SYNTHESIS REPORT

NON - CONFIDENTIAL

Contract no: **BRPR-CT98-0771**
Project no: **BE97 - 4584**
Title: **Mini Derucom**

Project coordinator:	• Codan Gummi A/S ("**Codan**")
Partners:	• Université Pierre et Marie Curie – Paris VI, Laboratoire Rhéologie et Mise en Oeuvre des Polymères ("**UPMC**") • Universität Paderborn, Institut für Kunststofftechnik KTP, ("**KTP**") • Thona SA ("**Thona**") • Trelleborg Industrie AB, Materials Division ("**Trelleborg**") • Lusorol GmbH ("**Lusorol**") • L'Isolante ("**Isolante**") • DSM Elastomers, Global R&D ("**DSM**") • Volkswagen AG ("**VW**")
Subcontractors:	• Degussa AG ("**Degussa**"), Bühler GmbH ("**Bühler**")

Reference period: from 01.11.1998 to 31.10.2001
Starting date: 01.11.1998 Duration: 36 months

Date of issue of this report: 28.03.2002

Project funded by the European Community
under the Industrial & Materials Technologies
Programme (Brite-EuRam III)

2 Index

3 Summary

3.1 Keywords

- Raw Material Quality Parameter Variations
- Raw Material Changes in the Mixing Room
- RELMA
- RPA
- Quality Assurance Concept

3.2 Executive Summary

In the Mini Derucom project quality routines like raw material acceptance tests and similar were investigated in the mixing rooms of the processing partners. It was found that most of the raw materials are specified sufficiently with a number of quality parameters. The only quality parameter, which varies significantly with respect to its specification limits, is the pellet hardness of carbon black. To the contrary, the carbon black fines content is low in the condition of delivery but can change dramatically within the mixing room in dependence of the conveying system and its settings.

Some quality parameters of EPDM polymers were found not specified exactly though influencing the process significantly. The degree of long chain branching and the content of processing aids can be named here. These parameters have been investigated with respect to end article quality in case of different applications within the Mini Derucom project. Furthermore, the exchange of raw materials at constant raw material specification in special application like covered rolls, rubber sleeves and closed cell insulation materials was found to some extend impossible. This means that raw material specifications can't be standardized. The specification limits of raw materials must be fixed between the rubber processor and raw material suppliers with respect to end article needs. However, only a number of selected raw materials have been able focused in the project, which have been appropriated for investigations in the project. Furthermore, it was decided to focus those raw materials, which are used in recipes in large amounts. On the one hand these parameters are supposed to have influence on the process respectively the quality but on the other hand the information gained in the project is surely limited. The main results of all studies in the Mini Derucom project can be summarized as follows:

It is almost impossible to find general rules in terms of quality prediction of end article properties. The connection between end article properties and influencing factors depends strongly on the end article itself, its production process and the type of raw materials used. It can be stated that there are recipes showing significant variations of compound properties in case of provoked variations though the end article quality stays constant. The opposite effect was observed, too: The compound properties exhibit no variations at all though the end article quality varies in dependence of the parameter variations applied. If a direct correlation between e.g. raw material quality parameter variations, compound properties and end article quality could have been discovered, only highly sophisticated testing methods like RELMA, the RPA or the TOPO method were found to have predictive capabilities. On the other hand the success of end article prediction can't be addressed to a single testing method. It can be stated that the type of testing method depends on the recipe composition respectively the type of application, too. The reason must be seen in the fact that raw materials have a "statistical" nature, which are handled through processes that are also "statistical". It follows that whatever the complexity of a test on the material is at a given point of the process, the results of testing procedures have very limited predictive capabilities in a generalized way. On the other hand it was found that the mixer is a very sensitive machine to discover influences arising from raw materials. Assuming good sensor technology and data assembling units are installed on the mixer, internal mixers can be used as a sensor indicating quality problems of raw materials. However, this only works if the mixing process will be not controlled.

It was concluded to set up a quality assurance concept for the mixing room covering different fields of quality impacts: At first, knowledge about the influence of raw material quality parameter variations has to be assembled in a data base working like an expert knowledge system. Such expert knowledge includes more general information, which is independent of the application, as well as strictly application related factory floor information. The Mini Derucom project clearly demonstrated corresponding guidelines to gain expert knowledge on basis of a so-called "Mixing Room Study" and a "Parameter Analysis Study". Every rubber processor must perform these studies by himself due to the strong relation of quality influencing factors to the recipe respectively certain end article properties. Some general information like the influence of the carbon black pellet hardness, the fines content or the EPDM quality parameter long chain branching has been clarified already in the Mini Derucom project, which then can be used in expert knowledge systems. Furthermore, such systems should also contain information about the mixing procedure like the optimum fill factor, which is often disregarded. Another field of quality impacts in the mixing room covers ideal equipment, which is recommended used. Herewith, homogenous compound quality within one batch and from batch to batch can be obtained with respect to process efficiency. If the type of application will be un-sensitive against impacts of any kind, such equipment would at least have the advantage of improved process efficiency. The type of advisable equipment or special features, which are in some cases known to improve compound quality and mixing efficiency for long, can be listed as follows: a) mixing room control: systems to handle and to control raw material data, weighing procedures, the mixing process and testing results like MixCont or simpler ones; b) mixer: latest rotor geometry, hydraulic activated ram, variable rotor speed, automated oil injection; c) conveying system: dense phase conveying system including a rubber in-liner if low structured blacks have to be conveyed; d) minimum testing equipment: RELMA, RPA, QS extruder. The basic idea is to serve sufficient compound quality in terms of dispersive and distributive mixing behaviour and to test only these properties in the mixing room. Rubber processors would only have to investigate correlations between the results of these methods and end article quality.

The advanced testing methods RELMA and RPA have been improved within the project, too. RELMA was developed to be a tool for raw material characterization as well. Furthermore, the whole unit was improved in technical respect significantly. The flexible optical cable was exchanged towards a fixed optic. Herewith the signals of sulphur could be amplified. The burned spot was homogenized and handling of the unit was improved by the application of new standard software. At the same time new sensor technology (spectrometer) and new gratings were bought, which have further amplified the sulphur signals about factor 40. Thus RELMA was developed a significant step forward to be a standard analysis tool in the mixing room. Similar developments were done with the RPA. Standard protocols were set up and processability indicators were introduced, able to distinguish the processing behaviour of rubber compounds more properly than e.g. the Mooney device. Furthermore, software was developed, able to qualify and quantify the non-linearity of compounds and it was found that the distortion of the response of a sinusoidal signal depends of the type of gum and the amount of fillers in the recipe. Last but not least the temperature conditions within the RPA cavity have been found far away from isothermal conditions.

The most important realization of the Mini Derucom project must be seen in the fact that the recipe composition, the raw materials' quality parameters and the processing equipment strongly depend on each other and to end article quality. There are no general rules available, which are able to describe quality of rubber articles. It makes not much sense to optimize only one step of the quality influencing fields: recipe composition, raw material quality, weighing accuracy and raw material handling, mixing quality, testing strategy. It only makes sense to develop a strategy, which can be then applied in a certain mixing room and to certain recipe respectively end article needs.

4 Mini Derucom Consortium

4.1 Partner Contact

Karl-Heinz Freitag
Bühler GmbH
Ernst-Amme Straße 19
38114 Braunschweig
Germany

Tel.: 0049-(0)531-594-2142
Fax: 0049-(0)531-594-2123
e-mail: karl-heinz.freitag@buhlergroup.com

Dr. Jürgen Fuehrer
Osnarol GmbH
Frankensteiner Straße 15a
49205 Hasbergen
Germany

Tel.: 0049-(0)5405-96120
Fax: 0049-(0)5405-96121
e-mail: fuehrer@12move.de

Frédéric Gouaderès
European Commission
Directorate C.I.
Rue de la Loi 200
1049 Bruxelles
Belgium

Tel.: 0032
Fax: 0032 – 2 – 2958046
e-mail: frederic.gouarderes@cec.eu.int

Villy Hasemann
Codan Gummi A/S
Københavnsvej 104
4600 Køge
Denmark

Tel.: 0045-56646587
Fax: 0045-56646546
e-mail: vh@codan.com

Prof. Dr. Jean Léopold Leblanc
Université Pierre et Marie Curie
L.R.M.O.P., Paris
60, rue Auber
94408 Vitry sur Seine Cedex
France

Tel.: 0033-1-49605782
Fax: 0033-1-49607066
e-mail: jleblanc@ccr.jussieu.fr

Prof. Dr.-Ing. Andreas Limper
Universität Paderborn
Institut für Kunststofftechnik, FB 10
Warburger Str. 100
33098 Paderborn
Germany

Tel.: 0049-(0)5251-60-3052
Fax: 0049-(0)5251-60-3821
e-mail: limper@ktp.uni-paderborn.de

Urban Magnusson
Trelleborg Industri AB
Henry Dunkers Gata 2
SE-231 81 Trelleborg
Sweden

Tel.: 0046-(0)410-514-57
Fax: 0046-(0)410-16298
e-mail: urban.magnusson@trelleborg.com

Thomas Riedemann
Degussa AG
Rodenbacher Chaussee 4
63403 Hanau (-Wolfgang)
Germany

Tel.: 0049-(0)6181-59-3108
Fax: 0049-(0)6181-59-4482
e-mail: thomas.riedemann@degussa.com

Carsten Rüter
Thona Belgium
Industriestraße 36
4700 Eupen
Belgium

Tel.: 0032-(0)87-595430
Fax: 0032-(0)87-744473
e-mail: Cruter@thona-group.com

Dr. Peter Wöss
Isolante Service GmbH
Hövelmarkt 7
33161 Hövelhof
Germany

Tel.: 0049-(0)5257-98400
Fax: 0049-(0)5257-984955
e-mail: isolante@aol.com

Dr. Lutz Wohlfarth
Volkswagen AG, Abt. K-GQLB
Postfach 1437-1
38436 Wolfsburg
Germany

Tel.: 0049-(0)5361-926049
Fax: 0049-(0)5361-978095
e-mail: Lutz.Wohlfarth@volkswagen.de

Dr. Germ Visser
DSM Elastomers
P.O. Box 1130
6160 BC Geleen
The Netherlands

Tel.: 0031-46-4761084
Fax: 0031-46-4761197
e-mail: germ.visser@dsm.com

4.2 Description of the Consortium

Codan Gummi A/S, Denmark

CODAN GUMMI A/S belongs to the Danish A.P. MØLLER Group, which is operating in Oil & Gas exploration, Shipping, Aviation, Retailing, IT, and Industry and run activities world wide in more than 100 countries, totally more than 40.000 employees.
CODAN GUMMI A/S is part of the ROULUNDS Group, which belongs to the MÆRSK Industries, part of the A.P. MØLLER Group.
CODAN GUMMI A/S is divided in an Automotive and an Industrial part. The head quarter and main production is placed in Køge, Denmark, and as ROULUNDS CODAN, production sites are placed in England, Wales, France, Argentina, Mexico, India and Korea, furthermore associated companies in China, Thailand and Malaysia.
In Denmark CODAN GUMMI A/S manufacture hoses and hose assemblies for automotive as well as industrial applications, and for general industrial applications profiles and mouldings by IM, TM and CM.
CODAN GUMMI A/S Automotive is an OEM supplier to the European car industry, with approx. 85% of total turnover to Europe and 15% to the rest of the world.
CODAN GUMMI A/S employs 450 people in Denmark, and, together with the foreign production sites, totally 800 people.

Thona SA, Belgium

Thona Group is dedicated to technological leadership in the development and production of high quality rubber compounds.

Thona Group produces a wide range of synthetic compounds for the automotive, construction, and other industrial markets. As one of the world's leading EPDM compounders, Thona supplies many of the top automotive and electrical appliance manufacturers. The Thona Group, as a major force in rubber compounding:

- Provides an unmatched portfolio of high quality compounds;
- Combines expertise in materials formulations with advanced technical production methods and technical service capability.

By focusing exclusively on compounding, Thona has taken a leading role in mixing innovation, introducing a high degree of automation and in-process quality assurance."

Trelleborg Industri AB, Sweden

Trelleborg is a global industrial group. Operations are based on spearhead competence within polymer materials and a high level of industrial know-how, combined with functional solutions and systems designed to meet the needs of our customers.
The Group has annual sales of SEK 19 billion (pro forma 2001, including recent acquisitions), with approximately 16,500 employees in 40 countries. Trelleborg consists of four business areas: Trelleborg Automotive, Trelleborg Wheel Systems, Trelleborg Engineered Systems and Trelleborg Building Systems. The Trelleborg share has been quoted on the A list of the OM Stockholm Exchange since 1964.
Through the work within MiniDerucom projects, Trelleborg mixing plants have gained quite interesting results, which are to be considered and implemented in our operations. Please find below some milestones.:

- Clearly, it is possible to reach a new, higher level of quality performance and conformity for rubber compounds when using real time process control systems.
- It has been shown that distribution and dispersion of ingredients can be measured with great accuracy in new ways, which will help to develop an ideal mixing process.
- Environmentally safer process oil can be used more widely, without disturbing technical performances.

The work has been done in a close cooperation between partners along a normal product flow chain - from raw material producers, research centers to industrial end users - which also has led to an useful and efficient cooperation basis for the future.

L'Isolante K-Flex S.r.L, Italy

Isolante was founded 1988 in Italy and developed over the years to be a specialist for insulation products and systems in the field of industrial and equipment insulation.
The turnover of ~ 50 Million Euro and the research and development results over the years puts Isolante on position two, worldwide, within its field.
Expanded rubber technology and its understanding and influence upon the end product quality is essential for the Company.
As a consequence the Mini Derucom project and its achievement improved the understanding of possible influence of Polymer characteristics during the production process steps and end product performance to a considerable degree.

Lusorol, Portugal

Lusorol was founded in 1992 as a daughter company of locksmith firm in Porto, which is in the north of Portugal. Lusorol is a family owned company having 20 employees and producing rubber covered rollers. The rollers are mainly used in the printing industry. Lusorol deals as well with inks and additives the demands of ist customers. The major advantage of Lusorol is their own mixing department and knowledge of rubber compound formulation. The annual turnover of the company is about 1,5 million €.

Bühler AG, Switzerland

Bühler is the global technology partner for efficient production systems, engineering solutions and the associated services in the food processing industry, in chemical process engineering and in die casting. The Bühler Group has committed itself to industrial progress, and has achieved global market leadership in many fields. With a payroll of about 6,600, Buhler is present in over 100 countries across the world and achieves net annual sales of about 900 million Euros. The Group spends substantially on research and development, tangible assets, human resources and customer training. Bulk materials handling belongs since a long time to the core competencies of Bühler. The mechanical and pneumatic handling technologies have not only been used to feed raw materials and products into the different process steps of the Bühler plants, but have also been successfully offered on the market. In the Rubber and Fine Chemicals market segment, many hundred conveyors have been commissioned. Highest reliability and lowest product degradation belong to the main characteristics of the Buhler conveying installations. By participating to the Mini Derucom program, it has been possible to show the positive impact of these two factors on the rubber quality and on the overall economy of the rubber mixing process.

Degussa AG, Germany

Degussa, founded 1873 in Frankfurt, Germany, is a company which offers a programme of big diversity to the market: It contains mainly semi-finished and intermediate products, which are used as special materials in practically all brnaches of the industry. In the rubber business, Degussa is heavily involved since it is the second largest producer of carbon black world wide. Also for silicas Degussa is one of the biggest suppliers in the world. Most of the products are being delivered to EEC and the rest of Europe – followed by markets in North America and Asia. Sales of the Deguissa group, which employs a work force of 63.000 worl wide, has reached 16.3 Billion Euro in 2000. The long and medium strategy of the company is to strengthen its already strong position in the field of fillers for the rubber industry. As problem with the processing of high active carbon black or new silica compounds arise by many important customers Degussa, improves the customer relationship by a more qualified support. In this sense the Mini Derucom achievements have led to very interesting aspects as a very detailed view into processing of carbon black compounds.

DSM Elastomers, The Netherlands

DSM Elastomers, with 700 employees, is the leading global producer of EPDM rubber and its derivatives (Keltan® and Petroleum Additives) with a total production ca-

pacity of 215,000 tpa, sourced from manufacturing facilities in the Netherlands, United States, Brazil and Japan. DSM Elastomers, is also a leading global manufacturer of EPDM-based Thermoplastic Elastomer (TPE) materials (also known as Thermoplastic Vulcanizates [TPVs] and Thermoplastic Rubbers [TPRs]) and provides solutions with a broad portfolio of Sarlink® TPRs.
DSM Elastomers is a business Group of DSM, a highly integrated group of companies with worldwide interests in life-science products, performance materials and chemicals. DSM recorded sales of EUR 8 billion (approximately US $7.2 billion) last year and employs a total workforce of some 21,500 distributed in more than 200 operations throughout the world.
Keltan & Sarlink are both used in a wide range of applications, including; automotive (e.g. hoses, weather strips and petroleum additives, building & construction (roofing, window gaskets), W&C, impact modification plastics (e.g. TPO’s) and as a raw material for TPV’s.

Volkswagen AG, Germany

Volkswagen is of the largest automobile manufacturer in Europe, offering products and services along the entire automotive value chain. With nine independent brands the Group is able to offer a unique range of models from the extremely efficient 3-litre car to the great sporting tradition of Bentley. While each of the brands has a distinct personality, it also benefits from its membership of the Volkswagen Group with its global manufacturing base and its international sales and marketing strength. The following brands belong to the Volkswagen group: Volkswagen-Nutzfahrzeuge, Audi, Bentley, Bugatti, Lamborghini, Rolls-Royce, Seat and Skoda. Volkswagen produces 5,1 million cars annually and employs about 322070 people. The company turnover is 88,5 billion € annually having a net profit of 2,9 billion € after tax. Volkswagen produces passenger cars and small commercial vehicles as well in 14 countries. As a global player, Volkswagen is represented in all significant markets of the world. In terms of market share in Western Europe, almost one car in five originates from the Volkswagen Group, while worldwide Volkswagen currently holds a position of 12.4% (as of 30th September 2001). The Volkswagen Group is also becoming a mobility provider, and its broad spectrum of services include financing and leasing business through its Financial Services Division. It also provides one of the leading information technology consultancies in Germany.

Institut für Kunststofftechnik (KTP), Universität Paderborn, Germany

The KTP is a quite big research group with about 20 researchers (Ph.D.) in the field of plastics and rubber processing in Paderborn. The work guided by 2 Professors is mainly focussed on the modelling of processes, i.e. rheology, scrwe design, die design and quality assurance, welding and extrusion of plastics and rubber. The working group of Prof. Potente is world famous concerning plastics extrusion simulation. The working group around Prof. Limper is quite experienced in rubber processing as he himself has more than 15 years experience in rubber research and within the rubber industry. Rubber technology in this group is of strategic importance. The current working fields in field of rubber are: Quality assurance concepts for the rubber mixing room, flow analysis, rotor and dust stop design for internal mixers, costing in the mixing room, process description for the extrusion process of rubber compounds for screw and gear pump extruders, the silanization of green tire compounds and continuous compounding, element distribution analysis in rubber compounds by laser analysis (RELMA) and rubber rheology.

Université de Pierre & Marie Curie Paris, Laboratoire de Rheologie et Mise en Œvre des Polymères, France (UPMC – LMROP)

UPMC is one of the component of the University of Paris and the largest french speaking university devoted to Sciences and Medicine. The LRMOP is a laboratory whose research activities concern the rheology and processing of complex polymer materials. Owing to an agreement between the University and the French Rubber Industrial Organisation (SNCP & IFOCA), the LRMOP has the position of “university-industry” laboratory. The working group around Professor Leblanc is studying, amongst other subjects, the rheology and processing of rubber compounds. He himself has gathered quite a large experience in this working field since, prior to his present position, he spent more than 15 years in the rubber industry: The long term strategy of his laboratory is to develop an advanced espertise in rubber rheometrical testing, rubber characterization and quality assurance. All the work performed at UPMC–LMROP is both of scientifical and practical interest, in such a manner that world-wide major rubber processors and suppliers are co-operating with the laboratory.

5 Technical Achievements

5.1 Mini Derucom Idea and Objectives

In comparison to plastics processing rubber processors have to be competent in a more complex way. The reason for this can be seen in the fact that the compounds their selves are not bought from one raw material supplier. Rubber processors only buy raw materials and mix their own „tailor made“ compounds for specific product needs.
Nowadays the rubber industry is exposed to an increasing economical pressure and to higher quality requirements at the same time. Following the chain of added value from raw material acceptance to end products one can define four influencing fields which have impact on quality: The raw materials and their quality parameter deviation range, the raw material storage, the compounding process and the processing of finished products.
Looking at raw material acceptance test procedures in the today's rubber processing industry it must be stated that not much work is done at all. The reason for this is that most of the suppliers are certified according to ISO 9000/9001 or similar standards. In addition, such testing would mean that one needs all the corresponding facilities and manpower due to the high number of ingredients. Furthermore one should realize that rubber compounds are rather complex mixtures: It is really difficult to get an overview of the meaning of all kind of raw material parameters in combination to their impact to the mixing process and the end article properties. Of course, mutual dependencies do also exist among the ingredients. Therefore, quality agreements are made between suppliers and processors. That's why companies have developed very capable experts for compounding but the knowledge about raw material parameter variations, the mixing process, as well as finished product properties is still incomplete.
A big problem of the rubber processing industry, which affects their competitiveness heavily, must be seen in the poor ability to preview finished product failures. Today, only 1% scrap is detected at the exit of mixing rooms with the standard testing methods available. On the other hand scrap rates at the end of the processing chain are around 10% reaching much higher peak values. It is common knowledge that a significant part of the scrap is related to problems in the mixing room, which could not be detected before. This problem is strengthened by the higher prices of the ingredients needed to produce rubber compounds. It can be estimated that the price of one kg rubber compound is factor two to three higher than in the plastics processing industry. Furthermore, decreased mixing times, which, of course are aimed, lead to additional problems because the time available to judge quality becomes shorter. Another special fact of rubber is the waste being usually impossible to be reused due to the cured material. All this means that scrap of rubber goods is very expensive.
It can be concluded that quality judgement in rubber mixing rooms is un-sufficient today and that there is an high economical potential if improvements are successful.

Consequently, it is the aim of the Mini Derucom project to develop measures, able to reduce scrap rates in the production process of technical rubber goods. Therefore, more knowledge about quality impacts in the rubber mixing room and their effects to finished product properties will be investigated. Furthermore, new testing devices will be applied and further developed in order to judge compound quality in a better way in comparison to standard testing methods. Hereby, processing problems and others will be foreseen in an earlier stage of the process. Finally, a quality assurance concept for the rubber processing industry will be developed. The project is divided in four tasks in order to reach the goal: In Task I, all kind of impacts coming from raw material quality parameter variations, raw material exchanges, raw material changes in the mixing room and mixing parameters have been investigated with respect to compound quality and the quality of finished products. Quality judgements have been done with standard testing equipment, which is commonly available at every rubber processor and such equipment used by raw material suppliers and end users like Volkswagen. The consortium was set up correspondingly in order to get access to the corresponding testing facilities of raw material suppliers and end users as well. Furthermore, the consortium should cover the broadest variety of products possible to allow a general view into the connection between the quality impacts mentioned before and the quality of finished products. Additionally, the judgement of compound/finished product quality was done with more sophisticated methods called RELMA and RPA. These methods have been further developed within the project as well in Task II (RELMA) and Task III (RPA). Additionally, the so-called TOPO method and the DIAS method were applied. All advanced methods will be briefly introduced in chapter 5.2.1 at first. The question of the predictive capabilities of testing equipment in general will be followed and answered in case of the studies done in the project. The ability of testing methods to judge the quality of finished products in an earlier state of the processing chain is very basic for the complete project and will be therefore discussed very detailed. Last but not least a complete process control model was aimed to be set up in Task IV, taking the results of the Tasks I-III into account. In this Task it was aimed to develop a model, able to describe quality impacts to technical rubber goods in a general way. The principle of CPC should be to correlate significant quality parameters of finished products with quality parameters of raw materials and process parameters. The basic idea of the CPC principle can be visualized as follows:

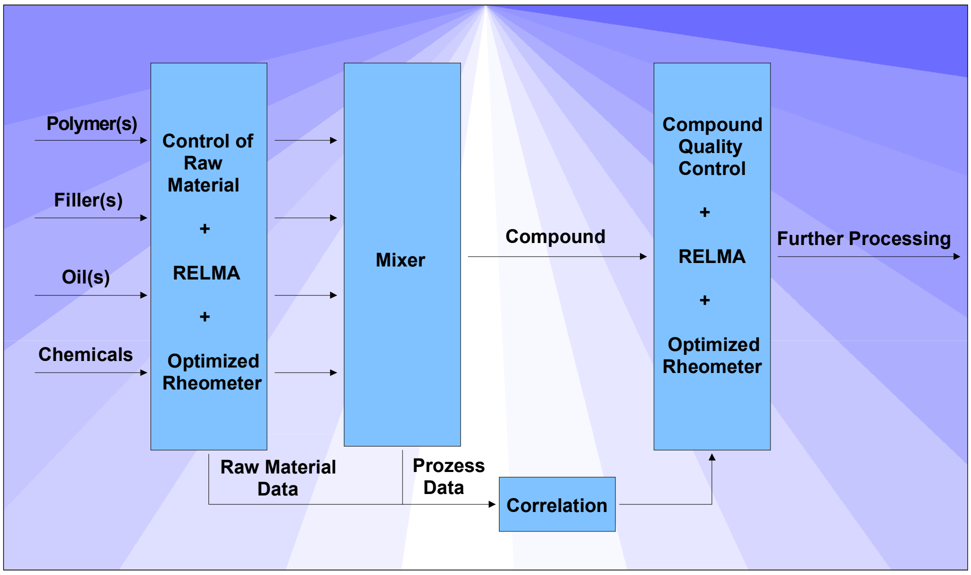

Figure 1: Principle of a controlled mixing room as a basis for a CPC model.

CPC was planned to consist of two major steps: In step one correlations between influencing factors and resulting properties of finished products would have to be learned by the system. In step two further quality measurements would be no more needed since the system calculates already quality based as a function of the measured material data and or processing data. For this, a broad database was needed representing the field of technical rubber goods. Task I provides the corresponding information. Task II and III were planned to provide more powerful quality control tools than commonly used today. The basic question of Task IV is if it is possible to transfer this data to different applications.

5.2 Main Achievements of the Mini Derucom Project

5.2.1 Mixing Room Study (Task I)

In Task I of the Mini Derucom project impacts on the quality of rubber products have been investigated. At first the so-called Mixing Room Study was done, revealing important raw material quality parameter variations, which are likely to influence the process and product quality. It was the objective of this work to detect those quality parameters of raw materials' ingredients, which are supposed to have an impact on processing behaviour in the mixing room and/or end article properties. In order to get an overview about specified raw material parameters, the quality routines at the processing partners of the Mini Derucom project were checked.
As said before, raw material suppliers are usually specified according to ISO 9000/9001 or similar standards. This means that rubber processors get raw materials, which are checked already. Such procedures do not have to be repeated. This would be even very difficult because processors would need to have all the corresponding testing facilities and handling knowledge. This is impossible in practice. Only in some cases production tests are being performed in order to obtain internal raw material releases for production processes. Sometimes, special product needs require a very narrow range of special raw material properties, which are usually not placed at disposal by the suppliers. In this case special agreements are made and processors are checking the agreements by the production tests mentioned before. It can be concluded that in most cases not much work is done in terms of raw material quality control at the entrance of a mixing room. Raw material quality control is mainly the work of raw material suppliers. The job of processors is usually reduced to the check of the actual raw material parameter values in certificates with respect to specification limits. The question is of course if raw material suppliers are providing the information really needed. Furthermore, there are no general rules about setting the specification limits. Sometimes, there are agreements between processors and suppliers. On the other hand it can be assumed that suppliers are fixing the limits according to their production accuracy in many cases.
For that reason, an high number of different raw material certificates were checked at the Mini Derucom processing partners in order to analyse raw material parameter deviations in practice. Of course it was impossible to take all ingredients of the 10 recipes into account investigated in the project. The work was focussed on the quantitatively most important raw materials. It must be noticed that every ingredient is specified with a number of quality parameters. If, for example, polymers are regarded, the kind of specified quality parameters depend on the type of polymer. The same is true for other ingredients. Sometimes, different suppliers don't even provide corresponding information for the same raw material. The most important ingredients of compounds are of course polymers. Carbon blacks and plasticizers must be named in many cases, too. At the processing partners, a high number of raw materials deliveries were analysed and the respective quality parameters of the ingredients mentioned before were edited. The objective of this procedure was to study the scattering range of quality parameters in order to estimate the most important ones.

5.2.2 Parameter Analysis Study (Task I)

If quality parameters were found varying significantly, the effects of these parameters were investigated with respect to the compound- and finished product properties of different applications. The investigations were done with recipes placed at disposal by the processing partners of the Mini Derucom project. Furthermore, the mixing experiments were carried out in the mixing rooms of the processing partners. This study was called “Parameter Analysis Study“. The compound properties and the finished product properties were performed with standard methods usually applied in the rubber mixing room and the more sophisticated analysis tools RELMA, RPA, TOPO, DIAS and a so-called Online Quality Assurance Tool. These methods will be briefly introduced below. The results of the studies can be summarized as follows:

It was found that most quality parameters specified in raw materials’ certificates do not vary significantly. This would mean that the production processes of raw materials are very constant. No rubber processor would have to care about raw materials’ quality parameters at all and quality variations of compounds and/or end articles can’t be traced back to raw materials. Of course this is unrealistic. The mixing room study clarified that there are quality parameters, which are not specified exactly today or which don’t provide full information.

This is for example true in case of the quality parameter long chain branching of EPDM polymers, which is caused by the complex and time consuming measurement method. This parameter was found to influence the mixing process. On the other hand the properties of finished products were found almost unaffected in case of the recipes investigated, which means that an exchange of the competitive polymers can be done, irrespective of the branching level. Obviously, the finished product properties are predominantly set by the recipe composition. Furthermore, processing aids are obviously being added to polymers in some cases, which can influence the mixing process significantly as well. It can be even said that the processing aids are influencing processing behavior to an higher extend than the quality parameter long chain branching. On the other hand it looks like such processing aids are applied in order to compensate the influence of long chain branching during processing itself. Today, such chemicals are not specified exactly neither. Both quality parameters discussed can be named as a gap in todays’ quality routines. Furthermore, it must be pointed out that the possible exchange mentioned before is only true in case of the recipes investigated. The situation can be different if other compounds/applications are regarded.

Other quality parameters like the fines content of carbon blacks are looking unproblematical in certificates. The fines content in the condition of delivery is rather low and the variation range is small. But, the fines content can change dramatically in the mixing room, due to the applied conveying system and its settings. The fines content must be known in the hopper above the mixer because the fines content can influence the mixing process and the property level of compounds significantly. The height of the first power maximum was reduced, meaning that the process of pellet destruction changes, the wall slip phase is more pronounced at increasing fines content and the BIT extends. As a consequence, the dispersion of carbon black and sulphur was influenced. It must be recognized that there is another gap in the quality routines in the mixing room. It is to be recommended to keep the fines content during conveying as low as possible in order to exclude quality variations.

The third quality parameter, which was investigated deeply in the Mini Derucom project, is the pellet hardness of carbon blacks. It was found that there is a significant scattering range of the individual pellet hardness within an adjustment of the mean pellet hardness. The scattering range increases with increasing mean pellet hard-

ness. The studies showed that the influence of the pellet hardness to the process and the end article properties depends on the structure of the carbon black. Hard pellets of medium structured blacks can only cause problems in case of low raw polymer viscosities and compound viscosities at the same time. Soft pellets of these blacks were found to be problematical in case of high filled compounds because then the wall slip phenomenon in the mixing process becomes more pronounced. If high structured blacks are regarded, the influence of the pellet hardness is pronounced to an even higher extend. Even low filled compounds of high compound viscosity levels lead to worsened compound- as well as end article properties. Furthermore, the applied mixing equipment seems to be an important parameter. An intermeshing rotor geometry ends up with better results than the tangential rotor geometry investigated and in case of the recipe regarded. In general, soft pellets were found to disintegrate easily, which means that fine material forms. Consequently, the fines of the soft pellets act like investigated in the studies of the fines content, meaning that wall slip phenomenon in the mixer increases. It can be concluded that the influence of the carbon black pellet hardness to product quality depends of a huge number of factors like the carbon black structure, the compound viscosity, the recipe composition, filler content and the mixing equipment.
Mutual dependences among raw material parameters were also investigated. The fines content of carbon blacks was found to influence the process to an higher extend in case of highly branched polymers. Low branching levels and low polymer viscosities favor incorporation of fillers and the effects are lessened. Furthermore, processing aids like e.g. zinc-stearate were found acting positive if high amounts of fines are applied. In this case, the carbon black dispersion improves significantly at increasing fines content.

Beside the Parameter Analysis Study, further investigations have been done in Task I covering the field of polymer exchanges at constant raw material specifications, raw material changes like e.g. caused by pneumatical conveying processes and the field of mixing parameters. Some of the results will be very briefly summarized as follows: The exchange of polymers at constant specification is sometimes not possible. If closed cell insulation materials are regarded, which are produced in a continuous line, require a narrower range of polymer viscosity than commonly supplied. If the usual range is run in the production, the product ends up too soft. Other applications like sleeves have shown bagging effects if the original polymer was exchanged to others of similar specification. If the compound was wrapped around a mandrel, the material tended to flow under impact of the gravity forces as a function of the polymer applied. It could be shown that the bagging effect is a very slow relaxation process, which is likely due to different branching levels of those polymers. The effect could be forecastet with RPA tests. In this subtask it was also shown that an exchange of highly aromatic mineral oils in a critical NR/BR based recipe used for shock absorbers is possible. Pneumatic conveying systems have been investigated with respect to the increase in fines content of the carbon black during the conveyance process and to the adhesion behaviour of different carbon blacks. Steel piepes and rubber pipes, dense phase systems as well as dilute phase systems have been taken into account. It was found that the increase in fines depends on the conveying principle, the type of carbon black and the pipe material. The lowest increase in fines can be achieved with dense phase systems however. Furthermore, low structured carbon blacks were found to stick to the pipes' walls. This effect can only be avoided if rubber pipes are applied because a pumping effect occurs, able to disconnect stuck material. In this subtask also investigations of weighing tolerances have been done in case of steering power hoses. The content of oil and carbon black was varied in steps of ±10%. Though the compound properties indicated the variations very clearly (Mooney, MDR, mechanical tests), the finished hoses were found far beyond the lower specification limits irrespective of the applied variation. This kind of article obviously contains big safety margins. In the subtask mixing parameters the mixing control system MixCont was checked and old mixing technology was compared to modern mixing technology. It was found that the MixContsystem was able to equalize raw material parameter variations (degree of long chain branching in case an EPDM based brake membrane recipe) and that there is huge economical potential if modern mixing equipment is used in the rubber mixing room. Furthermore it was investigated that intermeshing and tangential rotor geometries are reacting completely different in case of applied raw material parameter variations (EPDM based sealing recipe with varied degree of long chain branching). More detailed information can be obtained in [1].

5.2.3 Introduction of New Testing Methods

RELMA (Task II)

The RELMA unit was developed in a finished BRITE project (BE 3102-89). RELMA stands for **RE**mote **L**aser **M**icro **A**nalysis. This unit can be described in a short way as follows: A pulsed laser beam produces a plasma on the surface of a sample. The material is ablated from the surface and enters a plasma cloud. The electrons of the outer orbits are brought to a higher energy level, which is an unstable condition. Going back to a lower energy level, specific radiation is emitted which can be attributed to chemical elements. This radiation can be detected with spectroscopic methods. As the measuring time is quite short, the system can work with a frequency of 25 Hz. By scanning the surface, the content of chemical elements at each measured spot can be indicated. By statistical methods the distribution of the elements in the surface can be determined [2, 3]. The RELMA unit was further developed within the Mini Derucom project, too. The main work contents have been: a) installing of a new and more powerful laser tube, b) installing of a mirror system, which reduces losses at low wave length, c) installing of a new spectrometer having variable gratings and a 3-dimensional resolution, d) installing of a new software, allowing an easier handling of the RELMA unit, e) development of RELMA to be used as a raw material characterization tool.

RPA (Task III)

The **R**ubber **P**rocessor **A**nalyzer (RPA) is a further development of the well known **M**oving **D**ie **R**heometer (MDR), which is commonly available at almost every rubber processor as a standard testing device. In comparison to a normal MDR, frequency and temperature sweeps can be derived and the strain amplitude can be varied. About 450 samples have been tested with the RPA in the Mini Derucom project and the work content of the RPA Task was as follows: a) development of standard testing protocols, b) repeatability tests, c) analysis of temperature fields in the RPA cavity in order to investigate non-isothermal effects by FEM calculations and measuremenets in the cavity, d) development of a software, able to qualify and quantify the non-linearity of rubber compounds (on basis of a Fast Fourier Transformation routine, analysing the odd harmonics of the response signal), e) definition of processability indicators, allowing a simple data reduction of the RPA while providing a better distinction of the rheological characteristics of rubber compunds. For further information we would like to refer to the literature [1, 4].

TOPO

A method to determine the dispersion of fillers was developed at the company Degussa AG, using a very small needle. This method is called TOPO and was used in the Mini Derucom project to determine mainly the carbon black dispersion. A small needle scans the surface of a sample and creates the topography of a samples‘ surfaces roughness. High values of roughness mean bad dispersion and vice versa. In [1, 5] it was proven that un-dispersed carbon black particles generate holes and elevations when these particles are hit by knife while the sample is cut. This means that a deflection of the needle arises at exactly the same place because carbon black agglomerate was torn out of one half of the cut sample and remains in the other half. A big advantage of this method is e.g. the independency of different users.

DIAS

Optical methods like the DIAS method developed at the **D**eutsches **I**nstitut für **K**autschuktechnologie (DIK) [6] was also used to characterize the dispersion quality of compounds within the Mini Derucom project. The method needs to be calibrated with a grey scale, which depends of the type of compound and the operator. Using a razor blade, the vulcanizates are cut into thin pieces, which then have a glossy appearance. These test pieces are investigated under an optical microscope. The resulting photos having a wide scale of grey tints are then reduced to black and white. The black areas represent the filler, the white area the background. The dispersion coefficient can be calculated on basis of the size of the black and white areas, consisting of usually on 40 different photos. For the calculation of the dispersion coefficient the filler volume and a correction with the Medalia factor are taken into account. A semi-automatic analysis can be done with a CCD camera system and software able to count the black and white areas.

QS Extruder

During the first period of the Mini Derucom project investigations were focused on the analysis of raw material parameter variations and their influence to product quality. In some investigations no correlation between raw material quality parameter variations and rheological as well as mechanical compound properties could have been noticed. On the other hand, the surface quality of thin extruded stripes was observed to depend on the varied parameters. Such surface defects are not accepted on visible automotive applications like "sealing - systems". Today, sealing system profiles are “multi material products” (sponge, dense, metal, flock, etc.) produced on modern extrusion lines with high output rates. Therefore, an excellent and constant compound quality is needed to keep the scrap level as low as possible. Up to now, quality defects due to poor compound quality can only be detected after the production of the finished product itself, which is of course at the end of the production chain. A general rule, which was fixed by the automotive industry, says that up to two defects of certain size per meter profile are acceptable. More than these two defects will lead to a sort out of the sealing system profile. This is of course very expensive.

To assure a certain quality level of the supplied compounds it was decided to develop a new testing device, able to qualify and quantify such surface defects. The basic idea of the system is to combine the judgment of the surface quality with the measurement of the shear and strain viscosity of the compound. This idea seems likely because the compound must be extruded anyway to attain the thin stripe. Consequently, different quality information can be gained at the same time. The most important requirements of the device were defined as follows: 1. The system has to work online (implemented in the mixing line); 2. The results have to be independent from the operator; 3. The device must be robust enough to resist rough production environment; 4. Batch-to-batch variations have to be visualized; 5. A classification of the compound should be possible. In fact this new quality assurance tool was developed in the Mini Derucom project due to the impossibility to characterize poor compound quality, which leads to surface defects and scrap at the same time. The new device consists of an optical tool able to qualify and quantify surface defects very accurately and a rheological analysis tool, able to characterize the flow properties of a compound. It was aimed to determine poor compound quality in this manner at the exit of a mixing room, realizing a 100% control of the produced batches. With the help of this new equipment rubber processors are able to detect quality lacks before supplying the compounds to the extrusion departments and they are also able to classify the compounds. The new instrument provides useful quality parameters additional to the conventional test devices commonly used in the rubber industry. No additional work has to be done by operators if the system is installed as an online tool. In case of running out of specified limits, the operator can accomplish necessary actions to interfere into the mixing process. For further information we would like to refer to the literature [1].

5.2.4 Predictive Capabilities of Testing Equipment

A general problem also became obvious in the studies. It was found that the compound properties did not preview variations of the product quality in most cases. Especially the standard testing methods did not provide a clear picture in case of the Mini Derucom investigations. Only the more sophisticated methods revealed such variations. Such testing devices are RELMA, the RPA, the TOPO method, the DIAS method and the new QS extruder. All methods have been used additionally to standard testing methods to judge the quality of the compounds. The predictive capabilities of the testing methods applied in the Mini Derucom project will be summarized as follows:

Predictive Capabilities RPA

The RPA can be used for automated and routine testing procedures on uncured rubber compounds in the mixing room. Whatever the nature and composition of the material, simple test procedures give repeatable results providing some care is taken, e.g. clean testing cavity, constant sample volume, regular calibration of the instrument. Such test procedures can be operated through the built-in testing programming capabilities of the instrument. In the project it was found that, by combining frequency and temperature sweeps, the RPA provides a set of data that can be treated in order to yield a master-curve at a reference temperature. From this master-curve, a dynamic viscosity function is derived and the ratio of η' at two frequencies is calculated in order to obtain so-called "processability indicators" PI. By analysing Mini-Derucom results on a wide variety of rubber compounds, it was observed that the processability indicators are more sensitive to processing variations than the standard Mooney ML (1+4), considered as a standard test in the mixing room. At the time being, it would be excessive to assign predictive capabilities to processability indicators, because it concerns only the sensitivity of the material to shear rate variation. However, it might be expected that series of successive batches of the same material, tested at the same point on the production line and always exhibiting the same PI, will behave in a similar manner during the subsequent steps of the process.

Predictive Capabilities RELMA

The situation is different in case of the RELMA unit. RELMA provides information about the dispersion of chemical elements, and here predominantly for metal elements in good quality, and the relative concentration of elements in all kind of rubber based material. If the absolute content of an element is required, a calibration must be done.

The dispersion of chemical elements is the basic operation of the mixing process and if it assumed that mixing is the key process in the processing of rubber based products as said above, RELMA must play a decisive role. Furthermore, RELMA can provide information in a macroscopic scale, meaning that large area of a sample can be analyzed quickly. This offers different applications for RELMA:

First of all, RELMA is able to characterize raw materials with respect to pollutions. In fact small chemicals, which are included to raw materials and which are not specified today, can affect the mixing process significantly. Consequently, poor product quality can result. Tracing of such ingredients offers a good chance to be very sure about the ingredients used in the processing line. If the RELMA compound characterization ability is regarded, it can be concluded that the unit works well. It is possible for the first time to measure the dispersion and the content of a big variety of chemical elements like sulphur and zinc accurately and in a reasonable time span. For example, RELMA can be used to check the feeding accuracy of small ingredients like those of the curing system when compounds are mixed. If e.g. sulphur or zinc has been left out during weighing, the changed relative element concentration would indicate the lack of those ingredients. Sulphur is one of the key ingredients of rubber compounds as long as sulphur cured recipes are considered. It can be said that if sulphur is dispersed well and the recipe formulation was set up right, appropriate information about the curing behaviour of a respective compound can be obtained with RELMA. If the sulphur or zinc dispersion will be found on a poor level for example, it can be directly concluded that mixing problems exists if it is assumed that the content of these ingredients is within specification. In fact the results of RELMA with respect to sulphur dispersion and the MDR results were found corresponding to each other very well in many investigations of the project. RELMA was even found more reliable in some cases and it provides direct information allowing to trace the origin of the effect. Furthermore, RELMA was also found in line with the dispersion quality of carbon black. If the carbon black dispersion is low leveled due to high carbon black fines content for example, RELMA indicated dispersion problems of sulphur, too. It is therefore imaginable that one would need only one method to characterize the basic result of the mixing process: providing sufficient dispersion of all kind of ingredients of the recipe. One would have to choose one ingredient for compound characterization with RELMA, which is known to end up with dispersion problems. The most complicated situation is surely exhibited if recipes contain this ingredient in small amount. Sulphur would meet these requirements well. A further advantage of RELMA is its ability to be used as a tool for compound development as well as for process development. Direct information about the dispersion of chemicals can be attributed to product quality and it can be checked, which requirements special products really must have.

Restrictions of RELMA must be seen in the resolution of the method. RELMA was found very sensitive in case of sulphur measurements because only a few phr of this

ingredient is usually added into recipes. On the other hand the burned spot of RELMA on the surface of the sample has a minimum extension of 1.7 times 0.3 mm, which is fixed optically by the mirror system. This is the reason for the good dispersion quality of ingredients, which are added into the compound in large amounts. Solutions can be surely obtained if the interplay between the size of the burned spot and the provided laser energy are improved. Another restriction of RELMA is its inability to characterize halogens. Therefore, RELMA was almost not able to support quality control in case of compounds, which are used to produce insulation materials containing a closed cell structure for example. Those compounds contain halogen based blowing agents, which must be dispersed very well in order to enable a well-adjusted interaction between the blowing reaction and the course of curing reaction during vulcanization. It would be important to have information about its degree of dispersion in such cases but this is not possible with RELMA. It was also noticed in the project while determing the sulphur dispersion of special compounds that its dispersion can improve along the processing chain and especially during vulcanization if the sulphur is applied in a soluble condition. In such a case of course it would make less sense to have knowledge about the sulphur dispersion in the compound.
It must be concluded that RELMA is a very promising tool to be applied for quality control in the mixing room. At the time being, RELMA is already very helpful to develop processes and to indicate the dispersion quality of chemicals like sulphur, which are added to recipes in small amounts. On the other hand still some work must be spend in order to develop RELMA to be an economical quality assurance tool in the mixing room.

Predictive Capabilities TOPO and DIAS

The TOPO method was found to be inline with RELMA in many cases. Furthermore, the results of the TOPO method are inline with those of the DIAS method [1, 5]. The TOPO method, which is characterized by an high resolution (2 μm square diameter of the tips' needle), does not distinguish between dispersion of carbon black and other fillers or even curing chemicals like sulphur. The roughness of a compound sample is simply measured. Of course this meets the basic requirement of the mixing process as well – providing sufficiently dispersed ingredients. The question is, if there do really remain agglomerates of e.g. sulphur in a sample. It can be assumed that the dispersion of sulphur is a problem that is related to variations of the concentration, which can be determined with RELMA far better due to large-scale analysis. Therefore, TOPO will be mainly used for characterization of filler dispersion. Carbon black agglomerates have to be dispersed during the mixing process as discussed in the studies focusing the pellet hardness. It can be even assumed that the dispersion of carbon black is a much bigger problem than the dispersion of white "extender" fillers like chalk or others, which are not pelleted for handling reasons. The main advantage of the TOPO method in comparison to others is its independence of the content of white fillers. This is for example a problem if the optical DIAS method is regarded, which, in principle, provides the same information as TOPO with good correlation, but is influenced significantly by the content of white fillers. Due to a grey scale necessary to be chosen, different rate of light reflection influences the result as a function of the white filler content. In this sense TOPO shows a clear advantage against DIAS. On the other hand, restrictions of the TOPO as well as the DIAS method to be established as a quality control tool in the mixing room must be named as follows: It is rather difficult to prepare samples and the measuring time is quite long. At the company Degussa, measurements are done over night, using a set of samples then being tested one after another. It can be concluded that the TOPO method is a very good method to be used in lab scale but its applicability on the factory floor is not useful at present time.

Predictive Capabilities QS Extruder

In contrast to TOPO and DIAS, the new optical method overcomes the problem of sample preparation. A thin strip is being extruded, which can be done online. The surface quality of the strip is characterized with a CCD camera system and a software analysis tool very accurately online, too. This method was found more sensitive against variations of e.g. the carbon black quality than any other method regarded in the project. The sensitivity of the system is of such height that a modified carbon black, which shows exactly the same specified quality parameters in the specification, was judged to be different with this tool. Furthermore, this kind of quality tool can also be used to characterize rheological compound properties. It is possible to measure the shear and strain viscosity with the system at the same time. The key task of mixing, which is providing sufficient dispersion and distribution of ingredients within the compound, can be controlled with this type of device. Therefore, this method is the most promising tool to ensure quality in the mixing room.

Predictive Capabilities Mixer

In contrast to standard testing devices and even the more advanced testing methods discussed before, the mixer was found very sensitive against almost any quality impact. It must be pointed out once more that predominantly raw material quality parameter variations have been investigated within the Mini Derucom project. The power curve of the mixer indicated these parameter variations very well at least in those cases, when the variation was transferred to the quality of respective finished products. It can even be said that the mixer only showed non-distinguashable power curves in cases where no influence could have been observed whether on compound nor on product quality. Consequently, the mixer can be used like a sensor, indicating variations of any kind, which occur before the mixer in the mixing room like in case of raw material quality parameter variations for example. This, of course, only works if the mixing procedure is kept absolutely constant. If the mixing process is controlled by any routines, the sensor ability will be lost.

Conclusions Testing Strategy

The results can be briefly summarized with respect to predictive capabilities of finished products as follows: There is no general rule, which favours a special testing device to be the one and only. The connection between product properties and quality influencing factors depends strongly on the finished product itself, its production process and the type of raw materials used. If for example sulphur is used as soluble material its dispersion can be improved significantly during the vulcanization process when imposed to curing temperatures. Other examples have shown that finished products have rather high safety margins like investigated in case of power steering hoses. In this case, the more advanced testing methods RELMA, RPA, and TOPO were not helpful in order to provide more distinctive quality information. Sometimes, even no correlation could have been found at all, meaning that no testing method was able to predict poor product quality. In other cases, the compound quality was found on such a poor level, though the finished products showed excellent properties. Consequently, a general forecast of product properties by testing equipment is impossible.

However, it can be said that standard testing methods, which are indirect methods in most cases, provide less reliable information about finished product quality. The more sophisticated methods RPA, RELMA, TOPO and QS extruder were found to be more distinctive as shown on many examples of the Mini Derucom project. Furthermore, the mixer was established to be a very sensitive machine, able to indicate all kind of significant raw material quality parameter variations. Consequently the mixer as the heart of the mixing room can be used as a sensor. The efforts, which must be spend on such a machine like installing corresponding sensors and a computer (if not yet available) are very low if compared to the effort of testing, which must be done afterwards to trace failures.

The qestion is then of course which strategy should be applied in the rubber mixing room to ensure product quality respectively to have appropriate quality information in hand as early as possible in the processing chain. If the production process of rubber compounds is subdivided, the following steps can be named, which are responsible for the attainable compound quality. Further processing and the quality of the finished products will be excluded at first.

- Recipe composition.
- Raw material quality.
- Weighing accuracy.
- Mixing Quality or quality of the dispersing compound matrix:

The main requirement of sufficient finished products quality is the composition of the recipe, which is being developed to match special end article requirements. On this basis, the type of polymer(s), filler(s), plasticizers and chemicals are chosen. This key requirement, of course, can't be taken into account in terms of predictive capabilities of testing devices.
Raw materials are specified in certificates and quality parameters have to provide such information that allows sufficient characterization. The variation range of raw material quality parameters permitted must be known with respect to respective finished product quality. Predominantly raw material suppliers must minimize this kind of quality impacts. If there is a lack of quality information in raw material certificates, research work like accomplished in the Mini Derucom project should be done to clarify the situation.
Weighing procedures were found very accurate in different mixing rooms. However, it must be ensured that all ingredients are included to the batch. There are a number of techniques available on the market for this job using e.g. barecode scanners, which are to be recommended.
If the mixer is regarded it must be repeated that its sensitiveness should be used to detect defaults of the steps before. Therefore, the mixing procedure must be kept constant. It will be assumed of course that the recipe was developed appropriate according to the needs of the product. As found in the Mini Derucom project, raw material quality parameter variations and weighing variations can be detected on basis of the power curve of the mixer. It can be furthermore posited that all kind of finished product properties will turn out best if sufficient dispersion quality of the ingredients within the compound is being provided. The mixing process including the roller mill has to fulfill this job and can be regarded as the key process in the mixing room. Herewith, the link to the finished product will be achieved because it can be assumed that the compound quality will not turn worse in following processing steps.

It can be consequently concluded, that testing methods, being able to forecast finished product quality, simply have to fulfill the requirement of providing sufficient mixing/dispersion quality of the ingredients. This means, it would be already sufficient to ensure good dispersion of all kind of ingredients in the recipe directly after the mixing process respectively behind the mill.

If finally a minimum testing strategy will be defined for the rubber mixing room, first of all the sensor ability of the mixer must be used to detect raw material quality parameter variations and weighing defaults and a method must be selected, able to provide reliable information about the dispersion quality of the rubber compounds. The most promising method in this manner according to the experience gained in the Mini Derucom project is the new QS extruder. The method can be applied online and to the greatest possible extend automated. The method was found sensitiv enough to reveal carbon black quality differences in case of slightly modified material. It must be pointed out that the specified quality parameters of the carbon black stayed the same. Furthermore, the method is able to characterize rheological compound properties at the same time because the compound must be extruded anyway. Last but not least a method is needed that allows to characterize the curing behaviour of a compound. For this, whether an MDR or an RPA can be used. The RPA of course provides rheological information very accurately as well and it must be kept open here, if the Online Tool is able to replace the content of information of the RPA. Such a decision will surely depend on the complexity of the respective compound in terms of processing behaviour. Therefore, a minimum testing strategy depends on the type of application as well. It will be reminded that there are also applications, which seem to have big safety margins that don’t require highly sophisticated testing facilities. In such cases, the standard testing devices Mooney and MDR will be almost sufficient. However, the following testing strategies will be suggested as most senseful on basis of the results of the Mini Derucom project:

- Mixing Data – QS extruder – RPA
  (highly sophisticated applications)
- Mixing Data – QS extruder – MDR
  (normal applications)
- Mixing Data – Mooney Device – MDR
  (un-sensitive applications)

It must be noticed that the standard testing methods did not show a clear picture of the situation at all. Their ability to predict poor properties of finished products in case of quality impacts in the mixing room failed in almost every investigation of the Mini Derucom project. There are several effects able to explain the situation: At first, only a small amount of compound is tested. It needs no further comments to state that the bigger the mixer and the poorer the technical state-of-the-art of the equipment used on factory floor, the poorer the compound homogeneity in terms of dispersive and distributive mixing capability is to be expected. Consequently, it is of statistical nature to find the “right” sample able to characterize the compound properties “right”. The second reason might be seen in the quality of preparing samples, which is sometimes difficult and needs to be done very accurately. In the Mini Derucom project major differences of standard testing results were found in different laboratories, which can only be explained with sample preparation because testing procedures are all specified in norms. Herewith, it must be questioned if it makes sense to apply todays’ standard testing devices with the exception of very unsensitive compounds, due e.g.

to big safety margins. Those devices can only provide information in case of bigger quality deviations. A more general reason for the results described before, can be seen in the fact that the raw materials have a “statistical” nature, which are handled through processes that are also “statistical”. It follows that whatever the complexity of a test on the material is at a given point of the process, the results of testing procedures have very limited predictive capabilities.

5.3 Quality Assurance Concept for the Mixing Room

5.3.1 General Conclusions Mini Derucom

Regarding the results of the Mini Derucom project it can also be concluded that the composition of recipes seems to play a key role in terms of the quality of finished products. Specially required product properties are likely to be set essentially by the recipe. Furthermore, it seems to be possible to adjust the recipes to the processes established on the factory floor. If the field of raw material parameter variations is regarded, it was found that there are some parameters which can affect the properties of finished products though it is sometimes impossible to detect the variation with any kind of testing device. If such a variation is actually transferred to a finished products additionally depends on the application itself, the mixing equipment, the mixing procedure and many other influencing factors, which surely have not been able focussed within the project. It becomes very complicated if there are raw material quality parameters which are not yet specified exactly and which can’t be traced due to unsensitive testing devices available. Hence it is impossible to notice such variations. The complexity is even enhanced if it is taken into account that raw material quality parameters also show mutual dependencies among each other. It can be concluded in a more general way that all influencing factors on product quality depend on each other. Therefore, it can be deduced that it makes no sense to derive models able to describe the quality of finished products in a generalized way. Such models would only allow the prediction of finished product quality in case of a single recipe in combination with the production equipment applied on the factory floor. It must be even questioned if such a procedure would make sense in every case because if there are major safety margins provided for example, the prediction of finished product quality would be senseless. If such models are derived for single processes, where it is supposed to make sense, the kind of model must be selected. For this, statistical models, cluster analysis, artificial neuronal networks or expert knowledge can be used.

The question of how to predict finished product quality in order to reduce scrap rates during rubber processing might be also turned around: It must be demanded if many recipes are over-engineered today in order to meet a specifically required quality. It looks like such procedures are necessary to attain a secure process due to raw material parameter variations, mixing parameter variations, the kind of mixing equipment used on factory floors, safety margins and so on. If there are for example raw material parameter variations, the process must be run accordingly in order to overcome such influences, if this will be possible at all. If furthermore poor technical equipment is applied in the rubber mixing room at the same time the risk of failures increases and the process becomes less economical. It can be said that the number of weak points in the mixing room in terms of raw materials and equipment are adding up, necessitating an even more un-sensitive development of recipes to meet required product properties. It is a kind of reinforcing effect. It is then no longer surprising that it is a very tough job to control product quality already in the mixing room.

On basis of these statements, it must be concluded that the only real chance to discover economical potentials in the processing chain, which will most probably allow to reduce scrap rates and to ease over-engineered recipes, is based on three steps:

The first step is the field of raw material parameter variations. Quality parameters of raw materials have to be treated in a database, which will lead to expert knowledge. In general, raw material quality parameters are specified sufficiently in raw materials' certificates but there are some weak points in the quality assurance system leading to a lack of information like shown in this project. These weak points must be discovered and the corresponding quality parameters must be investigated with respect to their impacts to the quality of finished products on basis of a broad application range. The strategy to do so was clearly demonstrated in the Mini Derucom project. However, it is to be assumed that only some important quality impacts of raw materials have been able focused in the project, due to the complexity of rubber formulations in general and the high number of different applications.
The second step is to recommend such equipment in the mixing room being judged as efficient and reliable in the Mini Derucom project. Herewith, stable processes are supposed to be obtained. Dense phase conveying systems, the latest rotor geometry of mixers providing best cooling efficiency possible, an hydraulic activated ram, and an advanced mixer control system like MixCont can be named exemplarily to ensure stable processes.
The third step is the predictive capability of testing devices. It was shown in the Mini Derucom project that such capabilities are very limited if standard testing devices are regarded, which are commonly applied in mixing rooms today. New devices have been found more distinctive. However, there is no testing device covering all actual and very special needs of every kind of compound. This was proven by the fact that even RELMA and the advanced RPA are not able to provide additional information in every case. As far as dispersion problems are concerned ending up in poor surface quality of corresponding applications, which for example can raise from carbon black quality problems like a varying pellet hardness, a new testing device was developed. This device can be seen as a very promising tool to detect quality problems of compounds.

5.3.2 Quality Assurance Concept for the Rubber Mixing Room

As discussed before, different steps can be named to ensure quality in the rubber mixing room, which take finished product quality into account. These steps are as follows in a more distinctive way:

- Recipe composition,
- Raw material quality,
- Weighing accuracy and raw material handling,
- Mixing quality,
- Testing strategy.

Recipe Composition

The main requirement of sufficient end article quality is the composition of the recipe, which is being developed to meet special end article properties. On this basis, the type of polymer(s), filler(s), plasticizers and chemicals are being chosen. This key requirement, of course, can't be taken into account in terms of quality control strategies. However, it is to be recommended to develop recipes as easy as possible and to use the same ingredients in different recipes as far as possible within one factory or one company. Hereby, the total number of ingredients in a rubber-processing factory will be reduced and the complexity of the effects of different raw material quality parameters to the process as well as the end article quality decreases.

Raw Material Quality

Raw materials are specified in certificates and quality parameters have to provide such information that allow sufficient characterization. The variation range of raw material quality parameters permitted must be known with respect to respective product quality. As discussed before, the interdependencies are very complex. Predominantly raw material suppliers must minimize this kind of quality impacts. However, it is to be recommended to think carefully about possible impacts of raw material quality parameters to ones products and to make corresponding agreements about specification limits with suppliers. All information gained in the Mini Derucom project and information commonly available should be fixed in an expert system. If there is a lack of quality information in raw material certificates, research work like accomplished in the Mini Derucom project must be done to clarify the situation. This will probably be different from processor to processor. On basis of the Mini Derucom results, the polymer parameter long chain branching should be specified exactly because the processing behaviour can be influenced. Furthermore, every kind of ingredient added to the polymer should either be specified in certificates or exact information should be placed at disposal elsewhere, e.g. in the internet. If the carbon black quality parameter pellet hardness is regarded, it is to be recommended to order medium hard pellets. This quality parameter was found deviating at most if compared to the variation range of other raw material quality parameters. Obviously, the process of pelleting seems to be rather difficult. Therefore, this parameter must be observed carefully during raw material acceptance procedures. The best quality indicator is the maximum pellet hardness, which should be specified. The fines content is almost unproblematical if today's specification limit of 7% is not exceeded. The fines content firstly rises during pneumatic conveying processes or similar. Consequently, the effects arising of too high fines content must be seen as a problem of machinery equipment.

Weighing and Material Handling

Weighing procedures were found very accurate in different mixing rooms. However, it must be ensured that all ingredients are included to the batch. There are a number of techniques available on the market, which are able to fulfill this job and which are based on bar code systems. It was also found that dry-blends of small ingredients could even improve the weighing accuracy. For this a small dry-blend mixer is needed. The question if it makes sense to install such a system is directly linked to the number of different recipes and their respective output in a factory.
Storing and handling of materials was investigated in case of pneumatic conveying processes. Today, such systems are most frequently used in case of high carbon black throughput rates but the type of systems are varying significantly. It is to be

recommended to use dense phase conveying systems providing the lowest fines content possible. If low structured blacks have to be conveyed in such a system, the pipes should be equipped with a rubber inner-liner or similar systems to prevent sticking effects, which again can lead to total blockage of the system.
Other quality problems, which may arise from storage or material handling in the mixing room, can be solved with temperature and humidity controlled rooms.

Mixing Quality

Regarding the mixing process, it can be posited that all kind of product properties will turn out best if sufficient dispersion quality of the ingredients within the compound is provided in relation to the application. The mixing process including the roller mill has to fulfill this job. Therefore, mixing can be seen as the key process in the rubber processing chain.
The mixer of course has different features, which can influence compound- and product quality and which were not discussed yet. The following facts are commonly known: The rotors are of decisive importance with respect to design and cooling efficiency. The latest rotor design surely acts positive on mixing quality because of improved dispersive and distributive mixing capabilities. A better cooling efficiency of rotors leads to reduced cycle times meaning that process efficiency improves. In this sense it must be pointed out that providing constant cooling water temperatures is important, too. In [7] it was demonstrated that the black incorporation time changes if the inlet temperature of the cooling water varies. An hydraulic activated ram is also known to reduce costs and to improve batch-to-batch uniformity [8]. Further progress in terms of quality can be achieved with automated oil injection systems instead of manually operated ones [8]. An automatic process control unit is able to stabilize the mixing process during oil injection on basis of power consumption calculations.
Automation of the mixing process like made possible by e.g. the so-called PKS 500 system [9] can be extended if the whole mixing room technology is taken into account. Hereby, the whole process in the mixing room becomes visible. For this, “black box” concepts can be applied, starting at silos, including dosing, weighing, feeding and mixing procedures, ending at the downstream equipment like dump extruders and/or two-roll mills. Complete process information can be assembled and analyzed, which enables processors to react in case of appearing quality variations. Software, being able to process all data of the mixing room is e.g. sold by Eclipse Technical Software Services BV, The Netherlands [10]. This software is also able to enable customer report. However, the earlier mentioned MixCont control system combines everything in a more intelligent way. MixCont analyzes all process data and automatically controls the mixing process in case of raw material variations or quality variations of ready mixed compounds. If MixCont is able to provide solutions in case of any quality impact imaginable must be kept open here. It is on the other hand doubtful if this is possible at all. If the mixer will be used as a sensor, indicating raw material quality parameter variations or similar, MixCont is not evitable because the system controlls the process. However, MixCont is able to lessen compound quality variations of about 20%, as said by Trelleborg. It can be therefore concluded that the MixCont system is a promising tool at present time to be applied as a basic quality assurance system in the mixing room, covering several jobs at once. If it is the most effective tool must be also kept open here. Anyway, if a system like the MixCont control system is applied in the rubber mixing room, a different strategy of quality control would be followed, because the sensor ability of mixers can’t be used due to its active process control routines. Therefore, the MixCont system will be introduced and discussed separately in the next chapter as a different strategy for mixing rooms. The

capability of MixCont will be increased of course, if the latest mixing room technology will be installed whose advantages have been discussed before.

Testing Strategy

Last but not least the testing methods are of decisive importance in order to judge quality of mixed compounds as well as to correlate the testing results to product quality. As argued before, quality influences caused e.g. by unknown raw material quality parameter variations or mixing problems have to be detected by testing methods in order to avoid quality problems of the finished product. Therefore, the evidence of testing results is quite clear with respect to a quality assurance concept for the rubber mixing room. First of all, it is to be recommended to use the data provided by the mixer like the power and temperature signals. Such an analysis should be a basic control routine in order to detect variations of quality influences likely to occur before the mixing proces.

If mixed compounds are regarded, a method must be selected, able to provide reliable information about the dispersion quality of rubber compounds. As discussed before the dispersion of ingredients in rubber compounds can be seen as the most important task in the mixing process, which again is the most important process within rubber processing.

The most promising method in this manner according to the experience gained in the Mini Derucom project is the QS extruder, which was developed to detect surface defects respectively dispersion problems and rheological compound properties. The method can be applied online and automated to the greatest extend possible. Furthermore, it was found sensitive enough to discover carbon black quality differences in case of only slightly modified material. Rubber processors would only have to know the correlation between dispersion quality and the end article quality. This direct correlation must be investigated before a certain compound will be produced in series. However, the system was predominantly tried in case of EPDM compounds of low and medium viscosity levels yet. The question is if it will work with the same accuracy in case of high viscous compounds. If, for example, compounds are regarded containig fibres, the system will be not very helpful. The Surface Analyzer is additionally equipped with an rheological analysis tool, which is able to characterize the processing behaviour of rubber compounds online.

The rubber processor analyzer (RPA) provides information about rheological compound properties, which are important to know for further processing. In contrast to the so-called **M**oving **D**ie **R**heometer (MDR) usually applied in the mixing room, frequency-, temperature-, and strain sweeps can be derived with only one sample. This means that the RPA can be used as a processability tester and a curemeter as well. The RPA must be therefore seen as an enhanced development of rotorless curemeters, where the shear rate is independent of the samples' diameter and the strain applied on the sample. The complexity of running RPA tests was overcome in the Mini Derucom project by standard testing protocol developments and the introduction of the so-called "**P**rocessability **I**ndicators PI_I". Furthermore, software is in hand, able to qualify and quantify the non-linearity of rubber compounds. Thus it can be concluded that the RPA combines the two devices Mooney and MDR. The Processability Indicators PI_I have been found more sensitive than the Mooney results in the project and curemeter tests can be obtained with much extended information. Of course it is a disadvantage of the RPA that, assuming all testing capabilities will be exploited, the time to run the test will exceed usual mixing cycle times. This would mean that 100% of the produced can't be tested any more. On the other hand it must be asked if this

is really necessary. In this manner the rheological analysis tool of the QS extruder described before will fill the gap.
However, a general guideline for minimum testing cant' be given as discussed before. Such a decision will surely depend on the complexity of the respective compound in terms of processing behaviour and the type of application as well. It will be reminded once more that there are also applications, which seem to have big safety margins that don't require highly sophisticated testing facilities. In such cases, the standard testing devices Mooney and MDR will be almost sufficient. Therefore, the suggestion made in chapter 5.2.4 will just be repeated:

- Mixing Data – Online Quality Assurance Tool – RPA
  (highly sophisticated applications)
- Mixing Data – Online Quality Assurance Tool – MDR
  (normal applications)
- Mixing Data – Mooney Device – MDR
  (un-sensitive applications)

5.3.3 The Mixing Control System MixCont

The MixCont system is a mixer control system, which was developed to eliminate every kind of quality impacts to the highest extend possible [11]. It was aimed to minimize batch-to-batch variations. As mentioned before, there are different strategies to fulfil this job. MixCont can be seen as an additional tool to the strategy discussed in 5.3.2 to ensure compound quality in the mixing room. However, MixCont can only reduce the effort to investigate raw material influences because MixCont takes decisions about control operations by itself. Herewith, MixCont is able compensate such influences in some cases. Since MixCont will be available on the market soon, it was decided to develop no competitive system in the Mini Derucom project like introduced on basis the idea of a Complete Process Control Model (CPC, see chapter 5.1). Especially the restrictions and specialities of rubber processing as mentioned before clarified that this is impossible. Therefore, the MixCont system will be introduced more detailed and the capabilities of the system as well as the weak points/limits will be discussed in the following.
Ordinary mixing control systems use mixing parameters to control the process in a predetermined repetitive sequence. Key processing variable will be recorded and can be used to adjust the process manually in order to achieve constant product quality. However, the accuracy of adjustments depends on the skills of the process engineer. Therefore, the quality of the product and the process efficiency still depend on the user.
The MixCont system uses another approach [11]. MixCont performs an on-line evaluation of the situation within the mixer as well as an evaluation of the respective compound properties on basis of real time mathematically motivated control decisions. These calculations were proven to result in sufficient compound properties. The system processes the following parameters:

- Electric power,
- Ram pressure,
- Compound temperature,
- Ram position and
- Rotor speed.

The mixing process itself is controlled by the parameters:

- Rotor speed,
- Ram pressure,
- Total mixing time,
- Number of ram lifts within a cycle and
- Duration of different parts of the mixing cycle.

The control algorithm is based on a mathematical model and empirical information gained in about 10 years. An equation system relates the control variables, changes of mixing variables and elapsed time of the process, which then is going to be solved on basis of numerical simulation. Furthermore, the system takes quality information of previous mixed compounds and the history of the actually mixed batch into consideration.
Data of the power and temperature sensor of the mixer is averaged in certain time steps. The averaged information is then inserted to the equation system in a real time modus. The coefficients of the equations are adjusted after each mixing cycle as a function of: a) averaged power of all cycles mixed by the system in case of the recipe regarded, b) differences between actual viscosity obtained in the present mixing cycle and the requested viscosity, c) differences between the actual dispersion obtained in the present mixing cycle and requested dispersion. The frequency of computing the equation system and scanning the sensors of the mixer like power, temperature, ram pressure and rotor speed is higher than the free-running frequency of the object.

This means that for the first time quality information is taken into account for control operations of the mixing process. If for example changes of the raw polymer viscosity occur, the system would automatically adjust process parameters like the rotor speed to attain the requested compound viscosity. It was demonstrated in the Mini Derucom project that the system also recognizes variations of the polymers' quality parameter long chain branching and tries to compensate their individual effects in the mixing process. In [11] it was proven that it is also possible to compensate viscosity variations of three different grades of natural rubber.

The MixCont system also consists of a so-called "mixing quality management system", which exists of three modules. The first one is the PROCON system, which is PLC based and controls weighing and the data of the mixers' sensors like traditionally done. The second module is the MixCont system itself and the third one is an information-handling module, which includes a database and a data processing unit. The database is designed to enable statistical analysis of mixing data and quality information. It includes the following parameters: a) mixing data of the mixer and the MixCont system, b) process disruptions of any kind classified in planned and unplanned stops, and c) laboratory data and specification limits.
Further information like e.g. recipe data and material data can be attached. The main functions of this tool are to discover quality variations in dependence of mixing parameters and to enable reporting. Herewith it will be possible to establish clear practical solutions for process engineers to enhance product quality and the production process. Information can be provided on the net.

If the capabilities of the MixCont system are compared to the results of the Mini Derucom project, it looks very promising in various respect. However, there are also some weak points, which have to be mentioned. If for example polymers contain rest catalyst contents, which are able to influence e.g. the curing reaction or special properties of finished products, the mixing quality managing system might eventually recognize variations of the compound property, but it will be impossible to compensate the corresponding influence. It might be deduced that this is not the job of MixCont but it means that MixCont can be only a part of a total quality assurance system in the mixing room. Another example is linked to the carbon black pellet hardness. It was found that hard pellets of high structured blacks end up with an higher energy consumption during the black incorporation phase and consequently shorter mixing time as shown in chapter 5.1.1. This of course is already counterproductive. MixCont would probably have a problem of decision if exposed to this problem. It is hard to imagine that MixCont will increase rotor speed to overcome the bonding forces of the harder pellets if a batch consumes more energy. It would be logical to reduce rotor speed in case of higher energy consumption. However, it was not checked what would happen within the project. The problem is likely one of decision priority within the system. If information about dispersion quality of mixed compounds would be directly in hand, it is imaginable that the effect of poor dispersion would lead to an increase of rotor speed. Of course this would again affect the process due to a fster temperature rise of the batch. This conclusion also leads to another weak point: The quality information provided by standard testing methods was found problematical in many cases in the Mini Derucom project. This is especially valid for all kind of methods being linked with the carbon black dispersion. The QS extruder was developed as a result of that because the accurate TOPO method is not ideal to be applied in the rough production surrounding. Furthermore, the Mooney viscosity was discovered to be a rough method as well, not being able to distinguish all the raw material parameter variations investigated. Consequently, the feed back of quality information to the MixCont system can only be as good as the information provided by testing methods. Another porblem, which also became obvious in the Mini Derucom project, is the link of compound quality to product properties. This link was found missing in some cases, which reinforces the problems. However, if quality control will be understood in the way introduced above, providing sufficient mixing quality, MixCont is still a promising tool. Independent of the above mentioned disadvantages MixCont is known to reduce variations of compound quality about 20%. This of course will lead to improved product quality respectively less scrap in case of products, where corresponding correlations exist. It can be furthermore assumed that the savings will be strenghtened if MixCont will be applied in a new quality assurance concept, using improved testing methods and so on. On the other hand, there is still no information available describing the actual scrap savings. Anyway, the MixCont system will be offered on the market soon and can be seen as a part of a total quality assurance concept. However, it will be repeated once more that in case of the application of the MixCont system on top of a mixer, its sensor like ability will be lost, which was found more sensitive than most of the testing methods applied.

This discussion again underlines the complexity of rubber compounding and emphasizes the meaning of a total quality assurance concept, which is based on different parts trying to take all quality impacts into account.

6 Exploitation and Follow-Up Actions

6.1 Exploitation

6.1.1 RELMA

The job of the KTP is to ensure exploitation of the RELMA unit, which is one of the main outcomings of the Mini Derucom project. RELMA was planned to be applied for the characterization of raw materials in order to extend its application range in the mixing room. Furthermore, the RELMA hardware should be upgraded and an easy strategy to validate IR spectra should be developed. Finally, the evidence of RELMA results were planned to be checked in a field test by analysing factory samples more or less online. The question if RELMA is able to serve additional information in comparison to standard testing methods in the rubber mixing room was answered. Economical aspects of the RELMA unit have been considered as well.
At present time it can be stated that the objectives of the RELMA developments have been reached. The RELMA task was completed by implementing a new software, a new spectrometer and new sensors, to mention the main components. From this point of view, the RELMA unit is ready to be offered to the market. On the other hand the economy calculation showed that the unit is still as expensive as before the upgrade work. Therefore it can be concluded that the acceptance of the RELMA unit in the market depends strongly of the savings in the mixing room, which are achievable. The general advantages of RELMA have been discussed very detailed in chapter 5.2.4. It can be said that RELMA is a very promising tool to be used as a quality assurance device in the mixing room. However, the new equipment like the spectrometer, the gratings and the software are not yet completely tested because it was finally installed at the end of the project. Furthermore, there is room for improvements reducing the spot size which improves the capabilities for elements being included to higher amounts in recipes. The resolution of the method is still insufficient in such cases, which reduces the range of application. More work will be spend on these items in a new project, investigating RELMA to be used as a tool to detect chemical elements, which are forbidden according to the EC regulation for old cars. It is a so-called Common Research Project, where the companies Volkswagen AG, Freudenberg Dichtungs und Schwinungstechnik KG, Veritas AG, Eaton Fluid Power GmbH, Hutchinson GmbH, Rhein Chemie, Daiso, Contitech Schlauch GmbH, Zeon Europe GmbH, Pass Gummiwerke GmbH & Co.KG, Phoenix AG and Mündener Gummiwerk take probably part.
The commercialization of the RELMA unit will be done by the company Krupp Elastomertechnik GmbH, Gummimischtechnik Freudenberg. This company sells mixers to the rubber processing industry and therefore has a good access to the rubber processing market. Since Krupp is not able to manufacture the RELMA unit a partnership is needed, which can be found in the company Roper Scientific. Roper Scientific delivered all the components, which have been installed. However, more detailed negotiations have not been made yet because of the technical status of RELMA, which has to be further developed at first.

6.1.2 RPA

Will follow.

6.1.3 QS Extruder

Will follow.

6.2 List of Presentations and Publications

At present time a number of presentations have been held an some publications appeared in magazines. These presentations and publications are listed below:

1. H. Keuter, D. Ackfeld, A. Limper: RELMA - Ein Instrument zur Qualitätssicherung im Mischsaal – Teil 1: Kautschuk Gummi Kunststoffe **53** (2000) p. 566-573.
2. H. Keuter, C. Rüter, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Fines Content – Part 1; presented at a meeting of the Rubber Division, American Chemistry Society on April 4th, 2000 in Dallas, USA.
3. H. Keuter, J. Breuer, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Fines Content – Part 2; presented at the Kautschuk Herbst Kolloquium 2000 in Hannover on November 8th, 2000.
4. A. Limper, H. Keuter: The Influence of Raw Material Parameter Variations in the Mixing Room: Long Chain Branching of EPDM Polymers; presented at a meeting of the DKG in Bad Neuenahr on September 13th, 2001.
5. H. Keuter, A. Limper, A. Wehmeier, T. Riedemann, K.-H. Freitag: Feinanteilanstieg und Anhaftverhalten bei der pneumatischen Förderung von Ruß. Schüttgut **6** (2000) Nr. 4, Seite 385-394.
6. H. Keuter, A. Limper, A. Wehmeier, T. Riedemann, K.H. Freitag: Increase in fines content and adhesion behaviour in the pneumatic conveying of carbon black. Rubber World, Volume 224, No. **4/5**, 2001.
7. A. Limper, H. Keuter, D. Schramm: A Review of European Rubber Research in Practice – Mini Derucom and Prodesc. Conference booklet of the eponymous conference, held on January 9-10th, 2002 in Paderborn, Germany.
8. J.L. Leblanc, C. de la Chapelle: Two posters presented during the 8th international seminar on elastomer on May 9-11th, 2001 in Le Mans, France.
9. H. Keuter, C. Rüter, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Fines Content – Part 1; was send to KGK in 12/2001.
10. H. Keuter, J. Breuer, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Fines Content – Part 2; was send to KGK in 12/2001.

The results of the Mini Derucom project have been disseminated during the rubber conference “A Review of European Rubber Research in Practice – Mini Derucom and Prodesc” which was organized by KTP. The preparation started with a press conference, which was held in Paderborn on June 11th in order to get good advertisement. Flyers were printed and an internet page was created, showing the contents of the projects and allowing to register online.The conference was held in Paderborn, Germany on January 9-10th, 2002 in the PaderHalle. About 130 people from the industry attended the conference.

6.3 Follow Up Actions

Some more publications will follow, which are listed below:

1. H. Keuter, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Pellet Hardness – Part 1; to be send to GAK.
2. H. Keuter, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Pellet Hardness – Part 2; to be send to GAK.
3. H. Keuter, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Pellet Hardness – Part 3; to be send to GAK.
4. H. Keuter, A. Limper, N. Priebe, B. Kjellberg, U. Magnusson: The Influence of Raw Material Parameter Variations in the Mixing Room: Substitution of Aromatic Oils; to be send to GAK.
5. H. Keuter, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Long Chain Branching of EPDM Polymers; to be send to GAK.
6. T. Grüter, H. Keuter, A. Limper: RELMA - Ein Instrument zur Qualitätssicherung im Mischsaal – Teil 2: to be send to KGK.
7. H. Keuter, A. Limper: The Influence of Raw Material Parameter Variations in the Mixing Room: Carbon Black Fines Content – Part 3; to be written and to be send to KGK.

7 References

[1] H. Keuter, D. Schramm, A. Limper: A Review of European Rubber Research in Practice – Mini Derucom and Prodesc; Conference book of international conference, held in Paderborn, Germany, January 2002.

[2] H. Keuter, D. Ackfeld, A. Limper: RELMA - ein Instrument zur Qualitätssicherung im Mischsaal; KGK 53 (2000) Nr. **10**, p. 566-573.

[3] W. Häder, C.-J. Lorenzen: RELMA: on-line quality control in the rubber mixing room using laser analysis; Technische Mitteilungen Krupp 2/1992.

[4] J.L. Leblanc, C. de la Chapelle - Up-dating a torsional dynamic rheometer for Fourier transform rheometry on rubber materials – ACS, Rubber Division Meeting - Cleveland, Ohio, USA – Oct. 16-19, 2001 – pap. No. 110.

[5] A. Wehmeier: Entwicklung eines Verfahrens zur Charakterisierung der Füllstoffdispersion in Gummimischungen mittels einer Oberflächentopographie; Diplomarbeit Fachhochschule Münster, Abteilung Steinfurth, 1998.

[6] H. Geisler: Einfluss der Mischungsherstellung auf die Produkteigenschaften; in: Umdruck VDI-K Seminar „Mischungsherstellung für Elastomere – Workshop mit praktischen Versuchen".

[7] W. Häder, A. Limper: Der Mischsaal unter Systemaspekten; Kautschuk Gummi Kunststoffe 45, Nr. **11**, 1992, p. 951-956.

[8] J. Pohl, A. Limper: New angles in rubber mixing technology. Tire Technology International 1993, p. 125- 130.

[9] W. Häder, H.-M. Monyer: Integrated control. Tire Technology International, June 1999.

[10] D. Urbanik: Computer assisted compounding; Rubber Technology International 1998, p. 88-92.

[11] M. Sarbatova, U. Petterson, S. Brassas: Advanced real time control system for rubber mixers. Paper presented at the International Rubber Conference in Birmingham, UK, 2001 on June 12-13.