organic light-emitting diode (OLED) is a next-generation technology, which is already commercialised in display technology, e.g. cell phones, MP3 players and car audio systems. So far, the emphasis of OLED research has been on display applications, but a new application field of solid state lighting and signage is emerging. OLEDs such as lighting and signage elements can provide major advantages such as low power consumption and freedom in shape and colour design in comparison to traditional solid light sources. However, lighting technology has its own requirements in terms of high brightness, product life and emission uniformity in large areas. The printing of OLEDs on glass substrates was researched in the EC-funded integrated project called OLLA (“High Brightness OLEDs for ICT and Next Generation Lighting Applications”), which gathers experience and expertise in the field of OLEDs. Through the OLLA project, breakthroughs were made in various fields of technology (materials, deposition technology, device technology, and application requirements). [1]

INTRODUCTION TO THE ROLLED PROJECT
Traditionally, OLEDs are manufactured using expensive batch processes such as the vacuum evaporation technique. However, a new and interesting technique involves applying roll-to-roll printing methods for the manufacturing of flexible devices. The processes with high through-put capability will considerably reduce the fabrication costs. The printing techniques for the roll-to-roll production of OLEDs were researched in the 6th framework EU funded project, ROLLED - “Roll-to-roll manufacturing technology for flexible OLED devices and arbitrary size and shape displays”. [2]

The objective of the ROLLED project was to fabricate an entire OLED structure using roll-to-roll manufacturing methods. In order to attain roll-to-roll compatibility, all the materials, inks and device structures need to be suitable for printing.

MATERIALS AND METHODS
For the real breakthrough (using OLEDs in signage applications), the key element is cost-efficiency. Spin coating has been used as a deposition method for polymer solutions due to its advantage for processing uniform layers. However, a lot of material is wasted in the spin coating process and furthermore, the substrate size is limited. Instead, advantageous costs could be obtained by implementing high-throughput roll-to-roll printing techniques, e.g. gravure, for processing OLEDs.

The goal was to demonstrate OLED signage elements using printing techniques on rigid and plastic substrates. An OLED EXIT sign and an OLED anti-tampering product package were selected as the final demonstrators in the OLLA and ROLLED projects, respectively. The printing process and materials were optimised to achieve uniform and pinhole-free polymeric films. Both demonstrators contain a gravure-printed hole injection layer of PEDOT:PSS on an ITO-coated substrate. In the case of the EXIT sign, a white light-emitting layer was gravure printed on the top of PEDOT:PSS before the evaporation of the metal cathode. [3] In the case of the product package, green and red light-emitting polymers were roll-to-roll gravure printed on top of PEDOT:PSS. This OLED architecture also comprised an insulator and silver wiring layers, which were rotary screen printed after the printing and drying of polymers. Additionally, a
low work function cathode was printed before device encapsulation in order to realise fully printed OLED elements. [4,5]

RESULTS
The photograph of the OLED EXIT sign demonstrator is shown in Figure 1. The printed circuit board with the electronics was put behind the OLED. The whole assembly fit into a white housing. The brightness will be in the range of 200 to 300 cd/m² at a driving current of nearly 200 mA and a forward voltage of roughly 5 to 6 V.

The photographs of OLED indicators, which are wireless powered using an inductive power coupling of RFID reader, are shown in Figure 2. When the package is opened, an electrical seal in the form of a fuse is broken. Then the integrated electronics light up a red OLED indicator when the circuit is powered by an external RF field as illustrated in Figure 2(b). Otherwise when the seal is intact, a green OLED indicator is powered in case of RF activation as shown in Figure 2(a).

SUMMARY
We have proved the feasibility of gravure printing for the fabrication of large area OLEDs by demonstrating signage elements on glass and plastic foils. The use of roll-to-roll printing methods for processing of OLEDs is beneficial in signage applications in which the cost advantage of the processing method is crucial.

BUSINESS POTENTIAL
Organic and printed electronics represent a technology field that is developing extremely fast. New innovations in materials and processing technologies enable completely new application areas which strengthen the position of organic and printed electronics as one of the future's key technology areas. Printed and organic electronics are expected to become worth between 250-300 billion USD within the next 20 years. [6] For this market, OLEDs can be seen as an enabling technology that can create new markets and also strengthen the industry in areas like printing, packaging and the machine building industry.

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[1] www.olla-project.org

ACKNOWLEDGEMENTS
We sincerely thank the European Commission (FP6-2003-IST-2-004315 ROLLED project and FP6-2003-IST-2-004607 OLLA project) for funding the work presented on this paper. We would also like to thank ROLLED and OLLA partners for their guidance in the demonstrator issues.
The goal of the research is to integrate flexible OLEDs into polymer products using the efficient 3-D shaping technology of injection moulding. This synthesis of low-cost manufacturing technologies offers unique possibilities to add novel functionality to products in different application fields, such as product packaging, consumer electronics, health care and the automotive sectors - enabling the unobtrusive integration of electronics into everyday life. The benefits are novel functionality, decreased volume and weight, lower costs, fewer components and reduced electronic component packaging and assembly steps.

INTRODUCTION

In-mould labelling (IML) is the use of plastic labels during the manufacturing of plastic products through injection moulding. In the process, the pre-decorated label becomes an integral part of the final 3-D plastic structure. A great variety of plastic products are currently decorated using the IML process, including food packages, coffee cups and internal automotive components, such as dashboards. In-mould labelling is also a popular method for decorating injection moulded parts for consumer electronics: notebook computer and cell phone manufacturers are adopting IML technology to provide greater wear resistance than that attained through spray painting or pad-printing. Figure 1 presents the IML processing of foil comprising: 1) the foil is inserted into the mould, 2) the mould is closed and molten resin is then injected into the mould, and finally 3) the robot removes the final plastic product with the foil as an integral part of the 3-D structure.

But so far, products made with the IML process have only utilised passive labels with different graphics and textures. However, this widely used technology could be implemented with some modifications to directly embed active, flexible OLED foil into 3-D plastic product structures. The convergence of printing electronics and injection moulding process offers a unique opportunity to embed novel signage and brand promotion/protection elements into the plastic packages of high-value products such as clocks, cosmetics and jewellery – in these market segments, the plastic packages are already often being made with IML technology. Moreover, when the reliability of the OLEDs is further improved, the OLED foil could be embedded into plastic parts in automotive and consumer electronics industry to create signage, display and lighting elements integrated into the mechanical structure of car dashboards, cell phone covers, user interfaces in health care, smart home products etc. This type of integration offers a great number of benefits when compared to conventional electronics packaging and integration solutions: they boast a compact and conformal design, autonomous systems, weight and electrical connections as well as lower material costs and fewer assembly steps that result in major cost savings.

MATERIALS AND METHODS

The main issues that need to be addressed to successfully integrate flexible OLEDs into polymer matrix by IML process are:

- Ensuring the compatibility of overmoulding material and OLED foil material
- Developing 3-D forming methods for OLED foils
- Adapting the IML equipment for OLED foils
• Finding an optimal injection moulding process parameters.

Potential OLED foil and overmoulding materials are transparent thermoplastics such as PC, PMMA, COC, PET, etc. OLED foil and overmoulding material compatibility has to be ensured; the most critical issues are adhesion and warpage between overmoulding and foil material as well as barrier properties and cost. Adhesion between overmoulding and foil material can be promoted by special means, like plasma and laser treatments. Typical forming processes in the IML industry include vacuum drawing, high-pressure forming and thermo-bending. These processes can be used as a starting point for the development of a 3-D forming method that preserves the functionality and reliability of the OLED foil. Only minor modifications should be necessary to utilise conventional IML technology when using OLED foils. This development of IML process equipment includes the adaptation of foil feeding automation for OLED foils, like foil deformation and position control. Optimisation of injection moulding process parameters is crucial in order to ensure that 3-D OLED foils and their interconnections withstand the high temperature and pressure conditions inherent to the injection moulding process. Some injection moulding process parameters that need to be optimised include the temperature of the injected polymer, the temperature of the mould, the pressure of the injected polymer, the dose of the injected polymer and the cooling and cycle times.

RESULTS
Tests have been carried out in order to study the feasibility of the concept of embedding flexible OLEDs into the polymer matrix in the IML process. Before these tests, the issue of adhesion of the OLED foil substrate and the overmoulding material were examined in [1]. The OLED foil material with the greatest potential was found to be polyethylene terephthalate (PET) and overmoulding material polycarbonate (PC). OLED samples in the overmoulding tests had the following structure:

- ITO-PET
- Organic layers
- Ca/Ag cathode
- Barrier-protecting foil laminated with UV-cured epoxy

PC was used as the injection overmoulding material. Overmouldings were done with a conventional hydraulic injection moulding machine Engel ES200/50 HL equipped with an ISO 294-3 plate cavity mould. The optimised injection moulding process parameters are shown on Table 1.

<table>
<thead>
<tr>
<th>Table 1. IML process parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature of the IM material</td>
</tr>
<tr>
<td>Temperature of the mould</td>
</tr>
<tr>
<td>Pressure limit</td>
</tr>
<tr>
<td>Rate of injection</td>
</tr>
<tr>
<td>Packing pressure</td>
</tr>
<tr>
<td>Dose</td>
</tr>
</tbody>
</table>

Figure 3 shows pictures of the samples after overmoulding. OLEDs are still emitting light despite the harsh conditions of the injection moulding process.

SUMMARY
The embedding of flexible OLEDs into polymer matrix using the commercial IML/injection overmoulding process was demonstrated for the first time. A patent application has been filed for the invention [2].
BUSINESS POTENTIAL
The first applications for OLEDs integrated into plastic products will probably be in the area of smart signage and packaging: OLED technology can be used to highlight information or enhance the visual appearance of packages, toys etc. Polymer embedded OLEDs could also find their way into automotive, consumer electronics and health care products to create signage, display and lighting elements integrated into mechanical structures.

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ACKNOWLEDGEMENTS
This work was funded by VTT.
Combining inorganic material with a polymer-based solar cell helped to improve the shelf life and the stability of the solar cell. The buffer layer of 5nm CdSe between the photoactive layer and metal cathode enhanced the shelf life of a polymer bulk heterojunction solar cell.

INTRODUCTION

In the past seven to eight years, improved understanding of the operational principle of polymer-based photovoltaic devices has led to a rapid increase in the power conversion efficiencies of such devices. Polymer solar cells based on poly(3-hexylthiophene) (P3HT)/ phenyl-C61-butyric acid methyl ester (PCBM) blends have shown high potential for practical application. To date, the efficiency of P3HT:PCBM-based solar cells can reach up to 5% [1]. Such cells have also been proven more stable: the efficiency of solar cells using these components changed by less than 20% during 1000 h of light soaking at 70°C under an inert atmosphere [2]. To some extent, the low-cost structure of polymer-based solar cells can compensate for the shorter shelf life and the lower efficiency [3].

MATERIALS AND METHODS

The devices were fabricated by spin-coating a blend of P3HT (Rieke Metals)/PCBM (Nano-C). The blends were weighted with the ratio of 1:1 in the solid state, and then dissolved in distilled 1,2-dichlorobenzene with the concentration of 40 mg/ml. The ITO-coated glass substrates were cleaned by ultrasonic treatment in deionised water, acetone and isopropyl alcohol sequentially. Shortly after the UV-ozone treatment, a layer (about 40 nm) of polyethylene dioxythiophene/polystyrene sulphonate (PEDOT:PSS) (Clevios P VP A1 4083) was spin-coated. The PEDOT:PSS layer was fabricated by heating for 20 min at 150°C. After that, the P3HT:PCBM solution was spin-coated in the air to form an active layer on the PEDOT:PSS film. A thin buffer layer of CdSe was applied using E-beam deposition. The thickness of CdSe was about 5 nm. The devices were also completed by E-beam deposited Ca (25nm) and Ag (80nm) as the cathode. The shadow mask of ~10 mm² utilised during cathode deposition defined the active area of the device. All devices were encapsulated by the UV-cured epoxy encapsulating glue (DELO 681) in the glove box with nitrogen atmosphere. The structure of the device is shown in Figure 1.

All the characterisation was done in ambient air at room temperature. Steady state illumination tests were carried out under AM1.5G irradiation using a 300W Cermax lamp-based solar simulator. The illumination intensity is AM 1.5 (86±1 mW/cm²). Current density–voltage curves were measured with a Keithley 2400 source measurement unit. The UV–Vis absorption spectra were recorded on a Varian Cary 5000 spectrophotometer.

RESULTS

Figure 2 (a) and (b) present J-V characteristics of the encapsulated P3HT:PCBM composite devices with and without an interlayer of CdSe (5nm) during 130 days for the shelf life test. The $V_{oc}$, $J_{sc}$, FF and $\eta$ for the P3HT:PCBM sample were initially 0.59 V, 5.73 mA/cm², 0.65 and 2.41%, respectively. For the sample with the interlayer of CdSe, the parameters for the first-time test were 0.56 V, 5.72 mA/cm², 0.60 and 2.14%. The decreased efficiency of the sample with the CdSe interlayer may come from the increased serial resistance. In spite of the slight decrease in efficiency, the product life of the device with the buff-

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Figure 1. The schematic structure of solar cell device.
er layer of CdSe clearly increased. During 130 days, 8 J-V tests were done under illumination and in the dark. The sample without the CdSe interlayer gradually produced a concavity in the fourth quadrant which had a detrimental effect on the solar cell’s performance, while the J-V characteristics of the sample with the interlayer presented much less degradation. After 130 days, the $V_{oc}$, $J_{sc}$, FF and $\eta$ for the as-produced P3HT:PCBM sample decreased to 0.53 V, 5.24 mA/cm$^2$, 0.29 and 0.92% and for the sample with the CdSe interlayer, the parameters came to 0.53 V, 5.86 mA/cm$^2$, 0.47 and 1.71%. The $\eta$ decreased by 62% and 20%, respectively, for the P3HT:PCBM sample and the P3HT:PCBM/CdSe (5nm) sample. As can be seen, the decline in FF is one of the most crucial factors in the degradation of the device. According to the absorption spectra (the inset) of the encapsulated P3HT:PCBM film, there was no major difference after two months. Therefore, the significant degradation of the sample without the CdSe interlayer should not come from the photoactive layer, but from the contact on the cathode side. The buffer layer of CdSe can impede the erosion of moisture and oxygen in the air through stable inorganic materials. Another possibility is that the permeability of Ca on the photoactive layer causes the degradation of the devices. The CdSe interlayer avoids the direct contact of metal Ca and polymer, which improves the stability of the devices.

SUMMARY

By introducing an inorganic buffer layer into polymer-based solar cells, the product life and stability can be improved. During a 130-day shelf-life test, degradation in the device with the CdSe buffer layer was only one-third of that of the compared normal device.

BUSINESS POTENTIAL

In recent years, polymer-based bulk heterojunction solar cells have become more attractive because of their potential use in all-solution processes on flexible substrate. Thus, such cells could be manufactured in large volume using low-cost printing technology. Due to the absorption efficiency and unstable properties of polymers, the low efficiency and the shorter life of polymer-based solar cells are two major challenges. Introducing an inorganic material layer could be a promising solution that would not affect all-solution processing, since the development of nanotechnology has led to the creation of some solution-processed inorganic materials, such as TiO$_x$, ZnO, CdSe etc.

ACKNOWLEDGEMENTS

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Organic Transistors

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Organic transistors are studied from the perspective of printable electronics. Conjugated polymers are most commonly applied as semiconductors, but soluble oligomers can also be used. VTT’s effort focuses on the manufacture of transistors as well as device characterization, both for field-effect transistors and other novel types of transistors and related devices. As part of the device manufacture by printing methods, ink formulation is a major topic.

INTRODUCTION

According to the market forecast by IDTechEx, the market for printed and potentially printed electronics will rise from $1.92 billion in 2009 to $57.16 billion in 2019 [1]. Enabling this kind of market growth involves some major technological challenges in basic electronic building blocks such as organic transistors. The general technological targets are to achieve a significant improvement in the performance of the transistors and circuitry without compromising the broader processing concepts [2]. This is the main approach adopted by VTT in this research field.

MATERIALS AND METHODS

VTT’s organic transistor work comprises a wide range of topics in the area of printable electronics. Except for the chemical synthesis and optimisation of the actual base materials (polymers), all research areas related to organic transistors are covered.

The main activities in the transistor area are:
- Basic research of OFET device operation
- Applications of OFETs, especially printable devices
- Printability of conjugated polymers and insulators for thin film transistors
- Device and circuit optimisation for printable and low-cost electronics using organic transistors
- Printing ink formulation for transistor dielectric and semiconductor materials

We target mass production and low-cost applications and therefore aim processing at normal laboratory facilities, i.e. no inert atmosphere, no clean room requirement, commercially available materials or materials available through partners in sufficient volumes, etc.

We address all relevant layers and interfaces in the transistor device; from the electrode $\equiv$ conjugated polymer interface and insulator $\equiv$ semiconductor interface to gate dielectric thickness and morphology optimisation. The basic device used for transistor materials characterisation is the standard OFET device either in top-gate (shown in Figure 1) or bottom-gate configuration.

Figure 1. A OFET device structure used for basic characterisation.
Device processing is typically done using desktop printing tools or pilot printing machines. To reach our goal, both processing methods and materials must be modified and optimised, and major research efforts go into finding generalised recipes or process descriptions and into describing the relationship between process parameters (self-defined) and device performance parameters (measured data).

Due to lack of available printable electrode materials, metals are most commonly applied as circuitry materials and electrodes. The insulators used are typically standard polymers of good quality such as polymethyl methacrylate (PMMA) and polyvinylphenol (PVP). However, these are also modified and mixed with modifiers or other components in order to reach our research goal. Even non-printed materials and methods are used, such as metal oxidation, atomic layer deposition (ALD) and self-assembled monolayers (SAMs). For improved functionality and performance, multilayer gate dielectrics are often required. Polymer semiconductors are typically conjugated polymers such as polyalkylthiophenes and related materials.

RESULTS
Organic transistor research at VTT is mainly conducted as commission research in close co-operation with external partners. Some research, however, is of a general nature or partly performed in public projects. As a research example we here consider the use of an anodised gate dielectric or ALD dielectric in an OFET-type device using a standard commercially available conjugated polymer as a semiconductor (“PTAA”).

In this work, Al$_2$O$_3$ has been found to be the most promising approach to date, yielding OFETs that operate in air at -4 V with a high yield on both glass and PET-504 substrates. Different intermediate layers between the oxide and the semiconductor of thin organic dielectrics (poly(isobutylene) and poly(a-ethyl styrene) or self-assembled monolayers (octadecyltrichlorosilane (ODTS) and n-tadecylphosphonic acid) were tested to improve the OFET performance. ODTS proved to yield the best results.

The stability of the Al$_2$O$_3$ OFETs was studied over different periods of time. Even after eight months in the air, the OFETs still worked, but their performance degraded. Studies of the stability are under way to clarify the mechanisms behind this performance.

Additional tests with alumina were conducted. First, tests were done using anodisation in citric, chromic and sulphuric acid, but no significant difference was found (at least with these <10nm films). There were some hysteresis in all. Vth was -0.5V when processed in N2. When the ALD method was used (5 and 15 nm dielectric), 15nm-thick devices worked well, showing only minor hysteresis, and 0 V Vth. Plasma oxidation yielded better performance than anodised samples, with negligible hysteresis and -1 to -1.5V Vth. The results were not dependent on the substrate material; PET, PEN and glass substrates were tested. The oxide surfaces were characterized with AFM showing quite similar surfaces of good quality. Only ALD films had some significant defects. Plasma oxidation is very promising, but implementation to R2R system could compromise the quality of the oxide film.

The conclusions for this work are that after having considered top- and bottom-gate OFETs, both can be fabricated with R2R processes. Anodized Al$_2$O$_3$ is only suitable for bottom-gate devices, while the cross-linked polymers are, at least in theory, suitable for both. We have demonstrated that anodized Al$_2$O$_3$ can be used for both n- and p-type OFETs. This suggests that the approach is suitable for complementary transistor technology [3].
SUMMARY
Thin, light-weight, flexible, and inexpensive products enabled by organic transistors are entering the market during the next years. The best potential for this technology is in applications where large-area fabrication is a necessity but the transistor performance requirements are eased. VTT contributes to the ongoing very rapid progress in this area by developing materials, transistor structures, processes, characterization, and applications.

BUSINESS POTENTIAL
Among the different segments of printed electronics, the market for printed transistors will be especially interesting during the next decade. This market, currently almost non-existent, is expected to grow to $8 billion in ten years.1 It will be boosted by backplane transistor technology, which will be sold as part of active matrix displays and electronic reader applications.

VTT’s development of roll-to-roll based key technology for large-area manufacturing of flexible organic transistors and electronic circuits will enable a large variety of more advanced devices. The applications with the most potential for VTT’s technology in the field of printed transistors are packaging and displays. However, VTT’s development of the fundamental electronic building blocks and fabrication platforms will also impact other areas of organic electronics such as sensors, memories, batteries, solar cells, lighting and any combinations of these devices.

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ACKNOWLEDGEMENTS
The PESEP project was funded primarily by Tekes, which made the research example possible. Niklas Björklund and Prof Ronald Österbacka from Åbo Akademi are acknowledged for close collaboration on the aluminiumoxide transistor example.
PriMeBits - Printable Memory Solutions for Sensor, ID, and Media Applications

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In the PriMeBits EU FP7 project [1], printable electric low-voltage non-volatile memories are developed. The project builds on basic research of new materials and components and takes the results into prototypes relevant for new applications. The project started on 1.1.2008 and runs until 31.12.2010.

INTRODUCTION
Current printable polymer-based memory technologies typically suffer from i) a need for high operating voltage, ii) an extremely short lifetime in room atmosphere, iii) poor temperature stability, iv) chemically reactive materials that require encapsulation and/or v) time-consuming temperature-annealing steps in fabrication. Consequently, for many commercially attractive passive and battery-powered applications, the properties of current printable memories are unsuitied.

To overcome the shortcomings of prior-art approaches, printable inorganic metal-oxide nanoparticle-based ferroelectric FRAM memory and a resistive metal-based write-once-read-many (WORM) memory based on the rapid electrical sintering (RES) technology of VTT [2] is developed. To print the ferroelectric memory, new printing inks based on, for example, barium titanate (BaTiO3) nanoparticles are developed. For the WORM memory, the project utilises commercial metal nanoparticle inks.


MATERIALS AND METHODS
WORM Memory
The WORM memory is based on the electrical sintering technology of VTT [2], the basic principle of which is shown in Figure 1. The operation is based on a nanoparticulate (such as silver) ink that is used to print a conductor structure. After printing and drying this structure (i) is composed of separated and possibly encapsulated nanoparticles, (ii) has a high resistance (iii) conducts current via inter-particle tunnelling or leaky encapsulant and (iv) has a negative temperature coefficient of resistance. Consequently, a constant-voltage boundary condition, as indicated in Figure 1, results in a sintering process with positive feedback where the resistivity of the material typically changes several orders of magnitude in a time scale of a few microseconds [2].

Figure 1. The principle of electrical sintering.

FRAM Memory
The FRAM memory is based on ferroelectric polarisation that can be controlled with an external field. The polarisation has two states to represent the two memory bit levels. Typical materials suggested for printed ferroelectric memories are the polymer-based PVDF/TrFE derivatives. However, to achieve low-voltage operation, the inorganic metal-oxide nanoparticle approach is seen more promising as illustrated in the material comparison of Table 1. In particular, the voltage level needed to read and write the memory content is determined by the coercive field (polarisation flipping field) which is several tens of volts for PVDF for a 1 µm thick material layer.
GENERIC TECHNOLOGIES

RESULTS
Low-voltage/low-power WORM operation has been successfully demonstrated and quantified using silver-nanoink structures inkjet printed on photopaper (VTT, MIUN). Successful WORM writing has been demonstrated using flexible batteries (MIUN, VTT). The readout of the WORM coupled to an RFID antenna was tested as schematically illustrated in Figure 2 (MIUN).

The synthesis process for ferroelectric barium titanate nanoparticles is developed (INM) and ferroelectric nanoparticle inks have been prepared (Evonik). For the FRAM, the obtained results during the first project year are encouraging. For the non-printed prototype systems, \( P_r > 1.5 \mu C/cm^2 \) @ 25 V/µm and a storage time of the order of hours has been demonstrated (INM). For the fully-gravure-printed systems, \( P_r \sim 0.05 \mu C/cm^2 \) @ 10 V/µm and a storage time lasting minutes has been demonstrated (VTT). Furthermore, the demonstrated tunability of the ferroelectric capacitor structures is also interesting outside memory applications. Figure 3 shows a switching-current measurement of a fully-gravure-printed FRAM memory bit shown in Figure 4. The output current, which is higher for the first voltage pulse of the two consecutive pulses of same polarity, is evidence of flipping ferroelectric polarisation. Piezo-force-microscopy analysis (EPFL) has also proved the existence of true ferroelectric polarisation in printed samples.

SUMMARY
The PriMeBits project is developing new printed-electronics memory solutions that overcome the challenges of current state of the art, for example, in required voltage levels. The project focuses on inorganic nanoparticle-based materials to develop a metal-based WORM (write-once-read-many) and a metal-oxide-based ferroelectric RAM memory. In terms of reaching the project goals, the first-year results are encouraging.

BUSINESS POTENTIAL
The application analysis done in the project (Motorola, Sensible Solutions, Ardaco, UPC, Stora Enso) has resulted in several potential applications of the developed technologies in the areas of (i) event-logging/event-counting sensors with permanent data storage. Here storage of level-crossing information of temperature

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**Table 1. Material comparison of PVDF/TrFE and Ba-**

<table>
<thead>
<tr>
<th>Memory property:</th>
<th>Reading and writing voltage</th>
<th>SNR / bit area</th>
<th>CMRR / coupling</th>
<th>Max temp.</th>
<th>Processing temp.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Relevant material property:</td>
<td>Coercive field / [V/µm]</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>bulk BaTiO3</td>
<td>0.05</td>
<td>26</td>
<td>1000 - 2000</td>
<td>120</td>
<td>1650</td>
</tr>
<tr>
<td>Ceramic BaTiO3</td>
<td>0.5</td>
<td>5</td>
<td>1000</td>
<td>120</td>
<td>1000 - 1400</td>
</tr>
<tr>
<td>spin-coated thin-film BaTiO3</td>
<td>5.3</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>spin-coated porous thin-film BaTiO3</td>
<td>9</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>bulk &amp; thin-film PVDF/TrFE</td>
<td>50</td>
<td>7.4</td>
<td>10</td>
<td>100 - 120</td>
<td>150</td>
</tr>
</tbody>
</table>

---

**Figure 2.** The schematics of a WORM coupled to an antenna in parallel to an RFID chip.
(cold chain) or humidity (dry food, moisture in buildings, healthcare) are of interest. (ii) Smart cards such as game cards, e-wallet or greeting cards with dynamic data. (iii) RFID applications in general.

REFERENCES
[1] www.primebits.eu

ACKNOWLEDGEMENTS
The project is funded by the European Community’s Seventh Framework Programme (FP7/2007-2013) under grant agreement 215132.

Figure 3. Switching-current testing of a FRAM memory bit of Figure 4.

Figure 4. A memory bit of Figure 3.
The demand for low-cost electronic memory is rising. This has created a need for new manufacturing methods such as printing techniques and soluble materials. We present a device concept that could replace traditional electronic memory especially in certain low-cost applications.

DEVICE CONCEPT

A sandwich structure made on flexible substrates is presented using polymers and other organic materials. Only three layers, of which two are electrodes, are required for the device, none of which involves the use of stringent patterning or positioning. Thus, the memory device is potentially well suited for printing processes and very low-cost manufacture.

RESULTS

The active material can be a nano-structured film with charge acceptor domains embedded in an insulating matrix. Fullerene (C$_{60}$ or PCBM (functionalised C$_{60}$)) mixed with polystyrene (PS) was used as the functional nano-structured material (Figure 1).

Apart from electrical bistability, the PS/C60 devices also show a negative differential resistance effect which may be utilised in a memory device.[1] Additionally, we found that the threshold voltage values can be tuned by changing the composition, as shown in Figure 2.[2]

Metallic particles like Co or Au were also shown to produce NDR in an insulating matrix. On the other hand, if the layer thickness is below 100 nm, the chemical and electronic properties of the middle layer are no longer controlling the device operation. Then the operation is based only on filamentary conduction through the middle layer, arising from the defects on the bottom electrode. We showed that there are no differences if a partly conjugated triblock copolymer, polystyrene-block-9,9-di-n-hexyl-2,7-fluorene-block-polystyrene PS-DHF-PS (mimicking the semiconducting particles in an inert matrix), or a pure insulating polymer, polystyrene, is used as the middle layer [3].

The negative differential resistance can be utilised in the memory devices by applying short voltage pulses of high and low levels. The programming of both the polymeric and metallic nanoparticle-based devices was possible by applying a voltage pulse of 3–4 V to turn the device to a highly conducting ON state and a voltage pulse of 8–9 V to turn it again to the OFF state with low conduc-

Figure 1. The combination of a TEM micrograph and a schematic illustration showing the polarisation between the PCBM clusters separated by the PS matrix. Reprinted with permission from Laiho, A., et al., Applied Physics Letters, Vol. 93, pp. 203309, 2008. Copyright 2008, American Institute of Physics. [2]
Figure 2. a) Absolute current density as a function of voltage for pure PS (200 nm) and 2-6 wt % PCBM/PS compositions (250-260 nm) and b) $V_{th}$ as a function of PCBM concentration. Reprinted with permission from Laiho, A., et al., Applied Physics Letters, Vol. 93, pp. 203309, 2008. Copyright 2008, American Institute of Physics. [2]

Figure 3. Programming cycles of (a) the triblock copolymer and (b) 5% Co vs. PS250k between Al electrodes.
tivity. The state of the device was read with 1 V. Figure 3 shows some examples of the programming cycles for a device with Co-particles in a PS matrix and for the pure triblock copolymer.

Our results also show that the shape of the $I-V$ curve of the polymeric devices flattens out as the device area increases. This suggests that the probability of having enough working filaments is higher when the area is large. Thus miniaturisation below sub-mm devices is not possible with such simple devices. Nanoparticles in the matrix would help to control the smaller devices, but when printable and cost-effective devices are sought, this is not the optimal solution. Additional defects, e.g. patterned bottom electrodes, could be the answer for printable devices. Thus a critical step in forthcoming work would be to nucleate the filament-forming process.

BUSINESS POTENTIAL

Despite the fact that the cost per bit for Si based memories is negligible in large storage devices, for low storage applications (logistics, identification, anticounterfeit, toys, electric cards etc.) the situation is different. Here, the cost of the chip attachment becomes the dominant factor. Thus, printed memories integrated with other functionalities (electrical and/or optical), offers the main business potential for such devices.

ACKNOWLEDGMENTS

This work was performed in collaboration with the Molecular Materials Group at the Helsinki University of Technology and the Physics Department at Åbo Akademi University, and is partly funded by TEKES through the FinNano program.

REFERENCES


The aim of the FACESS project is to manufacture a flexible, autonomous energy source and storage device on flexible foil using cost-efficient roll-to-roll manufacturing techniques. The energy storage device consists of an organic photovoltaic module which harvests light energy and a thin film battery module that stores the energy provided by the photovoltaic module. Battery use is optimised using a control transistor circuitry implemented on a thinned silicon chip interconnected to the flexible foil.

INTRODUCTION

Several individual components such as OLEDs [1], OFETs [2] and OSCs [3] have been demonstrated and manufactured using cost-efficient roll-to-roll (R2R) manufacturing technologies. Generally, developments in flexible organic and large-scale electronics are heading towards larger operational devices and systems. This means that different electronics functionalities have to be integrated on flexible substrates.

The FACESS project addresses this challenge for integration. A flexible autonomous energy source and storage device is placed on flexible foil using the cost-efficient R2R manufacturing techniques shown in Figure 1. The energy storage device consists of an organic photovoltaic module which harvests light energy and a thin film battery module that stores the energy provided by the photovoltaic module. Battery use is optimised using a control transistor circuitry implemented on a thinned silicon chip interconnected to the flexible foil.

MATERIALS AND METHODS

In the FACESS project, three different components will be developed: an organic solar cell (OSC) module, a battery module and a control transistor circuitry. OSC and battery modules are fabricated using R2R technologies, mostly printing techniques. The transistor circuitry is produced using traditional microelectronics processes.

RESULTS

In the OSC module, research focuses on ITO anode patterning using R2R lift-off and etching processes. As an alternative to ITO, highly conductive PEDOT and metal grids have been considered as the anode. Gravure printing has been studied for printing the hole transporting and active polymer layers. Ink formulation optimisation for the gravure printing process has been very extensive. The current record for the photon conversion efficiency with commercially available materials in FACESS is 2.8% (the target percentage is 2.5%). Most efforts are now aimed at upscale production of pilot machines to start manufacturing the OSC modules.

The main focus of battery module research has been on printing the positive and negative electrode materials on current collector metal foils. Flexography has mainly been used but in order to obtain thicker layers and thus a higher battery capacity, screen-print is also used. Good electrical behaviour has been achieved for printed batteries (a capacity of over 50 mAh). An important part of the battery is the membrane upon which deposition methods have been developed.
Because the power of the OSC module depends on ambient lighting, a special algorithm has been developed for the control transistor circuitry to maintain the optical charge of the battery. This algorithm provides a battery charge even if not all OSC modules are operational. The circuitry is implemented on silicon with a chip size of 12.3 mm² and it is thinned down to 30 µm in order to make it flexible. Chips will be fabricated using the 0.35 µm CMOS process.

The OSC module area is 100 cm² and the goal is to attain 250 mW of power. The open circuit voltage of the module is approximately 4V. The OSC submodule consists of 8 solar cells connected in series and the OSC module has a total of 4 submodules. The OSC consists of a patterned ITO anode, a gravure printed hole transporter, active layers and R2R evaporated cathode.

The battery module area is 30 cm² and the goal is for the battery to have a capacity of approximately 90 mAh. The anode current collector is copper and on top of it, a graphite electrode is printed using flexography or screen-print. The cathode current collector is aluminium and on top of that, a Li-based positive electrode supplied by Umicore is printed using flexography or screen-print. The battery membrane is deposited between electrodes.

Because the performance of the active organic electronics is still relatively low, traditional silicon electronics are used in the control transistor circuitry. In order to make the chip flexible as well, it is later thinned down to 30 µm and then interconnected to the flexible backplane using a suitable interconnection method. The system layout for the FACESS device is shown in Figure 2.

The interconnection methods to attach the chip on the backplane have been studied. The most promising techniques involve the flip-chip interconnection using a combination of conducting and non-conducting adhesives or the direct printing of conductors from the substrate to the active surface of the chip with screen-print technology as shown in Figure 4.

**SUMMARY**

The goal of the FACESS project is to manufacture a flexible, autonomous energy source and storage device on flexible foil using cost efficient roll-to-roll manufacturing techniques. The energy storage device consists of an organic photovoltaic module which harvests light energy and a thin film battery module that stores the energy provided by the photovoltaic module. Battery use is optimised using a control transistor circuitry implemented on a thinned silicon chip interconnected to the flexible foil.

**Figure 2.** The system layout of the FACESS device.

**Figure 3.** The Printed OSC module.

**Figure 4.** Screen printed conductors for making interconnections to a 30 µm thin test chip on PEN substrate.
BUSINESS POTENTIAL

The FACESS project has especially emphasised demonstrator planning. Because the power capacity of the device will be relatively low (a few hundred mWs), the main applications selected will be focused on low power wireless sensor networks. This kind of flexible tags with rechargeable power sources will prove useful for many different applications. In the future, when the efficiency and capacity of printed power sources increases, the potential application field will be much broader. A special demonstrator platform that uses FACESS OSC, battery components and the ZigBee radio module has been developed (see Figure 5).

REFERENCES


ACKNOWLEDGEMENTS

We would like to thank the European Commission via the 7th framework program for funding the FACESS project (grant agreement FP7-ICT-1-215271) and the following project partners: the Interuniversity Micro-Electronics Centre, Commissariat à l’Energie Atomique, Politechnika Warszawska, Umicore SA, Coatema Coating Machinery GmbH and Suntrica Oy.
The GreenBat project focuses on the development of printable environmentally friendly thin film batteries. The aim is to make the manufacturing process cost efficient by utilising printing processes and roll-to-roll manufacturing methods. In addition, novel material solutions for different battery layers are developed. GreenBat project is funded by the European Union 7th framework programme and is coordinated by Varta Microbattery GmbH.

INTRODUCTION
Several independent analysts have concluded that there will be a growing need for thin film batteries in future. For example, a forecast of NanoMarkets suggests that the demand for thin film batteries is going to rise rapidly in the following years. The market of these batteries is expected to rise from $41 millions to $230 millions between 2006 and 2011. Main driver for this growth are different electronic smart products like smartcards, RFID tags, sensors etc.

Thin film batteries are typically manufactured by combining different techniques. The purpose of the GreenBat project is to develop manufacturing techniques that would enable whole battery to be manufactured by printing in roll-to-roll process. This would enable the batteries to be manufactured in a cost-efficient way. The other objective is to make the batteries environmentally friendly by substituting the harmful solvents used nowadays in battery manufacturing with less toxic chemicals. Battery chemistry is based on lithium ion chemistry that is investigated in the project by Commissariat à l’Energie Atomique (CEA).

VTT’s role in the project is to develop R2R production process for the batteries. This includes printing of active anode and cathode layers as well as assembly of the printed battery stack.

Figure 1. The general structure of a printed battery. In printed batteries, the different functional inks are printed on top of one other. Current collectors can act simultaneously as shell materials.

Project is coordinated by Varta Microbattery GmbH and other partners are: Commissariat à l’Energie Atomique, Plastic Electronic GmbH, Norbert Schläfl i Maschinen and Imperial College

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NanoMarkets http://www.nanomarkets.net/ Printed Battery Markets; February 2008

ACKNOWLEDGEMENTS
We wish to thank the project partners (Varta Microbattery GmbH, Commissariat à l’Energie Atomique, Plastic Electronic GmbH, Norbert Schläfl i Maschinen and Imperial College) and the European Union for funding, grant agreement FP7-ICT-2-224502.
In the MEMS DISPLAY project, all needed roll-to-roll production technologies were demonstrated for the manufacturing of Fabry-Pérot interferometer based MEMS (microelectromechanical system) display pixel on a flexible polymer substrate.

INTRODUCTION

The University of Tokyo has demonstrated an MEMS display based on a Fabry-Pérot interferometer on flexible polymer substrates using conventional MEMS manufacturing technologies [1]. In this project, VTT and the University of Tokyo were developing a suitable roll-to-roll (R2R) manufacturing technology for this type of display. The objective of the project was to develop a flexible large area MEMS display technology based on a Fabry-Pérot interferometer.

The structure and the operating principle of the MEMS display is illustrated in Figure 1. The display is built on a plastic substrate. A patterned bottom electrode layer is above the substrate and on top of that, there is an optical spacer layer. Above the optical spacer is a patterned air-gap spacer which forms the Fabry-Pérot cavity with the top layers of the display. The top layer consists of a deformable membrane with a top electrode.

MATERIALS AND METHODS

The manufacturing of MEMS display elements involves three different processes: electrode patterning, the deposition of the optical spacer and air gap spacer printing, which is combined with the electrodes lamination process.

The first step is to manufacture and pattern the 20 nm thick metal electrode layer. This is done in a lift off process where the reverse pattern is printed using flexography on the surface of the base substrate with soluble ink which works as resist. Actual metal electrodes are then sputtered or evaporated over the printed substrate. After metallisation, the resist is released with ultrasonic-assisted washing which also removes the metal layer from the printed patterns and lines. Then metallisation remains only on unprinted areas of the substrate surface.

The second step is to coat the whole electrode with an optical spacer which serves as a dielectric and waveguide layer. In the demonstrators of this project, the thickness target values for the RGB colour layer were 160 nm, 325 nm, and 245 nm, respectively. Optical spacer can be manufactured by sputtering SiO2 or printing using direct or reverse gravure techniques with suitable dielectric coating. Polysiloxanes were used in this project.

The third step is to print (using gravure or flexography) an air gap spacer layer with adhesive. Two-component polyurethane adhesive was used. The upper electrode is laminated together with a base substrate in line.

RESULTS

In this project, the operative pixels were demonstrated using vacuum-coated silver electrodes on the PET substrates. The layer thickness of the electrode metal was 20 nm and it was patterned using the R2R lift-off method. Gravure- and flexography R2R printed release layers were demonstrated and an ultrasonic assisted R2R release process in acetone was used with a speed of 2 m/min in the pilot line [2].

Figure 1. The Fabry-Pérot colour filter operating principle.
The optical spacer layer, which acts as the waveguide and dielectric layer, was demonstrated with direct- and reverse gravure R2R printing in the pilot line. Interfering layer thickness is in the order of hundreds of nanometres; all RGB main colours have their own layer thickness in orders of wavelength since the colour is determined by the optical characteristics of the spacer material and the thickness of the layer.

Thickness has to be adjusted to a level of 10 nm in order to output certain colour.

The air gap spacer layer was gravure printed with two-component polyurethane-based adhesive. Black pigment was added in order to improve the contrast. Layer thickness can be adjusted by varying the printing parameters. A lower air gap structure enables lower operation voltage but a drawback is that the adhesion between electrode films will decrease as a consequence of the thinner adhesive layer. Figure 2 shows red, green and blue Fabry-Pérot MEMS pixels. The demonstrator’s air gap structure is gravure-printed on a spin-coated optical spacer made of polysiloxane. The operation voltage is 25 volts.

**SUMMARY**

RGB Fabry-Pérot MEMS display pixels prepared using printing techniques were demonstrated. All printing techniques were also shown in a pilot roll-to-roll environment. All the sub-process techniques demonstrated also have potential for several other applications besides MEMS.

**BUSINESS POTENTIAL**

The described MEMS display has potential, for example, in poster applications and in signage, combined sensing and displaying applications like touch panels with signage features or signage boards with touching sensors features.

Even though the optical spacer layer thicknesses must be adjustable at the nm level, functional and flexible electronic devices can be manufactured by R2R methods with line widths of as wide as 50-100µm in lateral resolution. That is wider than in many other printed electronic devices. Low resolution requirements in lateral dimension enables fast scalability to industrial scale production.

**REFERENCES**


**ACKNOWLEDGEMENTS**

The project was funded by VTT, the Finnish Funding Agency for Technology and Innovations (TEKES) and the New Energy and Industrial Technology Organisation (NEDO) of Japan.
Online Inspection in Printed Electronics Production

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For high-volume printed electronics production, online quality control is essential. Optical measurements can be used, e.g., to analyze substrate properties like surface energy and print quality for thin film layers and thick film prints like conductors. The feasibility of contact angle measurement, 2D absorption imaging and 3D topography measurement using chromatic aberration and structural light is demonstrated.

INTRODUCTION
Roll-to-roll printing can be used for the high-volume production of the components and modules of electronics and optics. However, without sufficient feedback from printed component properties to printing controls, the economical benefit of high-volume production can easily be lost. The technologies especially suitable for online testing are non-contacting optical measurements. Measurements are needed, e.g., to analyze the properties of materials, the integrity of printed thin films and the topography of thick film conductors depositions. This study has especially focused on the feasibility of the online analysis of surface potential, the machine vision methods for the active layers of organic components and the 3D optical measurements.

MATERIALS AND METHODS
The main topics studied here are:
- Online measurements of the surface energy of printing substrates
- The print quality in the thin film layer of active organic components
- Online topography measurements suitable for printed conductor and antenna cross-section volume analysis

Contact angle measurement is an essential method for studying the surface properties on printing surfaces—which again has crucial effect on print quality. Measurements are done in the laboratory, using samples of substrate onto which droplets of analyzing liquid are dispensed and imaged. The contact angle can be calculated from this image and the surface energy can thus be analysed. Here high speed imaging has been used to study the contact angle on a moving web. Imaging is based on short pulse LED illumination, which enables the use of standard, low-cost machine vision cameras.
Similar imaging technology can be used for the thin film absorption measurement e.g. OLED active layer quality characterization.

Chromatic aberration 3D imaging is the online topography measurement method, which exploits colour aberrations in the depth sensing of surfaces and multi-layer structures. The measurement principle is illustrated in Figure 1. This method is based on intentionally produced high-colour aberration illumination and confocal spectral imaging. [1] As presented in the figure below, different wavelengths represent different z depths in imaged signal.

Alternatively, conductor topography can be measured online using structural light technology. Here a three-dimensional image is created from several images taken from a location subject to varying lighting conditions – e.g. a sinusoidally modulated light pattern.[2] The laboratory setup for structural light imaging is shown in Figure 2.

RESULTS

On-line Contact Angle Measurement

An image of the dispensed analysis liquid on the moving web is shown in Figure 3. As seen in this figure, short pulse driving of high brightness LED provides the illumination necessary for a high-quality still image of a droplet. The surface energy can be analysed from this image. In this project, image interpretation is done by Biolin Scientific with analysis software used in Atten-sion tensiometers [3].

Thin Film Measurements

An example of thin film printing quality imaging is shown in Figure 4. Even without any computational analysis, the picture reveals the imperfections of the film’s uniformity. Images can be analysed for fill factor calculation, edge roughness etc.

For printed conductive structures, the conductor thickness and cross-section are essential parameters in terms of conductivity. Offline feasibility studies show that the
3D structure can be measured from antenna structures. In Figure 5, a sample topography measurement is show.

**SUMMARY**

Optical measurements can be used for online inspection during the production of printed electronics. Evaluated methods can be used to characterize contact angle measurement, thin film uniformity and conductor topography.

**BUSINESS POTENTIAL**

For cost-effective production, controlling the process - and therefore attaining a high yield - is essential. The technologies described here can form a significant part of the measurements needed for process control. Additionally, the process instrumentation business itself can grow to a significant level as the number of printed electronics manufacturing lines increase.

**REFERENCES**


**ACKNOWLEDGEMENTS**

These studies have been mainly carried out in the Measurements for Printed Electronics and Optics (PEOMIT) project. The project is Funded by Tekes, VTT, Metso, Biolin Scientific, Offcode, Procemex, SR-Instruments and Inspex.
INK-JET AND FLEXO PRINTING OF LACCASE FOR BIOACTIVE APPLICATIONS

Maria Smolander • Stina Grönqvist • Anne Savolainen • Thea Sipläinen-Malm • Jouko Virtanen • Tomi Erho

INTRODUCTION

The interest towards the mass-production of biocatalyst based functional materials for e.g. applications in diagnostics, food packaging and environmental analysis has promoted the development of methods concerning the incorporation of biocatalysts like enzymes into or on textile substrates like paper or plastics by printing or other cost efficient reel-to-reel applicable processes, i.e. manufacturing of bioactive paper. The aim of this work was to obtain general and profound knowledge facilitating the manufacturing of bioactive paper by printing through studying 1) the effect of the printing ink composition on biocatalytic activity and 2) biocatalyst tolerance towards different printing methods and conditions.

In this work the activity and stability of Trametes hirsuta (Th.) and Melanocarpus albomyces (Mal.) laccases in different laboratory grade and commercial ink compositions suitable for ink jet or flexo printing as well as the maintenance of the enzymatic activity during printing procedure and subsequent storage of the dry, printed bioactive layers were studied.

MATERIALS & METHODS

ABTS (2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) was used as laccase substrate. Two printing substrates News, Kaipola, 42 g/m² (UPM) and Yes Bronze 80 g/m² (UPM) were used in printing trials. Ink-jet inks were based either on PVP or CMC, which were mixed together with appropriate solvents and surface active agents. Flexo inks and varnish compounds as well as commercial flexo ink 473-44329 (Sun Chemical) were also studied in experiments.

Ink-jet printed solution contained 85% of ink and 15% of laccase dilution. Laccase containing inks were analyzed in respect to their enzymatic activity after 30 min and 1 d storage. Allozymes of enzyme containing ink were mixed into 1.8 mL of 5 mM ABTS solution (pH 4.5) and the activity of laccase was monitored as consumption of dissolved oxygen in the presence of ABTS.

Based on dynamic luminescence quenching, the dissolved oxygen was monitored by using an Oxy-10 mini sensor oxygen meter (PreSens, Germany) in a closed 1.8 mL reaction vessel. The oxygen consumption was converted into enzymatic activity with the aid of a calibration curve.

RESULTS

Compatibility of ink-jet inks and laccase

Two different basic ink types for ink-jet based on PVP or CMC were studied in respect to the compatibility with the two laccases. The effect of nozzle voltage and printing substrate on the activity of the printed laccases was studied. In general the proportionality of the activity seemed to be better with lower enzyme concentration, but no clear effect of the nozzle voltage or the printing substrate could be seen (Figure).

Laccase inks were printed at ambient temperature on paper by ink-jet printing using FUJIFILM Dimatix DMP-2831 printer. Activity of laccase was monitored as consumption of dissolved oxygen as described above. The compatibility of laccases with flexo inks was studied as described above for ink-jet inks. For the activity analysis, 100 nL of enzyme was mixed with 1 g of ink and the ink solution was diluted with buffer solution.

CONCLUSIONS

Neither the composition of ink-jet printing inks nor the nozzle voltage used in printing had a remarkable effect on the activity of the two studied laccases either in ink or when ink-jet printed. On the contrary the composition of the flexo ink had a considerable effect on the enzymatic activity in the inks. It appeared that laccases are suitable for the production of bioactive layers based on these enzymes using different printing techniques.
ROLL-TO-ROLL PILOT FACILITIES FOR PRINTED INTELLIGENCE

VTT offers roll-to-roll (R2R) pilot manufacturing services of printed electronics and optics. Customer solutions can be tailored depending on the customer's needs to cover the whole manufacturing chain. Alternatively special experiments can be tailored focusing for example to printing or material formulation.

**PICO Pilot printing line**
- 2 gravure printing units
- R2R hot embossing unit
- Corona and lamination units
- Drying units (air, UV, IR)
- Web width 200 mm
- Max. web velocity 100 m/min
- Installed in clean room (ISO7)

**ROKO Pilot Printing line**
- 4 replaceable printing units
  - direct and reverse gravure, rotary screen, and flexography units
- Corona and lamination units
- Drying units (air, UV, IR)
- Web width 300 mm
- Max. web velocity 10 m/min

**PILOT coating line**
- Roll coating (2-roll and 3-roll)
- Spray-coating
- Coating width up to 500 mm
- Max. web velocity 30 m/min
- Drying units (air 5 pcs, UV)
- PLC-guidance
- Corona-unit

**Picosecond roll-to-roll laser processing**

**Roll-to-roll post treatment unit including die cutting and slitter units**
VTT CENTER FOR PRINTED INTELLIGENCE OFFERING

OLEDs
- Roll-to-roll printed flexible OLED
- Printed large area OLED on glass for lightning applications

Printed Indicators for consumer packed goods
- Product ID
- Tampering
- Anticounterfeit
- Use-by - information
- Visual, optical or electrical readout

Pilot scale ink modification
- Ink modification with pilot scale verification
- Thin layer gravure printing

Roll-to-roll hot embossing
- Optical effects for brand marketing, brand protection and security
  - on plastics, on paper, on paper with VTT developed biodegradable coating
  - high efficiency effects on high refractive index coating
- Fabrication of micro channels for e.g. biosensors
- Fabrication of optical component like lenses

R&D activities e.g. in
- Printable transistors
- Printed passives
- Printed sensors
- Silicon & Printed hybrid
- Electrical sintering
- Organic solar cells
- Printed batteries
- Online quality control
- Printed MEMS
- Printed memories
- Biomicrosystems
- Microfluidics
- Point-of-care diagnostics
- Bioactive paper
- Bio fuel cells

http://cpi.vtt.fi
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Research and development activities in printed intelligence

2008 2009 2010 2011