Preface

In many respects 2003 has been a very successful year for the Dutch Polymer Institute. Not only because the research programme confirmed the planned 8% growth from EUR 12.1 to EUR 15.3 million but also because of initiatives taken to strengthen the DPI programme and organisation as well as shape continuity.

The introduction of a ticket system in 2000 has been the key enabler for continued growth. On one hand, it allowed DPI Industrial Consortium members to focus their interests on particular technology areas and, on the other hand, it lowered the barrier for new industrial partners wishing to enter the alliance through the mechanism of a single ticket in a specific technology area. The ticket system for industrial participation, matching by universities and double matching by the Ministry of Economic Affairs have provided the budgetary resources for continued DPI growth. Since the introduction of the ticket system some twelve new companies have joined the alliance at an average of four a year. The first quarter of 2004 has already seen the entry of six new companies.

The system of tickets has been supplemented by the creation of the Core Programme Area, an initiative that allows new technology areas to emerge within DPI's programme. High Throughput Experimentation & Combinatorial Material Research is such a new area. This area, started in 2001 in the core programme, was already supported by six companies in 2003, a figure that is likely is double in 2004. New areas like Plastic Electronics and Biomedical Materials are likely to follow.

The DPI success formula for growth, however, not only imposes high demands and expectations on the DPI management in community, it also raises a number of questions on how to handle growth successfully. For instance, questions about our position as a leader in our technology fields, about the effectiveness of our organisation in triggering innovation with our partners and about our continuity. Answers to these questions were sought through a variety of special initiatives undertaken in 2003: the stakeholders survey, the portfolio analyses and the DPI "future blueprint task force". The results and conclusions were presented at the DPI stakeholders conference in November 2003 in the form of a mission, a focus on excellence and a restructuring of the organisation, all elements of a new five-year business plan to be issued in 2004.

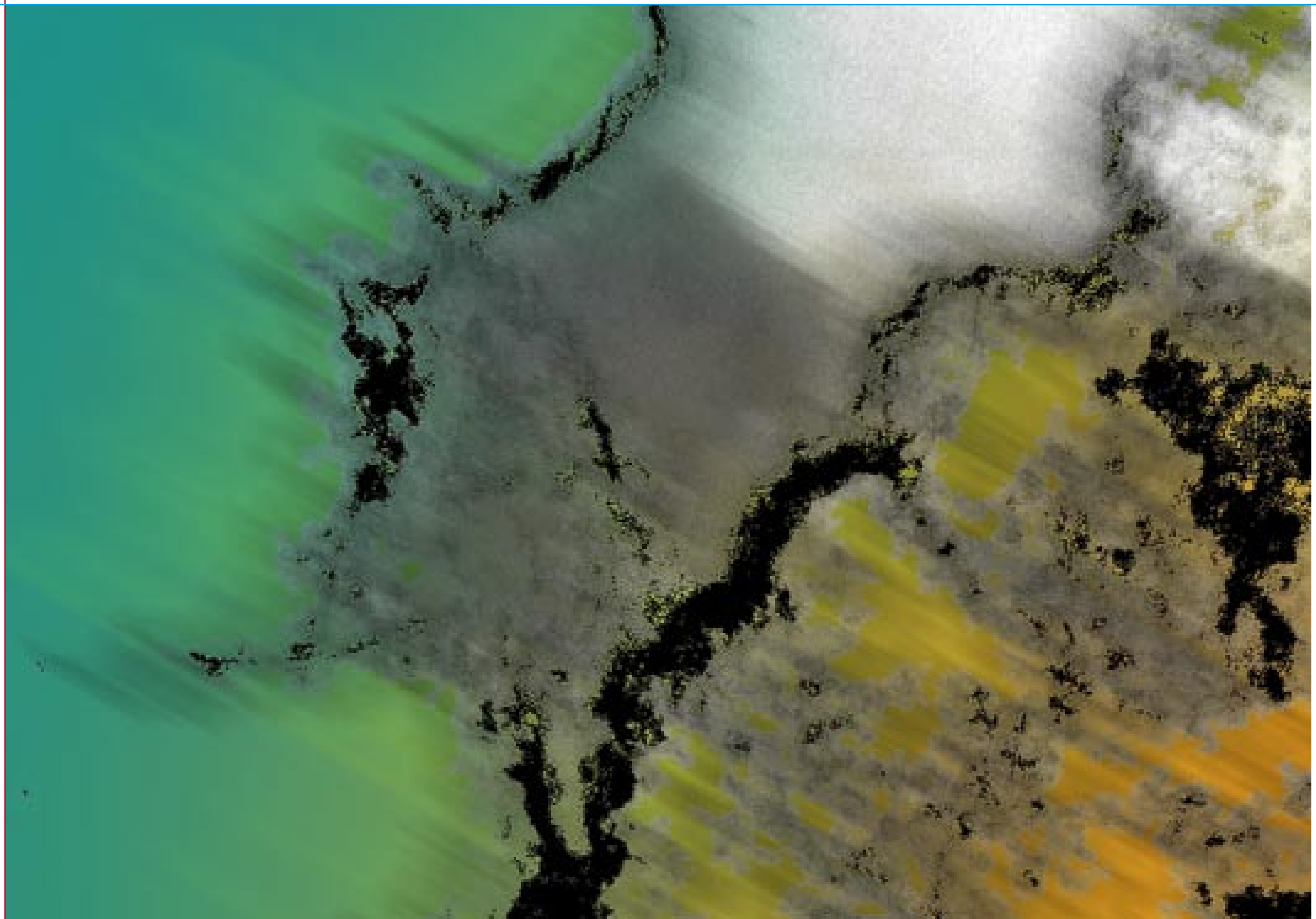
Not everything we did in 2003 was successful. Our attempts to participate in the EU's 6th Framework Programme failed. Neither the Integrated Project Proposal nor the Network of Excellence passed the test.
The Dutch invention of the TTI, the concept of the pre-competitive public/private research alliance, although recommended by the OECD, was found to be incompatible with the 6th Framework Programme rules. We will have to explore new routes together with the Ministry of Economic Affairs to explore the European funding programmes.

All in all, 2003 was, also keeping in mind the increasing number of patents, theses and scientific publications, a very productive year. Thanks to the dedication and combined efforts of researchers, DPI staff and management, a significant mark was set on the read to excellence and continuity for DPI.

George van Os, M.Sc.,
Managing Director,
Chairman Executive Board

Clauss:

"Success will be when others say they envy us and regret that they had not joined from the start."



c o n t e n t s

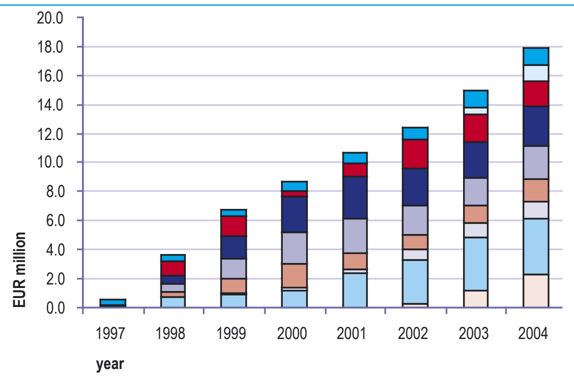
Annual Report 2003

The future blueprint for DPI

George van Os
Managing Director
Chairman DPI Executive Board

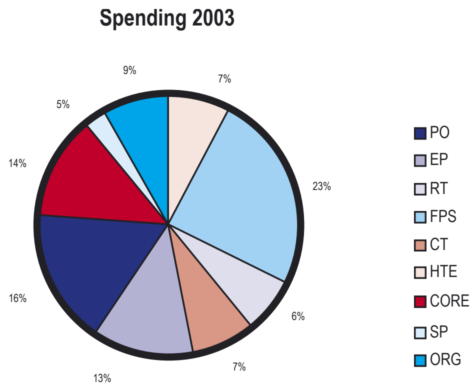
When DPI was launched In 1998, its mission was to become a leading European technology institute for polymer science and engineering, rooted in the Dutch knowledge infrastructure and focused on issues relevant to the polymer industry. It would be characterised by a chain-of-knowledge approach through integrating the know-how of academic disciplines. Today, six years later, DPI has grown into a sizeable, science-based network institute active in many areas of polymer technology, galvanising a broad range of academic science groups primarily within Dutch knowledge institutes.

Expenditure in 2003 amounted to some EUR 15.3 million, involving more than 220 researchers in primarily Dutch academia and Public Research Organisations (PROs), and equivalent to 140 full-time employees per year. On the industrial side the science-based network was linked to an industrial consortium of 21 international companies by means of six programme committees and numerous industrial contacts.



DPI growth

- PO Polyolefins
- EP Engineering polymers
- RT Rubber technology
- FPS Functional polymer systems
- CT Coating technology
- HTE High throughput experimentation
- CORE
- SP Special projects
- ORG Organisation



DPI Technology Area expenditure

Since the start-up in 1997 DPI has generated a continuous stream of publications and inventions. Today DPI 's library totals some 486 publications and 33 reported inventions which are being transferred into patents. Many scientists and engineers trained through DPI projects have taken up professional careers at one or other of our industrial partners.

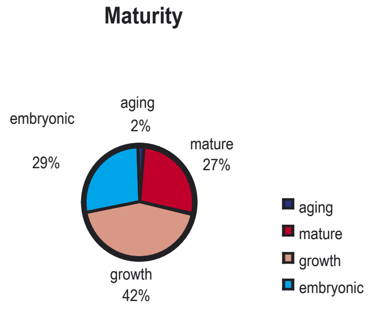
In order to make a blueprint for the future, DPI conducted a stakeholders survey and installed a task force to study the key strategic issues for the future. The stakeholders survey, carried out by Van Den Hooff International Consultancy, revealed that DPI is on the right track to becoming a real Leading Technology Institute. However, it also showed that we have to improve our organisation and operating procedures in order to be able to demonstrate real "excellence" with our academic partners and to demonstrate real " impact" to our industrial partners.

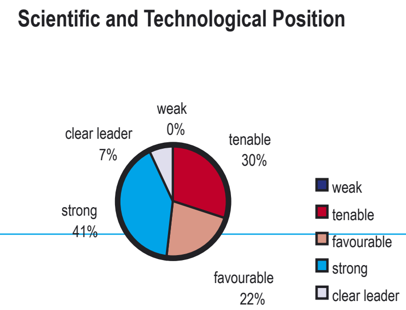
Lessons DPI Stakeholders Survey

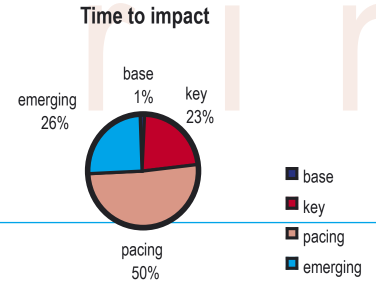
DPI as LTI is on the right track, but there is a need...

- *for clear strategic choices in the programme*
- *to improve project selection*
- *to involve more knowledge institutes, especially foreign ones*
- *to improve programme management*
- *to improve industry participation*
- *to improve communications to partners*
- *to demonstrate excellence and impact*

To assess the current status quo and to outline a future blueprint for DPI, the task force initiated a DPI portfolio analysis, to visualise the strengths, weaknesses and prospects of the DPI projects portfolio. The outcome of this exercise showed that, among other things, only half of DPI's current projects can be seen as "strong" or "clear leader" within the academic world.







Based on the outcome of the stakeholders survey and the portfolio analysis, the DPI taskforce came up with a number of recommendations, which should form the basis of DPI's business plan for the next five years. Within a two to three years at least 80% of DPI's programme should score "strong" or "clear leader". To enable this target to be achieved, the task force recommended reinforcement of DPI's programme management and the installation of a Science & Technology Reference Board. Although the majority of the programme will continue to focus on the need of the current industrial partners, DPI should enable incorporation of projects which potentially could create new economic activity, and adapt its mission for this purpose.

Summary and conclusions - implementation

- Modus operandi unchanged
- *Doubling of budget in next five years to EUR 30 million*

- Implement extended mission (as from 2004)
- *Broaden the basis of participating knowledge institutes*
- *More emphasis on incubation of new economic activities via core programme*

- Implement programme portfolio upgrade
- *80% In target area (2006)*
- *New projects acquisition by tendering (2004)*

- Reinforce programme management (2004)
- *Full-time programme management*
- *Install team of scientific area consultants*
- *Install Science & Technology reference board*

At the end of 2005 the present consortium agreement will expire after being active for two periods of four years since the start-up in 1998. By this time DPI should be able to demonstrate " excellence" and "impact" to its stakeholders in order to be able to convince them to continue their commitments for another four-year period.

The upgrading of the programme portfolio, the reinforcement of the organisation and the addition of a new dimension to DPI, in the form of the extension of the mission to include the creation new economic activity and involvement of SMEs, will form the basis for making DPI a sustainable initiative in the eyes of its stakeholders.

Summary of financial data 2003

Income (x EUR million)

Contributions from industrial partners	3.81	25%
Contributions from knowledge institutes	3.81	25%
Contributions from Ministry of Economic Affairs	7.64	50%
Subtotal Strategic Research Programme	15.3	100%
Contract Research	0.1	
TOTAL INCOME	**15.4**	

Expenditure (x EUR million)

By nature

Personnel costs		
Research related	11.41	75%
Non-research related	0.89	6%
Depreciation		
Research related	1.87	12%
Non-research related	0.03	0%
Other costs		
Research related	0.58	4%
Non-research related	0.44	3%
Contract research	0.1	0%
TOTAL EXPENDITURE	**15.3**	**100%**

By Programme Area

Organisation & support	1.39	9.1%
Strategic Projects	0.70	4.6%
Core Programme	2.17	14.3%
Polyolefins	2.50	16.4%
Engineering Plastics	1.92	12.6%
Rubber Technology	0.89	5.8%
Functional Polymer Systems	3.54	23.3%
Coating Technology	1.11	7.3%
High Throughput & CMR	1.00	6.6%
Contract Research	0.1	0%
TOTAL EXPENDITURE	**15.3**	**100%**

data 2003

2003 Key Performance Indicators

Number of industrial partners:

End 2002: 17
End 2003: 21

Number of partner knowledge institutions (universities etc.)

End 2002: 15
End 2003: 19

Industrial contribution (cash and in-kind) as % of total expenditure:

End 2002: 29%
End 2003: 25%

Number of patents filed by DPI

In 2002: 11
In 2003: 14

Number of patents licensed or transferred to industrial partners

In 2002: 5 (transferred applications)
In 2003: transfers on hold due to legal issues

Number of spin-off companies

In 2002: 0
In 2003: 0

Track record former DPI researchers up till 2003

Departed in total	133
Employed by partner knowledge institutes	49
Employed by non-partner knowledge institutes	6
Employed by industrial partner company	13
Employed by industrial non-partner company	7
Returned to native or foreign country	13
Retired	2
Unknown	43

Industrial follow-up

No details available due to confidentiality

European governmental funding (% of total funding)

In 2002: 0%
In 2003: 0% (two proposals submitted for 6th Framework Programme declined)

Participation of foreign knowledge institutions (% of total expenditure)

In 2002: 1%
In 2003: 2%

Research output

	2002	2003
Scientific publications:	104	128
PhD theses:	12	15

Overhead costs as % of total expenditure

In 2002: 7%
In 2003: 9%

Expenditure for knowledge transfer

In 2002: EUR 164,000
In 2003: EUR 225,000

Supervisory Board

Dr Ir H.G.J. de Wilt — Chairman
Prof. Dr E. Meijer, DSM Research — Vice Chairman
Prof.Dr M. Dröscher, Degussa AG
Dr Ir J.A. Pals, Philips Research till 2003
Dr H. van Houten, Philips Research from 2004
Ir A.C.G de Kok, Dow Benelux till 2003
Dr Ir M. Steijns, Dow Benelux from 2004
Drs N.P.J. Kuin, Océ-Technologies B.V.
Prof.Ir K.C.A.M. Luyben, Delft University of Technology
Prof.Dr R.A. van Santen, Eindhoven University of Technology
Prof.Dr W.C. Sinke, Energy research Centre of the Netherlands ECN
Ir J.H.J. Mengelers, TNO Industrial Technology till 2003
Dr Ir E.J. Sol, TNO Industrial Technology from 2004
Prof.Dr Ir H. Tijdeman, Twente University
Dr Ir J.A.J.M. Vincent, Sabic
Prof. Dr L. Böhm, Basell till 2003
Ir A. de Vries, Basell from 2004
Dr H.M. van Wechem, Shell
Prof. Dr D.A. Wiersma, Groningen University
Prof. Dr J.J. de Wit, Akzo Nobel

Executive Board

Ir G. van Os, Managing Director
Drs J.P. Bakker, Financial Director
Prof. Dr P.J. Lemstra, Scientific Director till 31.12.03
Prof. Dr M.A.J. Michels, Scientific Director from 01.04.04

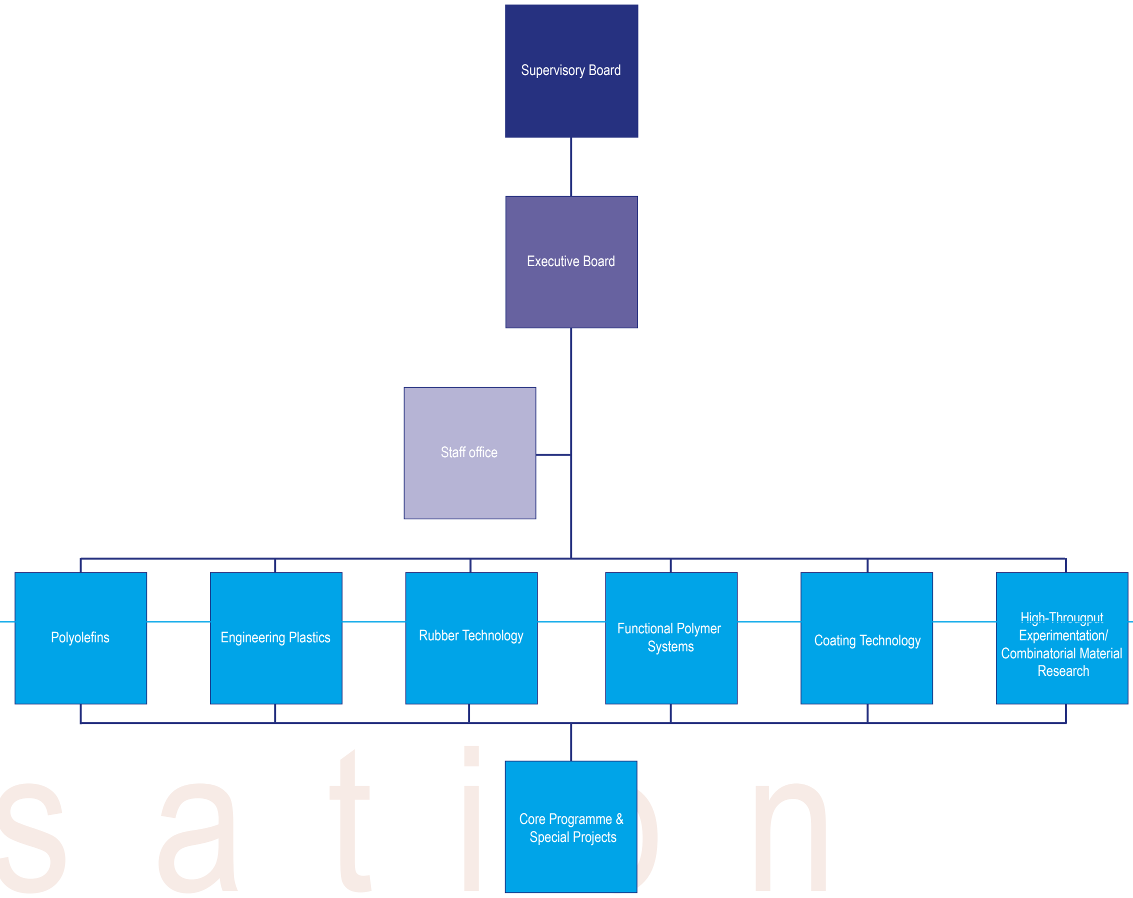
Programme Management Technology Areas

Dr B.C. Roest, Polyolefins
Ir R.P.A. van den Hof, Engineering Plastics
F. Thys, Coating Technology
Prof. Dr Ir J.W.M. Noordermeer, Rubber Technology
Prof. Dr M.A.J. Michels, Functional Polymer Systems
Prof. Dr U.S. Schubert, High-Throughput Experimentation
Prof. Dr P.J. Lemstra, Core till 2003

Staff organisation

Drs M.C.A. van Egmond, office manager
P.J.J. Kuppens AA, controller
S. Koenders, secretary
J.J.D. Tesser, communications manager
Ir S.K. de Vries, programme officer
Ir J.G.M. Nieuwkamp, patent attorney from 2004

DPI organisation



The advantages of the flexible DPI organisation

One plus one makes three. Tailor-made engineering polymers provide a good example of the advantages of the DPI project systems, involving several industrial partners rather than a one-to-one contact between a single university and a single industrial partner.

Van der Heide:

"I was very pleased both with the result itself and with the way the work was done."

Dr Evert van der Heide, senior researcher at Shell was the initiator of this project, dr Henri Grünbauer of Dow Chemical joined the project group later in the process and dr Reinoud Gaymans was the supervisor of the PhD-student at the University of Twente. They were among the partners in the project 'Hydrogen-bonding in block copolymers' which started in 1999 and completed with the PhD thesis of Martijn van der Schuur with Prof. Dr Jan Feijen as promotor in February 2004.

Foam

Van der Heide explains the starting points: "In 1997 Shell was interested in improving the raw materials and formulations used to produce flexible foam, in particular polyurethane. The structure of polyurethane for mattresses, car seats, shoes, clothes and packaging materials, largely determines their performance and production costs. We thought that by developing improved materials and formulations, we might be able to supply foam manufacturers with materials of the same quality for a lower price or with increased product performance at the same price. So we wanted to look into the fundamental properties of the materials and were looking for a university partner for this project. DPI was established at that time, so it seemed logical to set this project up within this framework. This would involve more industrial partners than Shell, but since this was pre-competitive research -making the material would require an additional step after the favourable properties of the polymeric materials had been established- we didn't mind."

interview

Original approach

Gaymans further clarifies the starting point: "We were already doing that kind of research into thermoplastic elastomers based on amides at that moment. A PhD student, Meike Niesten, was working on it. In 1999 Shell proposed studying polyurethane, a more complicated material; the thermal stability is limited and the working window is too small for a background study. More than 4000 papers were published on polyurethane in the ten years before we started. Our initial idea was that we, as relative newcomers in the field, would have a hard time to come up with new insights. We decided along with Shell to use a similar, thermally more stable amide polymer and we promised that we would try to translate the results of the amide polymers to the polyurethane system. We found that if the amide can be made to crystallise fast –by using relatively short, uniform segments– crystalline structures in polymers with a very high aspect ratio (ribbon structures) resulted. Both the modulus and the elasticity of the material had improved. We translated the results to the polyurethanes and the resulting polymer material met the expectations best." Van der Heide adds: "Their original approach resulted in unique properties and that really stood out among the many papers published in this field. I was very pleased both with the result itself and with the way the work was done. The PhD student did a very good job. That is the main reason for this success."

Synergy

Shell's motives for co-operating in DPI projects, however, are not entirely related to the end results of a specific project. Van der Heide puts it like this: "The first and most important reason for us participating is to establish and maintain contacts with excellent researchers outside our own company, including universities and other industries. Secondly, we aim to improve our own overall knowledge and the actual results of the project itself only come in third place. The disadvantage that more partners, even Shell's competitors in the same field, have the knowledge at the same time is counterbalanced by these advantages." The fact that more partners were involved was certainly of benefit to Twente University. Gaymans: "When Shell divested most of its polymer production activities and Evert van der Heide's responsibilities in the company changed, Henri Grünbauer of Dow Chemical joined us in a natural way."

Grünbauer: "As the industrial contact person on behalf of Dow Chemical I was involved in the project from the start. I realised that the technology developed in Twente could have a positive impact on the development of new products for Dow Chemical. So a more actively supporting role was a natural step to take." Gaymans adds: "If it had been a one-to-one contract with Shell alone, the project would almost certainly have been stopped. The synergy between industrial partners within DPI is one of the more attractive points for us."

Benefits

But surely there are more attractive points that are clear from the start? Surely you don't start a project on the basis that your major discussion partner will or can change his interest during the project. Gaymans answers affirmatively. "Compared to a one-partner project, we certainly see the benefits. The projects are guided by a users' group and as a researcher you can choose whom you listen to most. The other partners involved in the project, six in this particular case, in addition to Shell and Dow Chemical, are Océ, DSM, GE and formely Acordis. That is one reason why we like DPI projects, even when the financial circumstances are not as good as with projects that are subsidised via other channels, for example STW. Another reason for us to prefer DPI, is that considerably less paperwork is required to submit a proposal, compared to STW projects and certainly compared to EU projects. Not only does it take less work, you also hear what has been decided much more quickly."

At the end of the discussion Van der Heide, Gaymans and Grünbauer agree that the quality of the work done depends on the people that do it. Not only the PhD students and their supervisors but also the people who participate on behalf of the industrial partners. And in the case of this project it worked out fine. ●

Gaymans:

"The synergy between industrial partners within DPI is one of the more attractive points for us."

Grünbauer:

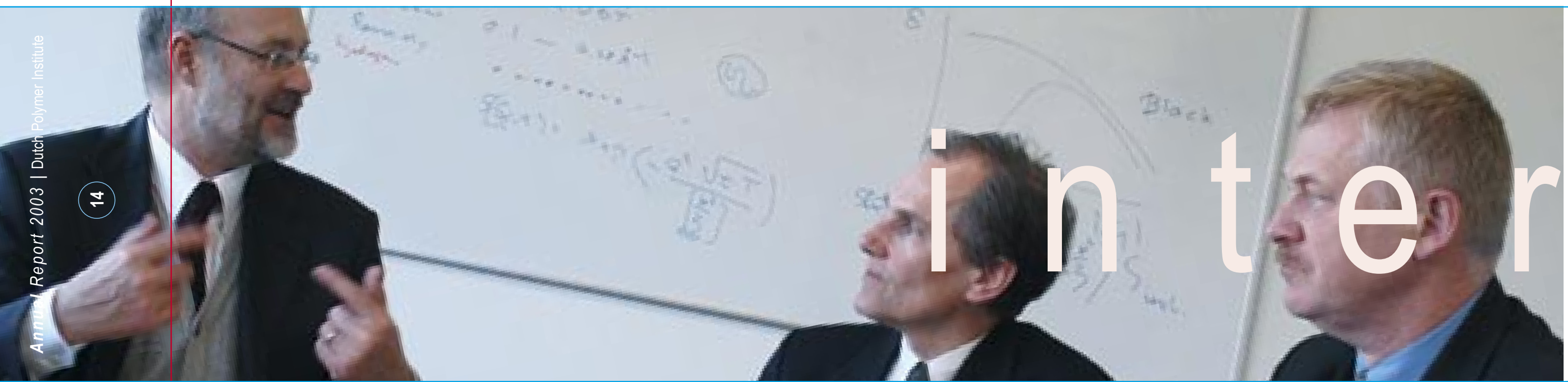
"... the technology developed in Twente could have a positive impact on our new products."

Networking gets more net working done

Research into functional polymer systems differs in a number of aspects from the work being done in the other cornerstones of polymer research in DPI. Not only are product quantities very small and material properties very sophisticated, it also took a bit longer to get the complete research programme going. But work is now in full-swing and inspiring results and satisfied partners are the order of the day.

Michels

"New cabinets are likely to change policies but in shaping public-private research, long-term thinking is needed …"



An interesting company gathered recently to discuss the proceedings, the ongoing business and the future of the Functional Polymer Systems cluster of DPI. Prof. dr Thijs Michels, in 2003 programme manager of the Functional Polymer cluster and currently DPI's Scientific Director, dr Germ Visser, project manager of DSM and prof. dr Michael Dröscher, responsible for innovation management at Degussa, which joined DPI only recently.

Aware

Politely, the two Dutchmen leave most of the opening words to the newcomer and Dröscher is only too happy to share his views with them. He first explains his or rather Degussa's reasons for joining DPI: "Degussa is what I would call a multi-merged company and now consists of 21 business units, about 17 of which have something to do with polymers in one way or another. We have a decentralised R&D organisation and were looking for a way to spend our R&D money effectively. Our present R&D efforts are mostly aimed at our own competencies but it is also vital to remain aware of what is going on in neighbouring fields of research. If we have to employ researchers in all those fields, we will never be able to afford it in the long run. As you will know, business unit managers always expect profits overnight from the money they have invested. So we were looking for a platform and I must say we are happy that we found exactly that with DPI."

"That is indeed how DPI works," confirms Michels, "Shell, an example I know very well, was involved in polymer research via DPI but when the company divested most of its polymer activities, they just moved their 'tickets ', but stayed on board just for that reason." "I always say that there are more brains outside the company than inside, so we 'hire' them to help us to keep abreast in the field. That was our motive at the start of DPI and it still is applicable. We are still very fond of DPI," Visser adds.

Europe

After this initial declaration of love Thijs Michels dares to ask his discussion partners about DPI's ambition to become Europe's leading polymer research institute. What are their views? "Well," Dröscher says "We came to you!", thereby qualifying both Degussa and DPI as top of the bill. He continues: "We were looking for a European Institute. You have an advantage over other European countries and with that I do not only mean that your borders are close, but also that it is easier to combine efforts in one field in a smaller country. We co-operate with many institutions in Germany, but not one of them is as widely oriented as DPI is. Co-operation surely is a natural habit in the Netherlands and I particularly like your open way of thinking. I do not know of another example of co-operation that has the same platform-like set-up that makes it easy for new partners to link in. Moreover, DPI covers the whole chain from fundamental research up to and including applications. So I think that DPI surely is one of the leading research institutes for polymer technology in Europe."

Manage output

DPI forms a link between industry and universities and Dröscher again emphasises one of the starting points. "If you have to set up a contract with a university every time you want to have a job done, it takes a lot of time. That has now been settled in one go." Michels observes that apart from that there is also the synergetic effect from other projects that helps every new project. "Don't forget that universities are traditionally 'input monitored'. Researchers write a project proposal, and once they get the funding, they do the work and in the end publish the results in the open literature. But for industry it is the output that counts, and we try to manage the output of the DPI projects. That is something universities are getting more and more accustomed to, but we are not yet successful in all areas."

Michels: "In the FPS cluster the programme was initially managed a bit differently from the other DPI clusters. Compared to other clusters it took two more years to get most of the programme going, but since then it has organically grown from bottom-up projects into a well-structured programme with five coherent themes. We are now entering the second round of proposals and I expect a good few new and interesting ideas." Visser adds: "I can underline that. We co-operate very successfully with the group of Dick Broer and Kees Bastiaansen of DPI and TU/e. The programme on optical applications has resulted in no less than seven patent actions in the second half year of 2003. Existing materials are manipulated for smart optics, structured coatings with improved optical properties for applications in, for instance, displays. It took a while to get people to trust one another. It is not only important to get the chemistry between the molecules in the reaction vessels going, but also the chemistry between project members. You can't expect changes overnight."

Long term

"That is true," says Michels, "And I only hope that the government keeps funding institutes like DPI for some time to come. New cabinets are likely to change policies. In shaping public-private research, however, long-term thinking is needed, an opportunity to complete the exercise and bring it to an end, as it were."

Dröscher: "In our companies as well, some of the managers expect too much in too short a time. In particular the business unit managers who are responsible for today's business and tomorrows profit tend to be a bit impatient. And what is also overlooked sometimes is that if a project does not yield the required result in the sense of a new product or technology, we still have learned something from it which can be used to our benefit in the future."

Michels puts it like this: "It is a delicate balance. We get funding from the companies and they expect results. But we still do fundamental research in DPI, though it is earmarked for future applications. It is obvious that we need good long-term planning scheme to manage that. It cannot be blue sky research." "We cannot do it just for fun," Visser adds.

"A few things also have to change inside our companies," observes Dröscher. "We are used to selling chemicals in bulk and are used to setting a price for the products per kilogram. We have to start thinking in solutions for customers. There are examples of Functional Polymers Systems where only milligrams do the job. Payment shouldn't be per unit of weight but for the value delivered. The amounts of materials required by our customers are sometimes made in a laboratory by highly qualified personnel in weeks."

Catalyst

Visser draws from his experience: "Looking back over the past few years and the successes we have achieved, there is one thing that can be improved upon. I think that the contacts between project group members can benefit if more of the meetings are held on the industrial premises. The university people should leave their ivory towers more. In a different environment, discussions are different and other ideas come up. Moreover, the PhD students can see how we work in industry and gaining greater understanding will give them a more solid basis for deciding which job they should go for in the future."

All three discussion partners are very positive about the future of DPI. The new open call for proposals holds the promise of good, new ideas that can lead to innovative products in the end. Although the field is not particularly suited for start-up companies –the reason being that most ideas are already being picked up by the large companies- Dröscher, -while pondering over new business creation- comes up with the concept of a market of ideas presented by the DPI staff where both large companies and parties interested in starting up a new business can take their pick. If ideas are not picked up by partners in a project, they get published and that's it. But it is a pity when good ideas don't come into full bloom because the company for which the work was done cannot or does not want to incorporate it in its business. DPI as a whole is in an excellent position to recognise promising innovative ideas. Its role there is, as in so many other areas that of a catalyst. ●

Dröscher

"… co-operations with many research institutions in Germany, but not one of them is as widely oriented as DPI is …"

Visser:

"… contacts between project group members can benefit if more of the meetings are held on the industrial premises …"

A new way of thinking

The High Throughput Experimentation cluster of DPI aims at more reliable and more reproducible experiments, achieved in a shorter time with less energy spent than before. A paradigm shift is required.

Schneller:

"We can handle 100 even 1000 new formulations a day, so we have a bigger chance of success."

Schubert:

"… we set clear goals, we can move faster."

No less than five people assembled to discuss the results achieved so far and the future outlook for the High Throughput Experimentation cluster of DPI. Prof. Dr Ulrich S. Schubert, programme manager of the cluster, Dr Arnold Schneller and Dr Joachim Clauss, both members of Core Technology of Ticona, Germany, Prof. Dr Matthias Rehahn, head of the Deutsches Kunststoff-Institut (German Institute for Polymers, DKI) and Dr Ing. Martin Moneke also of that institute. Ticona is one of the industrial partners and DKI is a knowledge partner in this cluster.

Not product-oriented

Schubert starts the discussion with an explanation why this cluster is different from the others: "First of all, the cluster has only been in existence for two years now, a few years less than the others. But more important is the fact that we are primarily not focussed on specific products or product groups, as opposed to the other DPI clusters. Our major activity is to explore a new approach to materials research that is more effective both in number of results and in understanding them. Our industrial partners are proportionally divided between big chemical companies, which can use this generic approach in their product development, and small technology companies with specific knowledge of the equipment and software we need, which can market the developed methodology. For DPI that is a very unusual mix."

Schneller explains this new way of doing research by referring to the traditional situation. "An engineering resins supplier such as Ticona looks for new formulations and new additives to make products with better properties, for instance, polymers with better heat and wear resistance, or better mechanical properties. Our resins typically contain many different additives, each with a specific function . During development each additive is investigated in a sequential way, step by step. Most of that is still black magic. A researcher can check up to five formulations in a day. But since additives influence each other there are many different combinations to try, even when you know beforehand which ten additives would help to improve the product. In the new High Throughput Experimentation approach we can handle 100 or even 1000 new formulations a day, so we have a bigger chance of success, a hit as we call it. Investments for this infrastructure are so heavy that companies cannot afford it for their own relatively small portfolio of products. But a number of them together can. That is where DPI comes in."

Schubert continues: "Edison worked that traditional way in developing and optimising the light bulb materials, try a thousand combinations, find a hit and optimise that. But in polymer research with its infinite number of combinations we had to find a cleverer way. Not only for financial reasons or speeding up the research. We need a new way of thinking, a new approach. If you know more, you have the opportunity to learn more, to do the right experiments, to gain knowledge. One company has specific interests, knows a lot about its materials and usually keeps its specialist knowledge to itself. Combining the knowledge will help us to understand more about the processes and the influences of the additives and the catalysts. So in the end it will give us a better product." Clauss puts it like this: "We can now proceed in a more evolutionary way, we try all of the combinations and make the database complete. In that way we have a better chance for a hit." Schneller adds: "And if you have tried all of the combinations, and also know which will not result in hits, you know that you will not miss anything. It is useful to know that our competitors will not find new materials there either. In that way it is also a defensive approach."

Hi-tech

How is done then, this twentyfold and more increase in the number of experiments per day? At the Eindhoven University of Technology the traditional chemistry laboratory with lots of glassware, burners and ovens is being transformed into a real high-tech environment where chemists, physicists and computer scientists work side by side. Robots that can be programmed to do several dozen experiments in parallel dominate the scene. With crates of the same kind of equipment waiting to be unpacked. The DKI laboratories in Darmstadt look the same, but on an even larger scale. It is clear that such a revolution in methodology means also an expensive change in equipment which a single company usually cannot afford on its own, not even if it is a very large company.

Schubert: "If you ask ten PhD students around the world to do the same experiment, you will get ten different results, that you have to explain. When we do such experiments with our robots controlled by computers we get reproducible, and more reliable results. We have already demonstrated that in the last two years. We can do 40 polymerisations in parallel, get comparable results and deduce information about the use of catalysts or additives from that. It is too early yet to have final breakthrough examples of successful new materials, but we will get them. If you look at the complete circle of design, synthesis, formulation planning, analysis and testing, right to product development, we have now shown that parts of that circle are running reliably and reproducibly. We will have to use the time the robots save us, for thinking, planning, modelling etc."

Paradigm shift

Rehahn says. "It is indeed not only synthesising new materials that has our attention, we need to analyse the results too. There is a slogan in the field: only synthesise in a day what you can analyse in a day. So new methods for a fast first analysis are needed, one that you need only a little bit of material for. That is another reason to look for an optimum overlap in the activities of partnering companies."

Schubert adds: "And after that, when we have assembled all the knowledge we need to make it accessible and exploitable. Just storing data in a computer will not help in these multiple parameter spaces. Techniques such as modelling, data handling and data mining are part of our projects as well."

Schnelller: "It really is a paradigm shift. It is not only we, in industry who have to think differently, it must become part of our education system as well."
Rehahn explains how: "Our group leaders in DKI are teachers at the university of Darmstadt, so there is a direct line to the educational system. The same holds for the university here in Eindhoven. After a while we will have educated students who are familiar with this new way of thinking and companies will no longer need to train their employees themselves in this respect. We think that this is a very efficient way to get our results directly to other companies. We will need these educated scientists in industry to safeguard innovation in Europe. We need a clever way to do such work in Europe, otherwise industry might as well go to Bangalore or Shanghai right away. We will need a technological advance and we can get that by the right planning, the right machining, the accumulated knowledge and a short line to the educational system."

Success

Schubert: "Students are attracted by the high-tech environment we are creating. Our department already has more students than before we adopted this new approach, even some attracted specifically by what we are doing here. We know we are doing the right thing because you see other institutions working on the same kind of things. In the United States, Japan, Singapore, you see them coming up. But here in DPI we are faster, more inclined to share the information we gather and certainly more focussed on education. So we will have an influence, certainly in Europe where DPI is the only example of such an institute."

Schneller knows one reason for the speed in DPI: "Procedures in DPI are very fast, not at all bureaucratic." Schubert adds: "When we set clear goals, we can move faster. We are more flexible than other institutions that are governed by academic subsidy rules. A company buys a ticket and is involved in the programme set-up as well as steering. The government does not interfere in the daily business and programmes. Decision lines are very short. New initiatives will fly fast."

Schubert ends the discussion with: "For us, success at the end of the day, four years from now when the present projects come to an end, comes when industry takes over the methodology, and the equipment developed together is actually bought and used. Success can take many forms: new equipment, a new infrastructure, new software structure, knowledge, a kind of material informatics, new processes, new or improved products. One thing is certain: we will do our experiments in another way. With robots and we will use the accumulated knowledge and computers to design our experiments." Clauss formulates it in one sentence: "Success will be when others say they envy us and regret that they had not joined from the start." ●

Rehahn:

"DPI's link with education is a very efficient way to get results directly to other companies."

Polyolefins

The year 2003 was for a large part dedicated to executing the projects already in progress, filing patents, publishing, lecturing at congresses, etc. The internal communications were fairly optimal, culminating in three polyolefin days filled with high-class presentations. Regular meetings of a more organisational character were arranged by the programme committee, the steering team of industrial representatives and the scientific support committee handled the scientific coordination and ensured close relationships between the main working areas.

This year we introduced two projects of a fundamental physics-oriented character. The behaviour of molten PP under stress is being studied using X-ray diffraction and modelling, a result of our general policy to justify projects only if a clear goal or set of goals is defined with pertinence to polyolefin.

A DPI initiative led to some self-evaluation about our future policies. The polyolefin partners are in general quite confident that their programme is sound and that the right subjects are chosen. The immobilisation projects were the result of a successful call made in 2001 and the programme committee is now preparing new calls. The apparent maturity of some projects is an inevitable consequence of the vast polyolefin world. From a scientific point of view, our polyolefin programme was judged as outstanding by an independent committee in 2003. In addition to this, the scientific support committee will report their ideas about the best scientific way to proceed.

The actual economic situation made it difficult to attract new partners. Much discussion was undertaken with the University of Twente (UT) to safeguard the strong position of DPI and the university.

Partners

Industry:

Akzo-Nobel, Basell, DSM, Dow, SABIC and Shell. In January 2004 NPC (Iran) and Borealis (Denmark) decided to become a partner.

Academia:

The COP centre (University of Groningen), TU/e and the Universities of Twente, Utrecht, Amsterdam (UVA), Nijmegen, East Anglia, Naples and Hamburg.

Budget

Total costs over 2003 were EUR 2.5 million (budget EUR 2.5 million). The number of PhD students and PostDocs was 30. Four professors or UHD's are paid for part-time coordination activities in the subclusters.

Communications

Three polyolefin days were organised during which there were more than 30 presentations in all. Some of these presentations were overview lectures by the cluster coordinators. New were the quarterly reports, edited by the cluster coordinators and providing an opportunity to gain a succinct picture of our progress. Both the programme committee and the SSC had three meetings. Presentations were made on virtually every major important seminar/congress dedicated to polyolefins.

Highlights

- Using quantum-chemical methods, Blok et al. (University of Nijmegen) elucidated the catalytic cycle for ethene trimerisation to 1-hexene with the titanium catalyst system developed by DPI researchers at the University of Groningen. The interest in this catalyst was reflected in the fact that recently French and Canadian researchers independently published quantum-chemical studies on the same system.
- The University of Naples started a block co-polymerisation project.
- Research on the melting/crystallisation behaviour of mixtures of polyolefins has led to interesting "shrink-free" concepts.
- The riser-downer project at UT is finished. An integral model containing kinetics, absorption of monomer and fluidisation in the slugging mode has been developed.
- A new project, modelling gas-phase reactors, will be based on radioactive measurements in cooperation with US scientists (UT).
- A project was executed by the TU/e (Koning), which led to a successful homogeneous mixing of carbon nanotubes in polyethylene. The results will be patented together with work on other thermoplastics.
- Arjen Bogaerds won the DPI Golden Thesis Award with a thesis on a polyolefin subject.
- Dr Sanjay Rastogi has the opportunity to work for a year at the Max Planck Institute in Mainz.

Projects/ targets

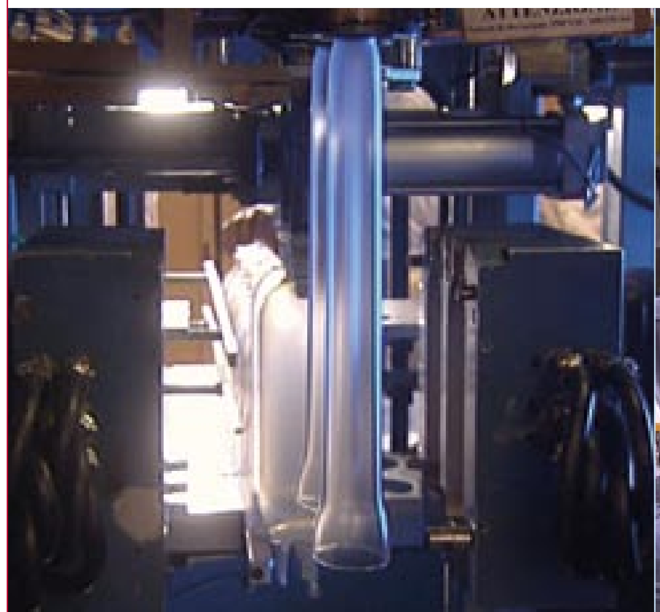
The TA comprises four thematic clusters with the financial borders indicated below. The borders are not yet completely realized in 2003, but form the lead for future projects:

1. Catalysis, chemistry
45% (co-ordinators: Chadwick/Hessen)
2. Process engineering
25% (Weickert)
3. Molecular studies, crystallinity, morphology
20% (Rastogi)
4. New themes
10 %

We have formulated targets/expectations for ourselves for the thematic clusters, as stated below.

1a: Catalysis, chemistry (Co-ordinator: Prof.Dr. B. Hessen)

The objective of this cluster is to understand the new ways of homogeneous catalytic behaviour. Model systems on the base of transition metals and late transition metals are synthesized and checked in their reactive behaviour with olefins or maybe non-olefins. Also the cocatalyst (MAO or derivatives) is studied, already leading to highly interesting results



Scientific papers

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Patents

Up to now seven patent applications have been filed on polyolefin results.

1b. Catalysis, chemistry and immobilization (Co-ordinator: Dr. J. Chadwick)

This is also a catalytic theme, but dedicated strongly to the heterogeneous Ziegler-Natta catalysis. The catalysts are sophisticated mixtures of supports ($MgCl_2$), titaniumchlorides and "donors" (e.g. silanes) leading to exceptional stereoselectivities. Main objective is to find out the precise locations and environmental demands of the active species. The support science is largely empirical and not subject to well-founded chemical theories. We want to change this.

2. Process engineering (Co-ordinator: Prof. G. Weickert)

Process engineering is needed to predict the performance of new catalyst systems in large production units. We have to build up a predictive scheme qua kinetics and morphology. The development of new unit operations was interesting, mostly directed at new reactor concepts.The development of models to predict behaviour of fluids, fines formations, etc. to assist in the engineering or operation of polyolefin plants.

3. Molecular studies, crystallinity, etc. (Co-ordinator: Dr. S. Rastogi)

Studying the microstructure of polyolefins and based thereon looking for processing and product improvements. Important is that the influence of polymerization conditions on micro structures is in some cases very profound.

4. New themes

To stimulate new ways of thinking in the polyolefin world, 10% of the budget will be reserved for striking new subjects. ●

Case

Polymer drawing facility

Polymer molecules possess inherent high stiffness. The random orientation and the coiling of these molecules, however, enormously reduce the modulus of the macrostructure. By orienting the polymer molecules in one direction, the mechanical properties of the macrostructure can be improved, allowing these polymers to compete with a wide range of engineering materials. The recent acquisition of a polymer drawing facility at TU/e allows the enhancement of the mechanical properties of fibres and tapes by solid state deformation.
The first stage in the process is the extrusion of a polymer tape, by one of a dedicated single screw extruder. The precursor tape is then stretched by drawing in a hot oven. The tape can then be pulled through a subsequent hot oven, in which it can be further drawn, and the stretched tape can then be collected by a purpose-built filament winding equipment. This two stage drawing process allows the creation of polymer tapes with excellent mechanical properties because of the alignment of the polymer molecules in the drawing direction of the tape. Current research focuses on a range of polymers including polypropylene, polyethylene, polyamide and polyethylene terephthalate, and the use of existing characterisation techniques allows immediate tape characterisation during continuous tape production.

Engineering Plastics

Engineering plastics are more than just the polymer(s) of which they are primarily composed. The aim is for engineering plastics to provide the solution that fulfils all requirements with respect to mechanical and thermal endurance over the expected lifetime of the parts made of the engineering plastics at optimal cost/performance level. The main characteristic of engineering plastics is their role in engineering applications where they mainly fulfil a mechanical construction function. They also possess specific advantages in terms of monolithic forming processes (combining several functions in one part), they are lightweight, have optical characteristics, are coloured, provide electro-magnetic shielding, are flame retardant, have low friction and wear, etc. In most cases these requirements can only be met by a combination of polymer(s) and additives, often reinforced by minerals, natural or synthetic fibres (glass, carbon or polymeric) and nanoclay, carbon nanotubes or other nano-particles.

Innovation in the area of engineering plastics is to be found in all components of the applications, from monomers to final disposal. The projects within DPI-EP are grouped accordingly:

- New polymers, new chemistry towards better defined molecular structures, lower-cost production routes or sustainable systems;
- New routes for reinforcements, new application areas for nano-composites;
- New applications through better processing; miniaturising, dimensional precision;
- Better lifetime prediction and routes to longer service life.

Solutions create far more added value when new technologies can be used in application areas not accessible until now, thus creating new markets. The challenge for the DPI projects therefore lies in developing new technologies, based on scientific principles, in polymer chemistry, advanced composites and fibres, processing and stabilisation and to select those projects that provide the best possible chance for future use in industry.

Partners

Industry:

Agrotechnology and Food Innovations BV, (A&F former ATO/DLO), Dow, DSM, GE Plastics, Océ Technologies, Shell, Teijin, TNO.

Academia:

TU-Eindhoven, TU-Delft, Twente University, Rijks Universiteit Groningen, TNO Industrial Technology, Agrotechnology and Food Innovations bv, Queen Mary and Westfield College London and Stellenbosch University South Africa.

Budget

Total costs over 2003 were EUR 1.5 million (budget EUR 1.9 million), 24 researchers are involved in EP related projects.

Communications

The programme committee met four times, with project review symposia organised in May and September. In May the emphasis was on polymer physics related projects. In order to strengthen the discipline integration, related projects from the coatings area and the core area (polymer structure vs. performance) were incorporated in the presentations. In September the emphasis was on polymer chemistry related projects.

Numerous bilateral contacts between researchers and industrial contacts took place, both on an incidental basis and in a more organised way in the presentation of results as in project meetings in Twente and in Delft.

Highlights 2003

- Three projects were completed successfully:
  (i) A post-doctoral position on the project called "Interphase formation during reactive blending of PA-12 and end-functionalised polystyrene using multiflux mixers",
  (ii) PhD entitled "Deformation and fracture of polymer systems",
  (iii) PhD entitled "Impact resistant vinyl polymers";
- Focus shifted slightly in view of the industrial interest expressed by the new members Teijin and A&F. New projects were defined on fibre-rubber adhesion, friction and wear of fibre-filled systems, nano-particle filled fibres and the development of sustainable monomers.
- EP symposia were held in 2003 on polymer physics (May) and polymer chemistry (September).

Projects/targets

Polymer chemistry and modification

In radical polymerisation good results were reached with both ARTP and RAFT polymerisation of copolymers from acrylates and octane or allylbutyl rubber. These techniques enable the synthesis of specially structured copolymers such as vesicle type morphologies or brush-type polymers that self-organise in rod-shaped structures through controlled alternating hydrophilic and hydrophobic side-chains in the brush. The project on impact resistant vinyl polymers was completed end 2003.
In the area of condensation polymerisation a number of different leads are followed towards the synthesis and characterisation of polymers with improved properties and processability.
In Twente the emphasis is on incorporation of mono-disperse, fast crystallisable blocks in polyesters, polyethers or polyurethanes. Major benefits are seen in improved solvent resistance, improved modulus, dimensional stability at elevated temperatures, fast processing through very fast crystallisation and good flow properties.

It also opens new application areas, e.g. membranes (breathable films) for which co-operation exists with the Membrane Technology Group of Twente University.
In Eindhoven several leads are pursued. Analogous to the efforts in Twente, improved polyester properties are sought for in synthesising specially structured copolymers. Here the route is to selectively incorporate (macro-)diols or diacids in the amorphous phase of PBT through solid-state co-polymerisation. Another solid-state polymerisation project aims to create very high molecular weight polyesters (PET, through mol-coupling), which should provide strongly improved fibre properties.
Another important issue for polyesters is to find fast synthesis routes based on terephthalic acid as one of the monomers, which need the exploration of novel catalyst systems. A new project in this area was started in 2003. For polycarbonate two projects aim to explore phosgene free synthesis routes with carbon dioxide as one of the monomers. For polyamide-6 a project aims for a shorter synthesis route. The major issue here is to find the right catalyst in order to reach economically viable reaction rates. Based on the catalysis expertise in Groningen a new project was approved on ring-opening polymerisation of propylene-oxide, an interesting area for the polyurethane business.

Processing for Properties; structure and performance

A project on deformation and fracture of polymer systems, in which the mechanism of toughening of semi crystalline polymers by both hard and soft particles was studied and modelled, was completed in 2003.
Another project in this field deals with the influence of molecular structure and morphology on tribological properties (wear and friction). Based on particular industrial interest in this area, two new projects were defined and approved in order to get a better understanding of governing load conditions and bulk-mechanical properties determining the wear and friction of moving devices. The industry interest stems from the realisation that more and more remotely controlled devices will enter our world (e.g. "drive by wire" in cars) for which miniature and lightweight actuators are needed. Plastics provide good solutions when properly engineered on life expectancy, mechanical performance and low cost.

Stabilisation

The planned study into the mechanism of (chemical) thermal and UV degradation of polyamides and polycarbonate will start in 2004 with the nomination of the (visiting) professor heading this research.
Two projects in this area, both situated in Delft, deal with physical ageing. By using fluorescent probes and positron annihilation lifetime spectroscopy the changes in local structure, free volume and dynamics of the amorphous phase is studied in order to understand the mechanical behaviour in the glassy state in relation to the age of the sample. In addition the changing free volume governs the mobility of antioxidants and other small molecules like radicals that are involved with chemical and environmental stability.

Advanced Reinforced Thermoplastics and synthetic fibres

Three projects (Delft) deal with the exploration of novel concepts in the field of "classical" composites, i.e. glass fibre-reinforced thermoplastics. One project deals with the topic of short fibre reinforced polymer blends, were the disperse component of the blend acts to bind the short glass fibres together. The interpretation and understanding of the mechanical bulk-properties involves considering the processing time (spreading and wetting) and allows modelling of the mechanical properties taking the load- and deformation mechanisms into account. Compression strength - or rather the lack of it - is a known problem in composite applications. Improvement is sought by using colloidal (nano-clay) reinforced polymer matrices. The latter project is therefore on the boundary between microcomposites and nanocomposites. These topics require a good understanding of the mechanisms occurring during creep and compressive loading of complex composites systems, the third project relates to this aspect, also providing concepts for optimisation of the properties. A project in Twente deals with fibre-matrix adhesion issues for aramid fibre reinforced rubbers, an important issue for improved performance of e.g. conveyor belts.
Four more projects at TU Eindhoven and at Queen Mary and Westfield College London deal with studying carbon nano-tube composites. Major issue here is the dispersion of nanotubes into a polymer matrix, which proves particularly difficult with more a-polar polymers like PP. First successes were reported for polyamide and polystyrene (through emulsion polymerisation). A particular difficult but challenging project aims for self-assembling, three-phase structures towards nano-foam materials. Finally a project was approved for developing nano-particle reinforced synthetic fibres, which in their application can be part of a micro-scale composite. ●

Single wall Carbon Nanotubes

Single Wall Carbon Nanotubes (SWNTs), having a diameter of 1-3 nm and a length of 1-2 μ m, are easily dispersed in an aqueous solution of the surfactant sodiumsodecylsulphate after a gentle ultrasonication treatment. This stable dispersion of predominantly individual SWNTs is subsequently mixed with a latex of a high molar mass polystyrene (PS). *After freeze-drying (Picture 1)* a powder mixture of PS and 0.3 wt% SWNT is compression molded into a thin film, which is submitted to a *four-point electrical resistivity measurement (Picture 2)*. Whereas an unfilled PS film exhibits an electrical resistivity of 1014 Ω.cm, the nanocomposite containing merely 0.3 wt% SWNT has an electrical resistivity of 102 – 103 Ω.cm. For obtaining an antistatic character an electrical resistivity of 106 – 107 Ω.cm is already sufficient. With a resistivity of only102 – 103 Ω.cm, applications in plastic transistors come within reach. This simple concept is widely applicable. For a polymethylmethacrylate and a polyethylene matrix very promising results were obtained as well.

Output 2003

Thesis

A.J.P. van Zyl, Synthesis, characterization and testing of nano-structured particles for effective impact modification of glassy amorphous polymers, PhD thesis, University of Stellenbosch, December 2003

I.A. van Casteren, Control of microstructures to induce dutility in brittle amorphous polymers, Eindhoven University 2003

Dommelen, J.A.W. van, Micromechanics of particle-modified semicrystalline polymers, Ph.D thesis, Eindhoven, April 2003

Schrauwen B.A.G. , Deformation and failure of semi-crystalline polymer systems: influence of micro and molecular structure.

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Patents

Three patent applications were filed in 2003, concerning mono-disperse blocks in PUR, RAFT polymerisation in heterogeneous media and dispersion of carbon

Rubber Technology

In 2003 the Rubber Technology Area was fully operational for the first year, with complete project-coverage of the budget and all projects staffed with personnel. Most of these projects had started at the end of 2002; just two projects dated back to 2000: the year, which witnessed the first involvement of DPI in Rubber Technology. Publications started to appear in 2003 and the first three theses are due in 2004, for the two projects started in 2000. The Technology Area is in full swing.

Partners

Industry:

The industrial partners in 2003 were Akzo Nobel, DSM Research, Kraton Polymers, TNO Industrial Technology and Océ Technologies. Six potential candidate rubber companies were approached in 2003 for participation in DPI. None finally decided to join for a variety of reasons.

Academia:

Three institutes participated in the programme: TUD, TU/e and UT.

Budget

Total costs over 2003 were EUR 0.9 million (budget EUR 1 million). The number of FTEs allocated at the end of 2003 was 11.7 FTEs.

Communications

The Programme Committee of this TA met three times in 2003: to monitor progress and decide upon new projects, although on the short term no new projects can be accommodated anymore due to the fact, that the total budget is consumed within the present project portfolio.

The objective to hold at least three progress meetings for all running projects with representatives of the partner companies involved, was in the majority of cases achieved. Informal contacts between academia and industry take place more frequently, as need arises. Contacts with the outside scientific world are taking off, the writing and submittal of papers for scientific journals was more the objective for the year 2003, as a preparation for the firsts theses to appear on projects #236 and #252 in the year 2004.

Highlights of 2003

- A strategy of focal points of interest has been defined, on basis of which the various projects have been selected and are running.
- The TA Rubber Technology is presently fully staffed.
- Four papers in scientific journals have appeared in 2003, which originated in the first two projects #236 and #252 within this TA.
- Four presentations/posters were held at renowned conferences.
- The first patent application was filed, originating from project #236.

Projects

Structure - property relationships of Vulcanized Rubbers (project #317; 1 fte)

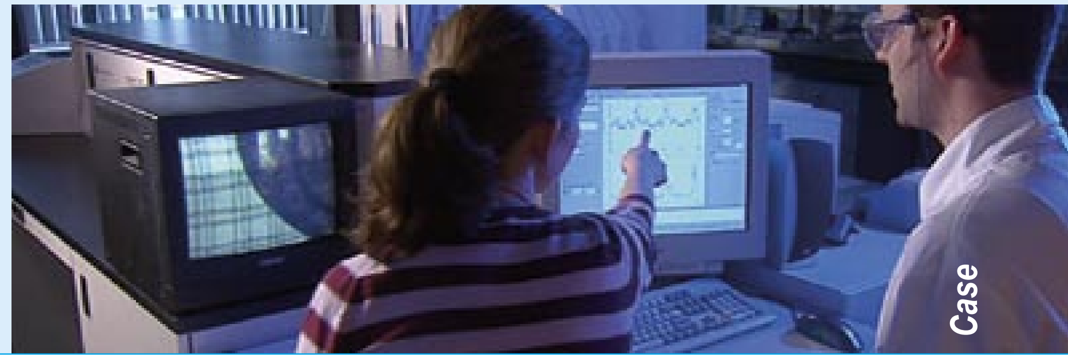
Objectives. The aim of this know-how area is the selection and development of vulcanized rubbers for specific application areas. So far one project has been lodged within this area, related to the specific requirements for toner-bands within Océ copying machines: to find or develop vulcanized rubbers with a proper balance between tear/fatigue properties and tackiness, incurred under prolonged exposure to ozone in the copier.

MALDI-experiment

Living anionic polymerization is used to make well-defined polymers, meaning they have a predictable chain length and a narrow molecular weight distribution. After consumption of the complete amount of monomer present in the polymerization system, all polymer chains remain active because they still bear a negative charge at the chain end. These anions are very reactive towards a variety of electrophiles, but most of these reactions suffer from unwanted side reactions. Our objective is to attach a certain functionality to the chain ends in a controlled and quantitative fashion.

Characterization of the products of these reactions is not easy, since only a minor change at the chain end is being made, while the entire long polymer chain remains unchanged. So, the product differs only slightly from the starting material.

Using MALDI-ToF-MS (Matrix Assisted Laser Desorption Ionization Time of Flight Mass Spectrometry) we are able to determine the molecular weight and the molecular weight distribution of the polymers very precisely. Comparison of these results with the theoretical masses of starting materials and products, gives us insight in the endgroups of the polymer chains, leading to a better understanding of the experiments we do.



Relevance. The specific problems involved in the characterization of surfaces of vulcanized rubber, in particular of the tackiness of rubber, represent a major challenge. Achievements within this project may have a broader bearing into a lot of other vulcanized rubber applications, including tires.

Deliverables. Project #317 has started in February 2003. The project-plan has been agreed upon in 2003 and first exploratory experiments have been done. It will be further pursued in 2004, to deliver first tangible results.

Chemistry of Vulcanized Rubber (projects 356 and 357; 3 fte's):

Objectives. The objectives of this know-how area are to advance vulcanization chemistry of specific rubbers for an improvement in overall properties.

Relevance. The rubber world is continuously searching for improvements in this combination of properties. Sulfur-like dynamic properties combined with superior ageing characteristics would provide a world of new possibilities for non-tire rubbers like EPDM: to widen the application range of these rubbers in the field of dynamic applications, and for tire rubbers: to expand the life-time of tire sidewalls in truck-tires.

Deliverables. Both projects #356 and #357 were started at the end of 2002. #356 deals with blending of EPDM/NR/BR for tire sidewalls. Focus for 2003 and 2004 is on obtaining a good morphology and a proper vulcanization-balance between the various phases. By chemically modifying the EPDM-phase a significantly improved blend morphology and also co-vulcanization was achieved. A patent application is in preparation, based on the first outcome of this project.#357 deals with the development of sulfur-containing co-agents for peroxide vulcanization of e.g. EPDM. A large series of potential compounds have been designed and are in the process of being synthesized. Some of them already show promising properties over common commercial co-agents. In 2004 they will be tested in actual rubber compounds for their enhancement of dynamic properties..

Ageing of vulcanized rubbers (project 412; 1.2 fte):

Objectives. For non-black reinforced rubbers UV-stability is a major problem, as nearly all common stabilizers are consumed during the vulcanization stage. We want to give this "old" problem another look, based on recently gained insights in stabilizer technology.

Relevance. Ageing performance is one of the first characteristics of all rubber applications. When a move is made away from carbon black as reinforcing agent, a multitude of aging problems do surface, impeding implementation. Aging aspects are implicitly included in nearly all projects within the present TA.

Deliverables. Emphasis will be laid on improving the UV stability of light-colored rubbers, in particular of EPDM-rubber. The project has started in April 2003: a project plan has been finalized, and the first preliminary results have appeared in 2003. It is still too early to judge, whether improvement can be achieved with present stabilizer technology, or that new routes need to be explored.

Structure-Property relationships for Thermoplastic Rubbers (project 252; 2 fte's):

Objectives. This know-how area is focused on morphology-rheology-properties of thermoplastic rubbers, with the eventual objective to find routes to improve their properties. It deals with a morphology-rheology-properties comparison between two classes of materials: SEBS/PP/Oil blends and

Thermoplastic Vulcanizates of EPDM/PP/Oil, which are completely different in morphology, but very similar and competing in processing and properties. The objective is to develop a morphological understanding of the similarities and differences between both systems, in order to lay the basis for new generations of both sort of products.

Relevance. Thermoplastic rubbers represent a class of materials of high industrial and academic interest, because they combine rubber-like properties with thermoplastic processability. Potentially, thermoplastic rubbers offer solutions to many outstanding problems with rubber technology, in particular the need to cope with the increasing pressure on recycling needs.

Deliverables. Project #252 has resulted in a clear understanding of the differences in phase behaviour between the two systems. In EPDM/PP/Oil TPVs, the phase morphology is fixed immediately after forming by the dynamic vulcanization process; in SEBS/PP/Oil blends the morphology is in dynamic equilibrium with the instantaneous shear intensity of the mixer or processing apparatusIt offers great scientific "understanding" of the phenomena to the developers of pertinent products in the participating companies.

Chemistry of Thermoplastic Rubbers Chemistry (projects 236, 344 and 346; 4.5 fte's):

Objectives. Project #236: dynamic property improvement of peroxide-cured ethylene-based polyolefinic thermoplastic vulcanizates. #344: development of new thermoplastic elastomers based on block-copolymers of acrylates and saturated olefinic blocks, as an entirely new line of thermoplastic rubbers. #346: development of a new thermo-reversible crosslink system for EPDM, based on maleic-anhydride grafting and neutralization with metal cations.

Relevance. Thermoplastic rubbers are strongly in focus, because of their potential recyclability. However, their penetration relative to vulcanized rubbers is still very limited, because of their problems to match the dynamic properties of vulcanized rubbers.

Deliverables. The first part of project #236 will be completed in 2004: the development of a peroxide curing agent, which substantially improves the properties of polyolefinic thermoplastic vulcanizates: patent applied for in 2003. A follow-up with further improvements via co-agents has just started end of 2002: the plan has been agreed upon and first results were gathered in 2003. #344 has reached a point, where the synthesis of block-copolymers has been achieved. The actual mechanical properties of the newly synthesized polymers are due in 2004. #346 has just started in August 2003: the first months were spent on literature study and project planning. •

Output 2003

Scientific Papers

K. Naskar and J.W.M Noordermeer: "Dynamically vulcanized PP/EPDM blends: Effects of different types of peroxides on the properties"; Rubber Chemistry and Technology, 76, 1001 (2003).

P. Sengupta and J.W.M. Noordermeer: "A comparative study of morphology and structure related properties of saturated olefinic thermoplastic elastomer blends of EPDM/PP/Oil and SEBS/PP/Oil"; Elastomerics, 38, 27 (2003).

K. Naskar and J.W.M. Noordermeer: "Dynamically vulcanized PP/EPDM blends: effects of different types of peroxides on the properties"; Elastomerics, 38, 167 (20030

S.C. Debnath, R.N. Datta and J.W.M. Noordermeer: "Understanding the chemistry of the rubber/silane reaction for silica reinforcement, using model olefins"; Rubber Chemistry and Technology, 76, 1311 (2003).

Proceedings:

P. Sengupta and J.W.M. Noordermeer: "The effect of composition and processing conditions on the morphology and properties of thermoplastic elastomer blends of SEBS/PP/Oil and dynamically vulcanized EPDM/PP/Oil blends": American Chemical Society, Rubber Division 163rd Spring Technical Meeting, San Francisco USA, 28-30 April 2003.

K. Naskar and J.W.M. Noordermeer: "Multifunctional peroxides as a means to improve properties of dynamically vulcanized PP/EPDM blends"; International Rubber Conference 2003, Nürnberg, Germany, 30 June – 3 July, 2003.

G.F.Sengers and A.D. Gotsis: "Distribution of processing oil in olefinic thermoplastic elastomer-compounds"; Polymer Processing Society Meeting, Athens Greece, 14-17 September 2003.

E.H.D. Donkers, B. Klumperman, M. van Dijk and C. Koning: "Well defined block copolymers by combination of living anionic polymerization and "living" free radical polymerization"; European Graduate School, 18 October 2003.

Patents

K. Naskar and J.W.M. Noordermeer: "Process for the preparation of a thermoplastic elastomeric vulcanizate".

Functional Polymer Systems

The interest in functional polymers and their applications is growing within industry and academia. Among the most significant drivers are the steadily increasing demand for information and communication, flexibly and at low cost, and for energy efficiency and energy generation. In these respects, functional polymers hold great promise. In contrast to traditional polymers, volumes will remain limited, and the added value is generally in the device. Academic developments are also providing a powerful impetus, where the major drivers are new synthetic capabilities, e.g. in supramolecular chemistry, analysis and manipulation possibilities down to the nanometer scale, and the boost in public funding for nanotechnology in general. The Technology Area Functional Polymer Systems intends to be a major player in the international research on functional polymers for new and breakthrough technologies, particularly in the areas of information and communication and sustainable energy. The present major lines of research in the DPI programme – displays and transistors, all-optical applications, and photovoltaic cells – are all very relevant, while the smaller programmes on nano-organised functional blends and on polymeric ion conductors may be seeds for future major activities. Most of the TA research is currently at the prototype level, but technological aspects of processing and long-term performance may also have to be considered. The decision has now been taken to set up a new FPS programme on responsive materials, sensors and actuators, which should start before 2005.

Partners

Industry:

Akzo-Nobel, Avery Dennison, DSM, Dow, ECN, Océ Technologies, Philips, Shell and TNO.
In the last quarter of 2003 Degussa joined as a new DPI and FPS partner.

Academia:

The research is being carried out in 8 institutions: UvA, ECN, LEI, LUW, RUG, TNO, TUD and TU/e. A new policy for invoking research proposals has been designed, which also considers leading international groups.

Budget

Total costs over 2003 were EUR 3.5 million (budget EUR 3.7 million). The number of FTEs allocated at the end of 2003 was around 40. There is a small number of vacancies.

Communications

- The Programme Committee, consisting of the PM, the partner representatives and four academic experts, met four times in the course of 2003.
- Subcluster theme meetings were held on LEDs/FETs (three, at TNO, the RUG and LEI), conductive blends (at TNO), all-optical materials (at the TU/e), photovoltaics (six times, twice at the TUD, at TNO, the LUW, RUG and UvA), and fuel cells (at the TUD). Extensive review reports have been written on each of the above themes, and are available on the Intranet.
- For individual research projects, bilateral meetings were held with the relevant partner industries when necessary, and on an informal basis.
- DPI FPS researchers frequently attended scientific conferences and regularly submitted papers to academic journals.

Highlights of 2003

- The DPI FPS Programme Manager and representatives of several DPI FPS partners have acted in a study by the Dutch Ministry of Economic Affairs on further stimulation of the area of functional polymers, with emphasis on new economic activity and wider applications. As a follow-up DPI has presented itself at several conferences with potential end-users.
- A new procedure has been implemented for generating projects via annual open calls for proposals, directed also to research groups outside The Netherlands. After a brainstorming meeting of the PC on long-term interests, a first call has been prepared for 2004, and the theme of 'responsive materials, sensors and actuators' has therein been identified as an important new DPI working area.
- In the LED/FET applications information about and insight in the charge-density dependence of the current is of utmost importance. In close collaboration between several industry and university groups, a new and very direct method has been found to obtain such quantitative insight at an unprecedented level of detail (#274); related to this the large differences as generally observed for mobilities in LEDs vs FETs could be explained consistently as being due to this density dependence only (#276).
- In the microstructuring of optical films a first proof of principle has been given of bi-layer photo-embossing, for the generation of complex components with dual optical and electro-optical functionalities (#298).
- First successes have been reached in optical nanolithography by employing the self-organisation of chiral liquid crystals (#277, 304).
- In the PV programme the research on PPV/fullerene blends has become an active third line of DPI research for prototype polymer solar cells, next to polymer/inorganic Graetzel-type cells and all-polymer blends. The strong influence of processing parameters on PPV/C60 morphology, critical for device performance, has been convincingly demonstrated down to the nanometer level (#326).
- A new class of materials has been identified and proposed for patenting which combines a mesogenic polymer backbone with ionic side groups substituted to a controllable degree. The materials hold potential for thermally, mechanically and chemically resistant products of which the ion conductivity can be tuned to specific battery or fuel-cell applications (#260, 409).

Projects

LEDs and transistors

In this theme, the main object of study has so far been the dye-filled LED for multicolour display applications, where charge transport is provided by a layer of conjugated polymer in a multilayer device, and colour emission is provided by the dispersed dyes. Secondary studies investigate the related phenomena of charge transport and device performance in polymeric field-effect transistors for plastic electronics. Although proofs of principle and LED products already exists, obstacles still remain for the industry in the barriers to charge injection, interface degradation, level of mobility, and transfer of energy from polymer to dye. For the future there is a shift of interest to new materials, towards transistors and to breakthrough device concepts.

Conductive blends

This theme aims at methods of making non-conducting polymers conductive in a controlled way by percolating networks of a dispersed second component. In commercial products, the filler fraction has to be relatively high to ensure a reasonable conductivity, which has a detrimental effect on other properties. One of the aims is to significantly lower the critical filler fraction. Other objectives are an increased level of conductivity and the introduction of attractive alternatives to carbon-black filler. Work is being undertaken to gain the necessary understanding and technological leads for making highly conductive carbon-black/rubber composites with good mechanical performance, especially for copier applications, to improve the outlet chances for polymer additives such as carbon-black, and to define routes to making thermoset polymers conductive in a controlled way using phthalocyanine salts, with patent options. The latter work, and more general scientific leads to morphology and property control via self-organisation networks, will be the future focus.

All-optical applications

In this theme the structuring of polymers on a nano- to micronscale via top-down aprroaches is combined with bottom-up techniques such as self-organisation of liquid crystals in order to generate new optical, electrical and electro-optical functionalities. A proof of principle is established in small-scale components and devices, with a special emphasis on LCDs and LEDs. Typically it is attempted to improve these devices with respect to brightness, light efficiency, viewing angle, and electro-optical characteristics. New initiatives are explored to use the generated concepts in soft electronics, optical recording, field-effect transistors and solar cells. Current projects focus on layered Bragg gratings, new polarization optics for LCDs, phase gratings for LEDs, large-scale nanolithography, and other new structuring routes.

Case

PolyLEDs

The fabrication of polymer light emitting diodes (PLED) consists of several steps that have to be performed in special conditions, i.e., a clean room and partly in a clove box with nitrogen atmosphere. Fabrication in these conditions is now possible in the state of the art plastic electronics device line at TNO Industrial Technology (picture 1). The substrates (glass covered with a transparent conductive oxide) are thoroughly cleaned before patterning (lithography).
The organic layers, respectively, PEDOT:PSS and the light emitting polymer, are applied by spin coating. Subsequently, the metal back electrode is evaporated in high vacuum.
The PLEDs are now tested (picture 2) and encapsulated to prevent device degradation by water and oxygen from the air.

Cleanroom at TNO Industrial Technology (picture 1)
The testing of a polymer light emitting diode (PLED) (Picture 2)


1.


2.

Photovoltaics

The PV programme is aimed at finding new concepts for photovoltaic cells, based on polymers. Such cells can develop into alternatives for current technologies, which employ crystalline silicon wafers or various types of inorganic thin film semiconductors. Polymeric solar cells hold the promise of very low materials and manufacturing costs and are therefore important candidates for large-scale use of photovoltaics. For this to become reality it is essential that sufficiently efficient, and equally important, very stable devices are demonstrated. As a spin-off, new photodetection technology may be developed.
The focus of the program is long term and the first aim is to realise working devices with a significant efficiency along two lines: hybrid inorganic/organic solid heterojunction systems and all-polymer n-p heterojunction devices.
A third concept, based on polymer/fullerene blends, might lead to a better understanding of nanomorphology vs device performance, and provide first true options for polymer PV. Given the large size, integrated structure and long-term focus of the programme, much of the new scientific insight generated will spin off into the polymer-LED area.

Fuel cells and batteries

In this area, the objective is to develop and demonstrate new polymer-electrolyte materials for PEM fuel cells and batteries. There is a need for alternatives to the current commercial PEM-fuel cell material and, more generally, for polymeric ion/proton conductors with good high-temperature stability for energy applications. It is hoped to develop proofs of principle based for LC sulfo-substituted polyimides as a high-strength, corrosion resistant and thermally stable solid-electrolytes.

Targets 2004

- DPI seeks to involve internationally leading groups with a track record in multidisciplinary research in the field and with first-hand access to new fundamental developments, in particular in the organic nanosciences.
- Choice by the FPS industrial members on new research directions and projects for a major part of the 40 fte 2004-2007 programme, via workshops and via a call for proposals to targeted Dutch and international research groups
- Increased attention for IP harvesting in programmes nearing their final stage
- Exploration of routes to meet the recent EZ intention in further stimulating the area of functional polymer systems
- Approach of several new industries for membership under the slightly improving industrial climate
- Follow-up actions on the outcome of the 2003 DPI portfolio analysis

Output 2003

Thesis

D.J. Versteeg, Direct laser writing and structuring of functional polymers for electro-optical switches, Eindhoven University 2003

Scientific publications

LED-FET
#241
Organic Electronics 4 (2003) 209-218, Interface stabilities in polymer light emitting diodes due to annealing

#274
- Synthetic Metals 135-136 (2003) 5, The Drude parameters for metallic PF6 doped polypyrrole
- Synthetic Metals 135-136 (2003) 243, The charge mobility in polymer LEDs - the low frequency impedance method
- Synthetic Metals 137 (2003) 891, Theory of bandwidth narrowing in oligo-acene crystals
- Phys. Stat. Sol. (c) 1 (2003) 144-147, Charge carrier properties below and above the metal-insulator transition in conjugated polymers – recent results
- Phys. Stat. Sol. (c) 1 (2003) 164-167, Temperature and field dependence of the mobility in 1D for a Gaussian density of states
- Appl. Phys. Lett. 83 (2003) 1246-1248, Effect of dye doping on the charge carrier balance in PPV light emitting diodes as measured by admittance spectroscopy
- Chemical Physics Letters 381 (2003) 392-396, Ab-initio study of energy-level alignments in polymer-dye blends

#275
Appl. Phys. Lett. 82 (2003) 985-987, Electro-optical properties of a polymer light-emitting diode with an injection-limited contact

#276
- Organic Electronics 4 (2003) 33-37, Local charge carrier mobility in disordered organic field-effect transistors
- Phys. Rev. Lett. 91 (2003) 216601, Unification of the hole transport in polymeric field-effect transistors and light-emitting diodes
- Polymer Preprints 44 (2003) 342, Charge transport in polymeric opto-electronic devices

Functional Polymer Blends
#294
- Macromolecules Rapid Communications, 24, 113, 2003; Automated Scanning Probe Microscopy as a New Tool for Combinatorial Polymer Research: Conductive CB/Poly(dimethylsiloxane) Composites

293
- Material Research, 18, 507, 2003, Victor,Subsurface Cracking during Indentation on Nanocomposite Hybrid Coatings on Polycarbonate
- Journal of Colloid and Interface Science, 2003, Surface Modification of Oxidic Nanoparticles using 3-Methacryloxypropyl- tri-methoxysilane
- Macromolecules, 36, 7585, 2003, Physical ageing of Polycarbonate

All Optical
#221
- Liquid crystals, Synthesis and properties of benzoate-based liquid crystalline thiol-ene monomers
- Polym. Mater. Sci. Eng., Multi-directional liquid crystal alignment techniques
- Adv. Mater., Patterned alignment of liquid crystals
- ACS Nat. M., Multi-directional Alignment techniques

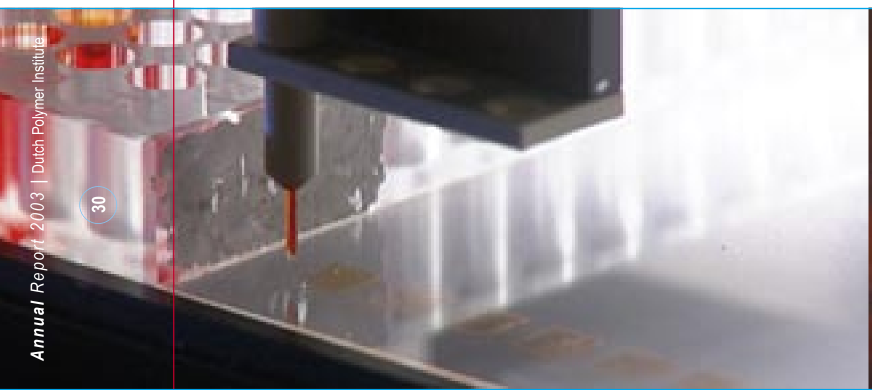
Photo-Voltaics
#324
- Journal of Polymer Science: Part A: Polymer Chemistry, 2003, 41, 3954-3964, New Soluble Functional Polymers By Free-Radical Copolymerisation of Methacrylates and Bipyridine Ruthenium Complexes
- J. Chem. Phys. 2003, 119, 10924-10929, Charge recombination in a poly(paraphenylene vinylene)-fullerene derivative composite film studied by transient, nonresonant hole-burning spectroscopy
- Angew. Chem. Int. Ed. 2003, 42, 3371-, Efficient methano[70]fullerene / MDMOPPV bulk heterojunction photovoltaic cells
- Adv. Mater, 2003, 15, 118-121, Photoinduced electron transfer and photovoltaic response of a MDMOPPV: TiO_2 bulk-heterojunction

Fuel Cells
#260
- Polymer, Synthesis and characterization of a watersoluble rigid-rod polymer
- J.Appl.Cryst., A small-angle neutron scattering investigation of rigid polyelectrolytes under shear

Patents
Eight inventions reported from the FPS programme from three of the five sub-clusters, have been chosen by the industry partners for further patent action.

Coating Technology

The refocusing of the program for coating technology that was initiated in 2002 came on stream in the course of 2003. Not less than ten new projects were started in the course of 2003, out a total of 16 active projects, making the portfolio of projects in line with the expectations as formulated by the programme committee in 2002. This high proportion of new projects in the portfolio has inevitably led to a temporarily reduction in the output. With external assistance the portfolio was analysed, confirming the directions as set by the program committee. The research is organised in five main themes:durability, adhesion, pigmentation, low temperature cure and biomaterials. This main themes are grouped in two subclusters.
The coating industry will be faced in the future



with an increasing demand of more reaction to health safety and environment and to quicker responses to changes in substrates and curing conditions. The most environmentally friendly coatings are the waterborne and powder coating as they can be applied virtually without the use of solvents. Some technical hurdles in the application process of these coatings are to be solved. The DPI projects on coating technology are focused on pioneering polymer research combined with application oriented material research. The research has been organised in five main themes: durability, adhesion, pigmentation, low temperature cure and bio-materials.
These five themes have been grouped into two subclusters.

In the future the coating industry will have to be more responsive to health safety and the environment and respond more rapidly to changes in substrates and curing conditions. Waterborne and powder coatings are the most environmentally friendly coatings as they can be applied virtually without the use of solvents, though some technical hurdles in the application process of these coatings are yet to be solved.

Partners

Industry:
Akzo Nobel, Dow, DSM, Océ, SEP, Shell and TNO. Degussa joined the technology area coatings at the end of the year

Knowledge institutes:
The research is carried out at Technical University of Eindhoven, University of Amsterdam, Wageningen University and Research Centre , TNO Industrial Technology, Agrotechnology and Food Innovations BV.

Budget

Total costs over 2003 were EUR 1.1 million (budget EUR 1.2 million)

Communication

The programme committee met five times in the course of 2003 and eight meetings took place with industrial consortium members. Two meetings were combined with an NWO-PPM project meeting.
The DPI coatings technology area participated in a combined research AIO workshop with the NIMR (Netherlands Institute for Metal Research) and the IOP-OT (the surface technology innovative research programme).

Highlights

- Dr van Benthem was appointed to the chair of coating technology at the Eindhoven University of Technology (TU/e)
- One project was successfully finished leading to a successfully defended PhD thesis.
- In the course of 2003 a total of 10 new projects started

Projects/targets

Durability
The resistance of industrial and decorative coatings to weathering as a result of outdoor exposure (UV radiation, heat/cold, moisture) is an extremely important topic in coating related industrial R&D. Yet as the vast experience and knowledge of industrial researchers on this topic is almost exclusively empiric in nature, the fundamentals of the changing coating properties during outdoor exposure are largely unknown. It is generally conceived that the outdoor exposure results in chemical changes in the coating composition, that the chemical changes result in changes in physical property changes of the coating and that the physical changes ultimately lead to changes in failure mode. Direct cause/effect correlation data have never been obtained but are essential for a better understanding of coating durability. It is the object of this study to make a start with such cause-effect studies on well-defined coating systems in a parallel project.

Adhesion
A first objective is to explain the adhesion of an organic coating on a polymeric substrate by studying in depth the relevant molecular and mesoscopic morphologies of the coating, the polymer support and their interface and by correlating these finding with the work of adhesion and total adherent energy.
A second objective is to obtain surfaces with very low adherence. It has been shown that very thin layers of low energy material can make a surface non-adherent. This films are however very vulnerable. By controlling the molecular structure of the coating network self-replenishing non-adherent coatings layer will be developed.

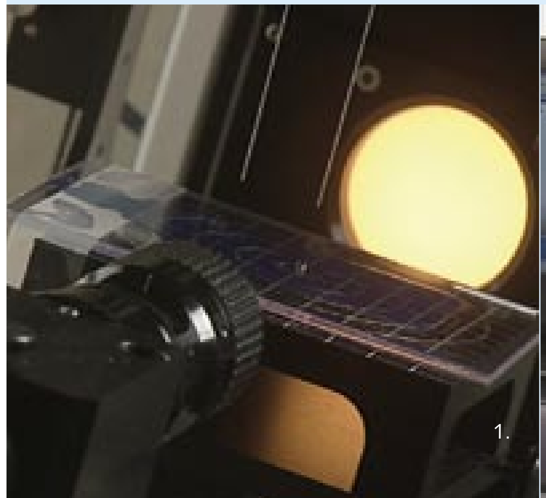
Pigmentation
The objective is to study the interaction between pigment surface and coating material and their influence on the final coating.
Adsorption of polymers and surfactants and the cinetic aspects of adsorption and desorption will be studied and compared to theoretical predictions.

Film formation
Powder coating is one of the most successful industrial paint systems that meet both environmental (zero emission of volatiles) and economical requirements (recycling of over-spray). A general trend in the powder coating technology is towards lower temperatures of film formation (flow, cure), typically lower than 180°C. The wish to coat heat sensitive substrates (wood, MDF, plastics) nowadays treated with solvent borne paint systems, with powder paints puts even more stringent demands to the cure window with respect to both rheology (levelling) and reactivity (cure)
In a former project (#194) the observation was made that acid functional binders for powder coating could be brought into an ionomeric form increasing thus the Tg, but without affecting the temperature of onset flow. This makes this type of ionomers interesting candidates as binder for powder coatings with improved flow. Other known options to circumvent this paradigm are latent systems with a “trigger”, e.g. radiation-induced (UV-curing powder coatings) and moisture-induced triggers, but they have their specific industrial disadvantages (performance, price).

Case

Functional coatings

Coatings are used for a variety of reasons, mainly for protection, decoration, and other “functional” reasons. Being non-wetting for e.g. water is often a favorable property, but a paint should also perfectly wet the object to be painted. This kind of properties can be determined by measuring surface tensions and wetting angles. The pictures (1,2,3) show the equipment used. From the shape of a pendent drop the surface tension can be deduced (the lower the surface tension, the more the droplet will be stretched vertically, i.e. it will deviate from the spherical shape). The ease of wetting can be analysed and quantified from a microscopic picture of the droplet on a plate. In the picture shown the wetting is fairly poor. Depending on the surface structure (roughness; composition heterogeneity) a surface that is not well wetted by a paint stays wetted once it has been wetted. This phenomenon can be studied by watching the shape of a droplet that is forced to walk over a plate by tilting the plate. In such a case the microscope image shows that the wetting angle in front of the walking droplet is much larger than the angle behind the droplet.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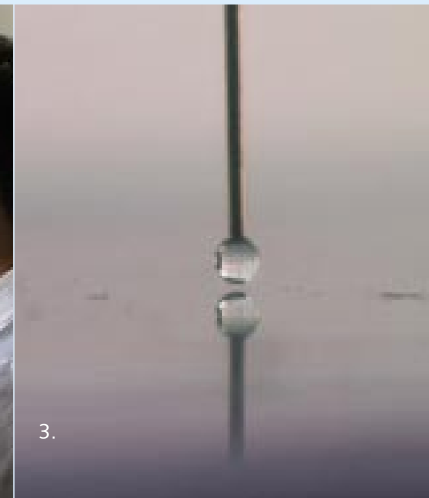
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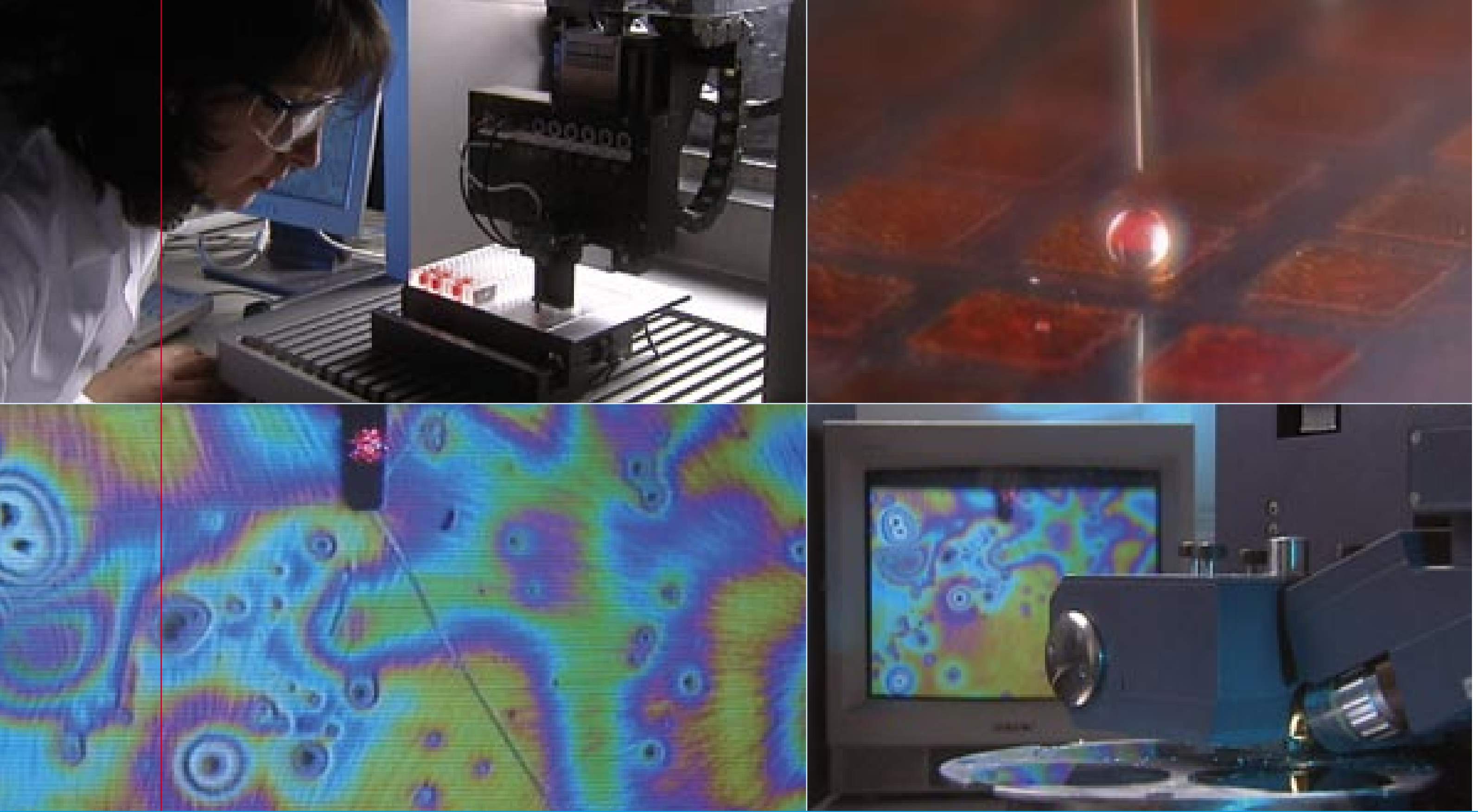

New is to encapsulate one of the reactants of the thermosetting formulation (c.q. the cross-linker), mix the encapsulated compound with the resin (and pigments) in the extruder and de-capsulate the cross-linker in-situ as a response to increasing the temperature to curing temperature, so as to allow molecular mixing of the binder ingredients and subsequent cross linking.

Biomaterials (#451)

The feasibility of the use of sustainable, renewable resources for the development of alternative monomers and co-monomers for the production of toner and powder coating resins, without compromising the final product properties, will be studied. Dianhydrosugars from different sources are very interesting monomers as they form rigid bicyclical compounds having a unique stereochemistry. This diols will be polymerised to polycarbonates, polyurethanes and polyester oligomers using specific polymerisation processes. Relationships will be established between the used monomers and reaction conditions and the final polymer properties.

Program targets 2004

With the partners of the program committee the possibilities for and interests in a new sub-area functional coatings will be explored, and potential project proposal ill be defined.

Look for cooperation between other clusters: Engineering polymers on durability and FPS on functional coatings

Potential interested member companies will be actively searched, both among coating producers, producers of raw material and end-users ●

Output 2003

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High Throughput Experimentation

From academic research to industrial applications

Over the past few decades, combinatorial and high-throughput techniques have revolutionized the way in which research in biochemistry, genetics and the pharmaceutical industry is performed. Nowadays, nearly all pharmaceutical companies employ automated high-throughput screening methodologies for the development of novel "lead" structures. The fast identification of "hits" as well as the elimination of potential but ultimately unsuccessful candidates at an early stage facilitates a substantial reduction of time-to-market and cost savings. Current research and product development in these fields cannot be imagined without the extensive application of a wide range of automated synthetic procedures and rapid screening techniques. In recent years, a similar development has taken place in the field of polymer and materials science.

Triggered by this structural change in R&D and by extensive discussions with leading technology and chemical companies, first activities in the area of automated parallel polymerizations were started within the DPI core programme at the beginning of 2002. Promising initial results were obtained quickly and the intense collaborations with CMR/HTE equipment manufacturers as well as leading chemical companies, which resulted as a consequence, led to the conclusion that a new approach for a successful R&D in this challenging and important area of combinatorial materials research is required. As a result, a new (sub)cluster was initially formed within the framework of the DPI and was only shortly later established as a new, independent Technology Area at the end of 2002. With Microdrop and AnalytikJena two additional technology companies joined the DPI during the course of 2003. This add-on of expertise and knowledge facilitated a significant widening of the program's scope. Furthermore, several chemical companies operating on a worldwide scale, research institutes and further technology companies expressed their interest in a membership and subsequently joined the technology area in 2004. This clearly demonstrates the success and acceptance of the established knowledge and technology platform and indicates its role as one of the leading centers of high-throughput experimentation and combinatorial materials research worldwide.

Mission

The mission of the Technology Area CMR/HTE is to develop and apply novel strategies and approaches for polymer research. For this purpose, new instruments and software are combined with state-of-the-art "conventional" polymer knowledge and statistical as well as theoretical methods aiming at a more detailed understanding of structure-property relationships. Ultimately, it is the vision to achieve a real design of materials and the development of a materials informatics platform. Our main focus is the creation of complete workflows, covering the design of the experiments, automated and parallel synthesis (incl. blending and preparation of hybrid materials), the fast characterization of molar mass, poly-disper-sity, kind and degree of functionalization as well as kinetics, the reliable preparation of thin film libraries, the fast and efficient investigation of important materials properties, formulation, up-scaling, combinatorial compounding, processing and a complete data-hand-ling and data-mining workflow. Besides these research-directed targets, the education of students, Ph.D. students and Post-Docs in the new field of combinatorial polymer science is of prime importance.

Communications

A second DPI workshop on Combinatorial Materials Research at the Eindhoven University of Technology on May 15 and 16, 2003 could repeat the success of the first event in 2002. Over 120 scientists from both industry and academia attended the event, with lectures by leading industrial and academic groups, experts from technology providers and Ph.D. students and Post-Docs from Eindhoven presenting results at the very forefront of research in this field. The scope of the hands-on demonstrations widened significantly and apart from automated parallel synthesis, included novel software developments in the field of combinatorial materials research (design of experiment (DoE), molecular modeling, solutions for data handling and data mining etc.) and ink-jet printing as a cutting edge technology in the area of automated and parallel preparation of thin films and coatings.

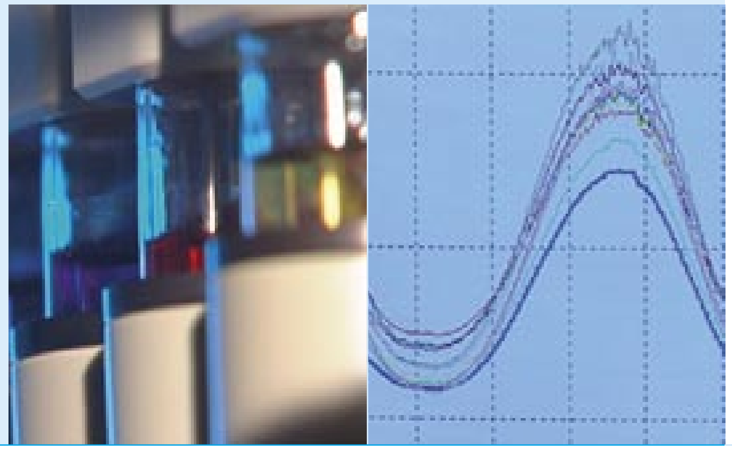
Most recent results in the field of Combinatorial Materials Research were published in a special issue of Macromol. Rapid Commun. 2003, 24 (guest editor: U. S. Schubert) with contributions from leading experts from both industry and academia.

In three meetings, the Programme Committee was updated concerning the latest results of ongoing research (with presentations by all involved Ph.D. students and Post-Docs).

Furthermore, the research of the Technology Area (incl. the related core project) was communicated to the scientific world in 24 publications and 47 oral and poster presentations at national and international meetings.

Automated parallel synthesis

2-Oxazolines can be polymerized by a living polymerization technique (cationic ring opening polymerization), which facilitates an easy access to block copolymers. Block copolymers as well as homo polymers from poly(oxazoline)s are used in a variety of different applications and exhibit economic potential. The screening for suitable monomers, initiators and termination agents, the variation of temperature and monomer/initiator ratios (molecular weight) and the combination of monomers as building blocks for copolymers strongly favor an automated parallel synthesis approach. Homopolymers and random copolymers of methyl-, ethyl-, nonyl- and phenyl-2-oxazoline were successfully prepared in automated synthesizers. The liquid-handling unit of the synthesizer realizes the full automation and acceleration of the polymerization recipe, special heating reactors enable the determination of activation energies in a single polymerization run and online GC and GPC set-ups makes kinetic data easily accessible.



Partners

Chemspeed, NTI and Avantium. In 2003, AnalytikJena AG/Jena and Microdrop/Norderstedt joined as new partners. Several companies expressed their interest in a DPI membership and joined the HTE/CMR cluster in 2004: Degussa AG, Bayer MaterialsScience AG, Dow Benelux, Forschungsgesellschaft Kunststoffe e.V., Hysitron, Ticona and Basell. In addition, CMR/HTE related research projects are dealt with in the Core Programme and the application relevance of the approach is addressed by a first cooperation with the Technology Area Coating Technology.

Budget 2003

Total costs over 2003 were EUR 1.0 million (budget EUR 0.9 million) and will increase to EUR 2.2 million in 2004.

Highlights 2003

- Successful examples of block copolymerizations, metallo-supramolecular and living anionic polymerizations on the automated parallel synthesizers have been performed.
- The new automated parallel synthesizer Accelerator from Chemspeed was set-up and established.
- Activities in data handling, modeling and data base construction in particular for the field of polymer research have been started up successfully.
- Ink-jet dispensing as cutting edge technology for polymer thin film library preparation was set-up and established in close cooperation with Microdrop.
- The toolbox of high-throughput analytical techniques could be significantly enlarged by an automated contact angle measurement device, an automated viscometry setup and the expansion of chromatographic analysis capacities.
- A second DPI workshop High-Throughput Experimentation and Combinatorial Materials Research was organized at the Eindhoven University of Technology.
- With AnalytikJena and Microdrop two new companies joined the Technology Area.

Projects

The CMR/HTE program of the DPI is intrinsically multidisciplinary in character, combining both scientific as well as technology aspects. With the participation of specialized manufacturers an unique standing with relation to the technology competences could be achieved. The Technology Area is at present subdivided into the research areas Synthesis & Catalysis, Library Preparation & Screening, Data Handling, Data Mining & Modeling and Cooperation with other TA. In 2004, this will be expanded by the subclusters Combinatorial Compounding and Formulation as well as Characterization.

Synthesis & Catalysis

The research of this area is dedicated to the development of fully automated, parallel polymerization methodologies, polymerization catalyst screening, the investigation of polymerization kinetics and the synthesis of novel polymeric materials (such as block copolymers) in combination with automated purification techniques and characterization methods. Automated parallel polymerizations were expanded from living cationic and controlled radical poly-merizations (ATRP) to anionic as well as supramolecular poly-meri-za-tions. Moreover, chromatographic techniques for kinetic studies as well as the determination of molecular weights have been implemented into the combinatorial workflow and are operated on a daily routine basis.

In the near future, the portfolio will be extended towards advanced technologies in automated parallel synthesis, which meet the requirements of industrial processes.

Library Preparation & Screening

The projects of this subcluster aim at automated preparation, investigation and characterization of polymer coatings and thin films of pure polymers, blends and formulations. The characterization covers the investigation of the chemical composition, the film morphology, roughness, hardness as well as stiffness of the films. With ink-jet dispensing, a cutting-edge technology for the creation of thin film polymer libraries of different film thickness and composition has been set-up and established in the subcluster Library Preparation & Screening. Furthermore, a fast parallel molecular weight screening methodology for polyelectrolytes as well as nitrogen and metal containing polymers with low solubility has been built up on the basis of an automated viscometry measurement device.

Data Handling, Data Mining & Modeling

The design of experiment, the data handling and database utilization is one major issue in com-binatorial material research and high-throughput screening. The efficient data evaluation, an identification of "hits" or trends, the establishment of "structure-property-relationships" and finally the development of a kind of "material informatics" is in the focus of the subcluster Data Handling, Data Mining & Modeling.

Cooperation with other TA

The scope of performing combinatorial materials research will be widened by cooperation projects with other Knowledge Institutes and Technology Areas within the DPI. First projects on the durability of coatings have been initiated in cooperation with the TA Coating Technology and TNO.

In summary, the infrastructure for synthesis, analysis, materials property testing and process optimization in combination with the chemical and technological expertise of the partner companies created a unique position for a research institute. In the future, this will be combined with a modular yet integrated data-handling, data-mining and modeling solution. •

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Core Programme & Strategic Projects

The DPI Core programme has been established at the start of phase-II (2002 – 2006) of the DPI. The DPI Core Programme has been initiated as an enabling science activity, supporting research in the various TAs with a twofold objective:

Enabling science:

- Fostering advanced characterisation techniques and modeling activities to support research programmes in the various Technology Areas, and

Emerging technology:

- Incubating new research activities, which are fostered for some time within the DPI Core, but may in due course develop into a new Technology Area with new and/or existing industrial partners (e.g. "High-throughput experimentation/Combinatorial Materials Research" , which is now an independent TA).

In 2003 the topics within the DPI Core Programme have been expanded with a third activity area (see also below at projects), the Strategic Projects.

Partners

Industry

The DPI Core Programme has no direct industrial partners. The Programme Committee for the DPI Core Programme is composed of the PMs of the various TAs with the Scientific Director as the Chairman. The proposals for the Core Programme are discussed in separate meetings, often in conjunction with the regular MT meetings, albeit with a separate agenda. Companies with a particular interest in specific techniques can do so by acquiring an "earmarked" ticket and via membership of the most appropriate TA, e.g. Dow in 2003 with an additional ticket for polymer characterization in the TA "High-throughput experimentation" .

Academia

Universities of Amsterdam (Schoenmakers c.s.), Twente (Vancso c.s), Eindhoven (Goossens, Loos, de With, Flipse, Meijer c.s., Michels c.s.), Maastricht, Wageningen (Cohen Stuart), Athens (Theodorou), Leeds (Mc Leish) and institutes such as A&F in Wageningen.

Budget

Total costs over 2003 were EUR 2.9 million (budget EUR 3.3 million). The number of allocated FTEs at the end of 2002 was close to 30.

Communications

- The Programme Committee for the Core Programme had regular meetings in 2003 discussing existing and new proposals including the new activities such as Strategic Projects. Often researchers have been invited to the meetings in order to elucidate their project proposals.
- In the area of polymer characterisation, various meetings have been organized with industry to streamline the activities. Prof. P. Schoenmakers (UvA) chaired these sessions.
- Ample discussions have been organized with existing DPI industrial partners and potential new partners to set up a new TA "Biomaterials".

Highlights of 2003

- A nucleus has been created for a nationwide activity on polymer characterization, serving the DPI community;
- The modelling activities, backed up by experimental validation, on the "toughness" topic reached a breakthrough status. Prof. H.E.H. (Han) Meijer has been invited to communicate the results at the IUPAC meeting in Paris (July, 2004) as plenary speaker;
- Dr Jasper van Gucht, DPI project #184, obtained the "Houwink prize" for the best thesis of the year 2003 at the PTN nationwide polymer meeting in Lunteren;
- Well-known expert groups such as prof. T. Mc Leish (Leeds) and prof. Theodorou (Athens) started cooperation within the DPI Core programme in 2003;
- Two out of the 3 DPI awards have been presented to graduates working on projects in the DPI Core Programme, viz. Dr A. Bogaerds and Dr I. Bazhlekov at the DPI annual meeting in November 2003.

Projects

Mesoscopic Chemistry & Physics

The projects in this subcluster cover a wide range of Science related to the fact that these projects have been transferred from the Subcluster "Mesoscopic Chemistry & Physics" in the DPI phase-I. Most of the projects have been finished in 2003 or have been transferred to other subareas in the Core, notably to the subarea "Structure vs. Performance". Ongoing projects are dealing with nano-composites (group Picken, TU-Delft) and "living polymers" (Sudholter c.s. Wageningen). In the case of "living polymers", scientific recognition is growing, e.g. the thesis of Dr Jasper van der Gught (#184) got the Houwink Award at "Dutch Polymer Days" in February 2003.

Polymer Characterization

In this theme various projects are clustered on chemical and physical characterization of polymer systems involving the Universities of Amsterdam (Schoenmakers c.s.), Eindhoven (Goossens, Loos, Flipse) and Twente (Vancso c.s.). The projects comprise topics such as Network Characterization, notably unsaturated polyester resins, accurate distributions on the possibility of using fast SEC techniques, chemical surface characterization and physical surface characterization. The subprogramme is co-ordinated by prof. P. Schoenmakers (UvA).

Bio-related polymers & polymers in medicine

Originally two related but independent routes have been envisaged:

a) To explore the possibilities of Biotechnology for making specific (co)polymers (e.g. enzymatic polymerizations;

b) Biomaterials (medical implants)

ad a)

Two projects are running with the focus on making specific block-copolymers albeit via different routes, respectively via enzymatic polymerization in the group of prof. C. Koning and via encoding of (pro)genes, a project in between A&F (Wageningen) and the Laboratory of Physical end Colloid Chemistry at Wageningen University (Cohen Stuart). The latter project is part of a multidisciplinary approach to novel block copolymers, designed for electrostatic self-assembly into non-brittle high-modulus fibers and/or nanostructured gels.

Ad b)

The projects in the area of Biomaterials (b) have been placed into Strategic Projects, see below.

Structure vs. Performance

A coherent set of projects focused on fluid dynamics (rheology) and solid-state properties is running at, on the one hand at the TU/e in the groups of Meijer (HEH) and Michels and on the other hand with foreign expert groups such as Theodorou c.s. (Athens) and McLeish c.s. (Leeds). The aim is to better understand and generalise experimental results as well as reducing the number of experiments significantly, placing an emphasis on modelling validated by experiments. A basic understanding of polymer properties, notably fluid dynamics (rheology) and solid-state properties, e.g. toughness, will deliver a strong knowledge base for the DPI community and a high profile and visibility in the academic world.

Strategic Projects

1. Breakthrough Projects

In 2003 various so-called Breakthrough projects have been proposed and granted. The basic idea is that researchers explore a novel idea/concept in the period of 1 year and then the projects are evaluated. Projects started in 2003 encompass "highly filled nano-composites" (chitosan with nano-sized hydroxy appetite) for medical applications, "Reaction intensification through electron beam initiation" (the use of EB radiation to produce a high concentration of active seed nuclei in a loop reactor), "Solubility of polymers in Ionic liquids" (to explore the use of ionic liquids in polymerization reactions and polymer processing). Some projects have been granted in 2003 but will start in 2004 such as "Mono-Polymer Composites" (to reinforce plastics with fibers of the same kind, e.g. all-PP composites) and "Microwave assisted polymerizations". The number of Breakthrough projects, running + starting is 7, all for the period of one year.

2. Biomaterials

The area of Biomaterials is considered to be an important topic for the DPI with the focus on "what can DPI offer to the world of Polymers in Medical Technology". A subgroup in between the TU/e (Baaijens/E.W.Meijer) and the UT (Feijen) involving 3 Ph.D. students is focusing on "Bioactive scaffolds for tissue engineering of cardiovascular substitutes" and started at the end of 2003. Related to that subgroup is a project on the production of scaffolds with tubular shapes aimed to be used for blood vessel/artery tissue engineering via electro-spinning. In close cooperation with the University of Maastricht a project on intervertebral disc prostheses has been initiated.

3. Miscellaneous

- A number of projects have been initiated which are business-related such as "Improved knowledge capture and data handling in polymer research" as a feasibility project and "Polymers in Road constructions" to explore the possibility for the use of polymers in advanced road constructions.
- DPI granted scholarships to the following PhD Students at the Delft University: Basavaraj Katageri, Shyam Parameswaran and Ciptanti Sisbandini
- DPI nominated Ivan Bazhlekov, Arjen Bogaerds and Josien Krijgsman for the golden thesis award. Dr Arjen Bogaerds carried of the golden thesis award.
- Certificates of Invention have been awarded to: U.S. Agarwal, C.W.M. Bastiaansen, A.M.G. van Benthem, D.J. Broer, J.C.M. Brokken,L. Corbeij, P.J.W. Deckers, R.J. Gaymans, B. Hessen, H.J.B. Jagt, L. Klumperman, W.P. Kretschmer, J. Krijgsman, V.G.R. Lima, S. Rastogi, R. Venkatesh, J.T.A. Wilderbeek and G. de Wit
- In order to prepare DPI's Future Blueprint a special task force has been set up. Directed by the task force an assessment of DPI's current position from the stakeholders perspective (stakeholders survey) and a portfolio analysis have been presented. Based on these analyses, the task force gave recommendations for DPI's blueprint for the future, which will be included in the business plan.

Targets 2004

- Restructuring of current programme into clear Enabling Science (polymer characterisation, structure performance), Emerging Technology e.g. bio-related polymers (both 'science oriented' corporate research) and strategic ('development/shorter-term-application oriented') programmes
- Clustering of Enabling Science and Emerging Technology programmes into coherent parts, with subcluster coordinators and thematic workshops
- Improvement of Core output management
- Strengthening of synergy between appropriate Core and TA activities
- Workshop on perspective of Emerging Technology programmes Biopolymers and Biomaterials, with new TA as option ●

f a c t s h e e t

Output 2002

Thesis

C.P. Beyers, In-line monitoring of controlled radical polymerisation reactions with near infrared spectroscopy, TU Eindhoven, 2003

I.B. Bazhlekov, Non-singular bounderay-integral method for deformable drops in viscous flows, TU Eindhoven, 2003

J. van der Gucht, Equilibrium Polymers in Solution and at Interfaces", Wageningen University, 2003

Scientific Papers

H. Fischer, "Polymer nanocomposites: from fundamental research to specific applications", Materials Science and Engineering C 23 (2003) 763-772

M.M.F. Adamy, A.M. van Herk, M. Destarac, M.J. Monteiro, Influence of the chemical structure of MADIX agents on the RAFT polymerisation of styrene, Macromolecules, 36 , 2293-2301, (2003).

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Pattern Replication by Confined Dewetting S. Harkema, E. Schäffer, M.D. Morariu, and U. Steiner Langmuir 19, 9714-9718 2003

Hierarchical structure formation and pattern replication induced by an electric field M. D. Morariu, N. E. Voicu, E. Schäffer, Z. Lin, T. P. Russell, and U. Steiner Nature Materials, 2, 48-52 2003

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Polymer characterization

The preparation of tough heterogeneous systems based on amorphous polymers is focussed on changing the deformation mechanism from crazing to shear yielding. This, in combination with cavitation for delocalisation of the strain, can be achieved by decreasing the morphology size such that the interparticle local thickness becomes too small to allow for craze formation. For a minimum loss in modulus and strength the size of the rubber morphology has to be decreased to below 1 micrometer. Several successful systems have been developed.
The change in mode of microscopic deformation has been studied by time-resolved X-ray scattering during mechanical testing using synchrotron radiation at the Dutch-Belgian Beamline (DUBBLE) at ESRF, Grenoble, France. At this SAXS/WAXS station, several sample environments, such tensile tester, pressure cells, rheometers, and heating devices, can be inserted. Simultaneously, wide- and small-angle scattering patterns can be obtained with a time-resolution of seconds probing structures of polymers from 1-1000 Å.