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SPECIAL PP'S FOR A DEVELOPING AND FUTURE MARKET

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Key Words: Special PIP, α -/ β -i-PP, Structure Property Relationship, MC-Catalyst, Soft-PP, Postreactor Technology

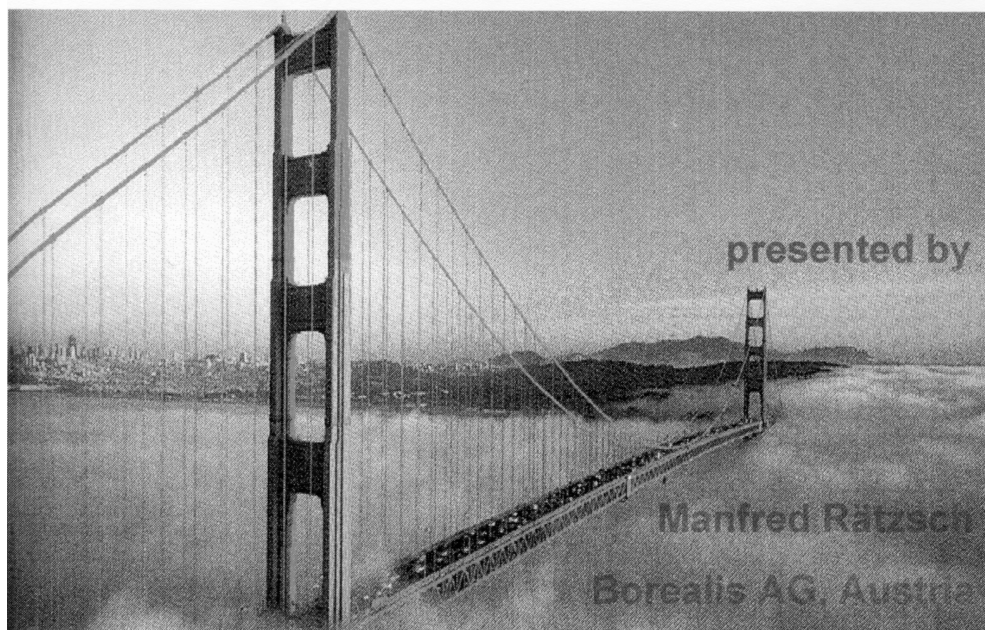
ABSTRACT

The present article starts with a short overview of the international development of the polyolefin production in the last 10 years and with a forecast for the next 3 to 5 years. The reason for these developments, as the cost/properties relationships will be discussed. The extended properties of the polypropylenes, their molecular and supermolecular structures, and the correlations to the properties play a central role in the paper. The reinforcing and compounding maximized the properties and reduced the weaknesses of the mechanical and thermal properties. The narrower molecular weight distribution and the possible new copolymers with better randomness and new comonomers explain the importance of the metallocene catalysts. Examples demonstrate the future developments of new PP's by orientation on a second generation of single site catalysts.

The super soft PIP's as a new interesting elastomer class, are described. The new developments from the post reactor modification to a new HMS-PP technology are described in detail. The new graft copolymers with styrene and methylmethacrylate are pointed.

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Special PP's for a developing and future market



INTRODUCTION

The bridge in the opening slide demonstrates the narrow connection between a R&D and marketing in the development of new high value polyolefins as an efficient working procedure for a polyolefin producing company. People in R&D can't develop new successful products for the market without the knowledge of what final producers and customers need in the next 5-10 years and what are the experiences of the customers with the present material. On the other side, the marketing people need all the information about the international product developments, the qualities and the possibilities for producing and timing of products, and the cost for development and production.

The experiences in our company demonstrate that new products are successful in the market with narrow interactions between R&D and marketing.

By looking into the near future, the forecast for the margins of the polyolefins (PO's) from Chem Systems [1] in Figure 1 is more pessimistic than opti-

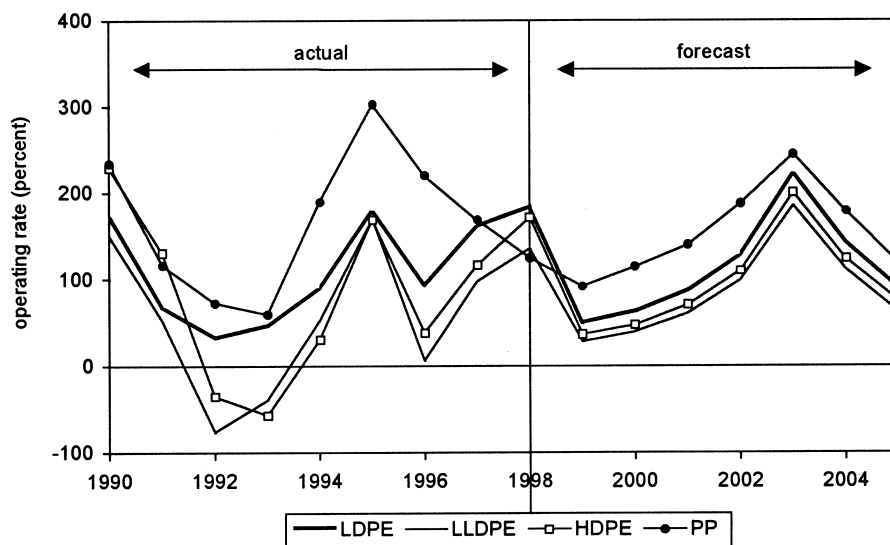


Figure 1. Polymer margins 1990–2005.

mistic. Interesting in this forecast is that polypropylene (PP) always has a higher margin than the polyethylenes (PE's). The reason for that is that the main application field for all PE's is packaging and in contrast to that, PP has only 37% in this application field. The main part of the packaging PO's are film and bottle grades with a commodity profile and so their prices on the market are very sensitive against overcapacities.

Figure 2 shows the end use applications for PP in Western Europe. A main reason for the better merging of PP is that the growing market in automotive, household and electrical industry always has higher value products. So, the decreasing of the margins is not as intensive as those of other PO's. The consumption in Western Europe in the last 10 years has the highest growth rate in PP as shown in Figure 3. The reason for that is that PP goes with its high mechanical potential in direction of the substitution of ETP's, e.g. ABS, PA, PET and also in the substitution of PVC with its stiff and its environmental problematic soft grades. The pyramid of thermoplastic-world capacities 1998 in Figure 4 shows the relation between PP and other thermoplastics and the main substitution potential. The growth rates of i-PP in the different continents of the world in

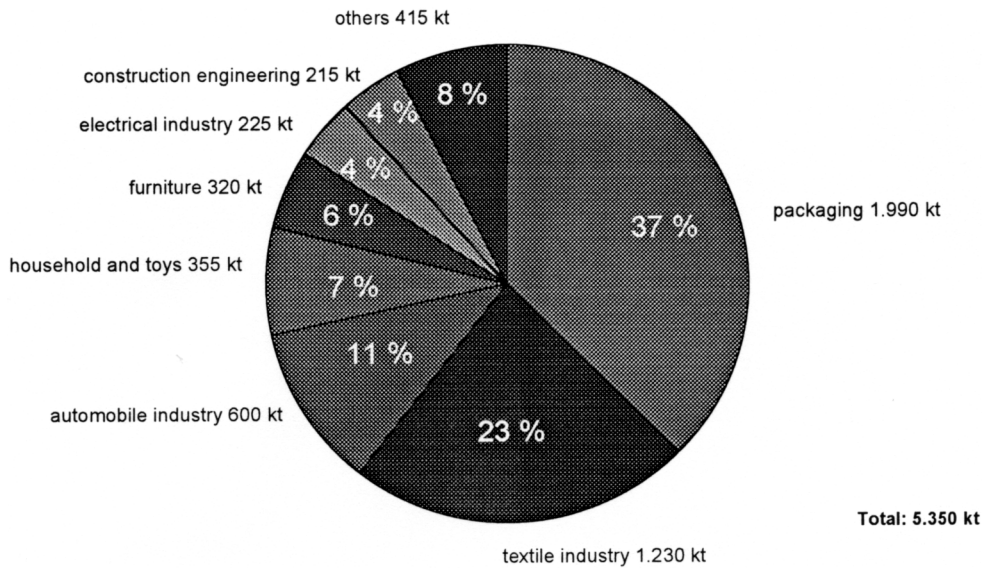


Figure 2. End use applications for PP in Western Europe 1996.

Figure 5 show the main growth in China and Asia as the main future market potentials [2].

In this paper, I will try to clarify the reasons for the enormous potential of PP and the future chances by developing new special materials based on PP [3].

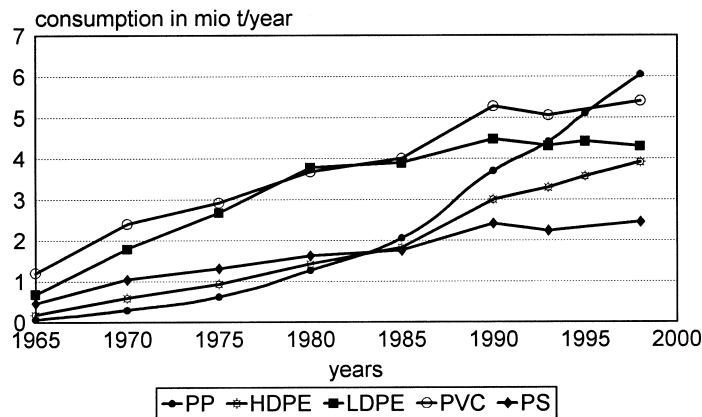


Figure 3. Consumption of plastics in Western Europe.

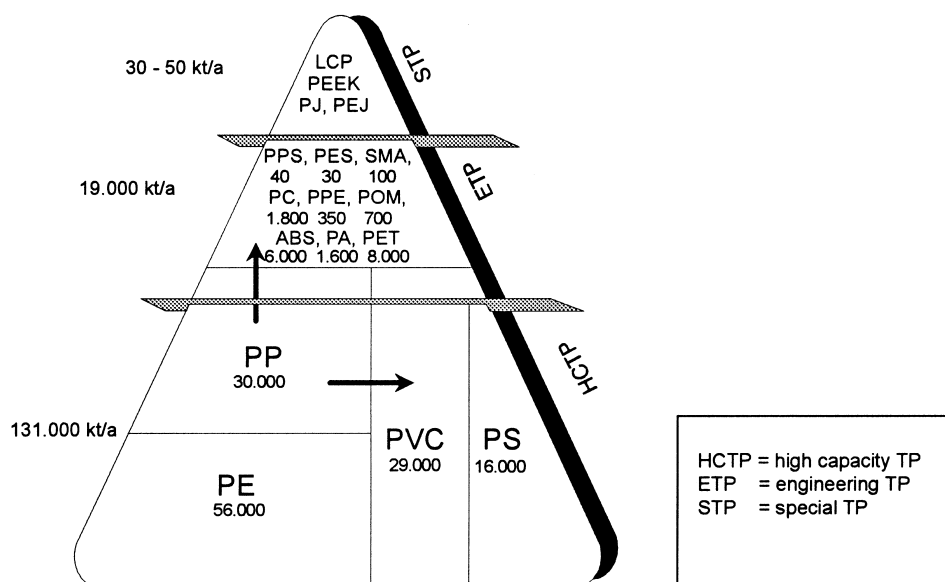
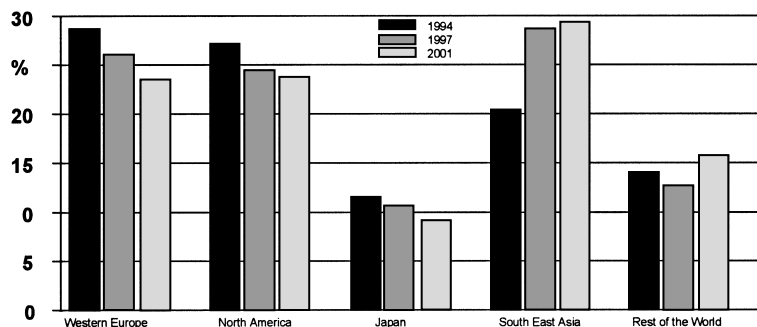


Figure 4. Pyramid of thermoplastics world capacities 1998.



region	1994		1997		2001	
	cap. (in 1000 t/a)	share (in %)	cap. (in 1000 t/a)	share (in %)	cap. (in 1000 t/a)	share (in %)
W Europe	5,7	28	6,9	26	8,9	23,5
N America	5,6	27	6,8	24	8,9	23,5
Japan	2,4	11	2,8	10	3,3	9
SE Asia	4,1	20	7,5	28	11,0	29
Rest	3,0	14	3,3	12	5,8	15
total	20,8	100	27,3	100	37,9	100

Figure 5. World capacity for PP according to regions (1994/1997/2001).

Structure Properties Relationship

All the special properties of PP are based on the molecular structure. The macromolecule of PP has a wide range of variability. There are different structures as isotactic, atactic, and syndiotactic based on the molecular orientation of the methylgroups in the macromolecule. The most important product with the highest mechanical potential is the isotactic PP (i-PP). The syndiotactic (s-PP) and atactic PP (a-PP) are in contrast to these elastomeric materials. In the melt, all these polymers are statistically coils with different chain lengths of the macromolecules. During the cooling of the melt, the material will get a more or less ordering of the macromolecules which depends on the degree of tacticity. The first order of orientation build-up helices with different packaging, which in the second order partially crystallizes and forms spherulithes as a third order. Table 1 shows the different helices and the crystalline structures of i-PP and s-PP. i-PP has besides its well known (α -modification (α -i-PP) also a isochiral, right handed structure with lower crystallite density, the so-called β -modification (β -i-PP), and further a γ -modification (γ -i-PP) which has not yet been discussed here, because of their practical unimportance at this time [4].

TABLE 1. Helices and Crystallinity of PP

modification	α i-PP *)	β i-PP	s-PP
helices packaging	only left handed or right handed	isochiral right handed	different hands
crystallite phase	stable monoclinic	metastable hexagonal	C-pseudocentered orthorhombic
crystallite change	α and β i-PP can exist within the same spherulithe at low temperatures (100-130°C) and high stress change from β to α		at 100°C change from C- to B-centered orthorhombic
crystallite/ melt-density/ temp. °C	high/163	low/151	low/168

*)

- Partly in β -modification. Helix in opposite chirality. Crystallite in triclinic (Newmann) or orthorhombic (Brückner) phase. β -----> α
- On surface crystallite in smetic phase.

The increasing of the crystallinity by nucleation agents can prefer one crystallite structure of the i-PP and increases the number of crystallites. With high efficient (α -nucleation agents very small crystallites can be realized. In practice, nucleation agents as a part of the catalyst or additional external nucleation agents which are added to the i-PP melt in the granulation extruder, are used. The crystallization is an entropic (ordering) effect during the cooling process. Thus, the crystallinity of the material with the same tacticity, in comparison to larger crystallites, can be increased. For the time being, with small crystallites, transparent film and blow molding materials result, as the crystallite dimension are under the wavelength of the visible light.

With special β -nucleation agents, 90-100% of the β -structure are being built. The speciality of the β i-PP is that they can exist with the α -modification in the same spherulite and can change at defined conditions to (α -modification [5]. The changing from the β i-PP to the α i-PP modification is one of the energy dissipation mechanisms to increase the impact strength of the material [6]. The application of pipes with enlarged impact strength is only one application of β i-PP. The potential for the β i-PP applications and market is higher than now used.

The high mechanical level of i-PP as the modulus and the mechanical properties are based on the crystallinity. The weak point of the i-PP material is the amorphous phase between the crystallites, but they are responsible for the toughness of the material and as often, the material is a copolymer to increase the toughness additionally. The importance of the crystallites for the thermal and mechanical properties can be demonstrated by the reinforcing of the i-PP coupled with glass fibers. By adding glass fibers, we get a new additional impact mechanism, the so-called, pull-out energy" from the glass fibers out of the material during the fracture of the material. Additionally, and this is amazing, the glass fibers act as a bridge between the crystals, and as schematically demonstrated in Figure 6, increase very intensively the heat distortion temperature (HDT) from the material. Figure 7 demonstrates the increase of HDT with 30 wt% chemical coupled glass fibers from 70 to nearly 150°C, that means in a value of the melting temperature of the α -crystallites.

The high mechanical and thermal properties of the glass fiber reinforced i-PP is an important fact in the applications in the automotive industry. But, the glass fibers bring problems with their high specific weight and the environmental problems during the thermal recycling (burning processes). Hence, the customers asked for a substitution of glass fiber reinforced i-PP's. One answer was the development of i-PP reinforced with synthetic or natural fibers. Figure 8

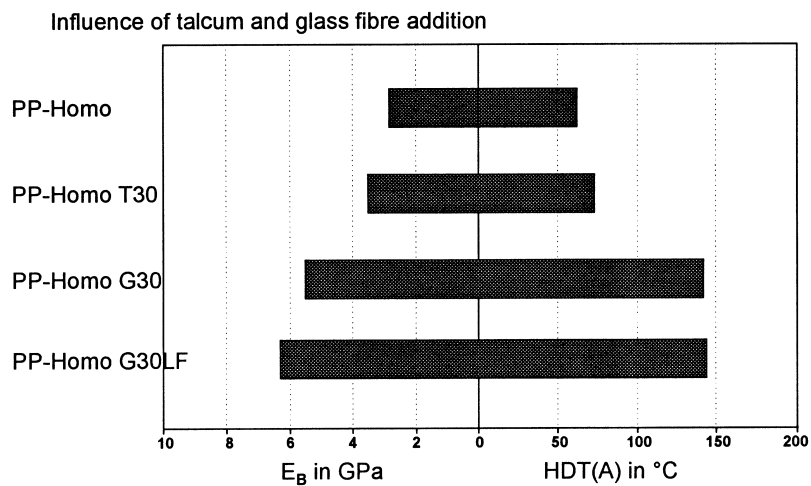


Figure 6. Flexural modulus and heat distortion temperature of PP grades.

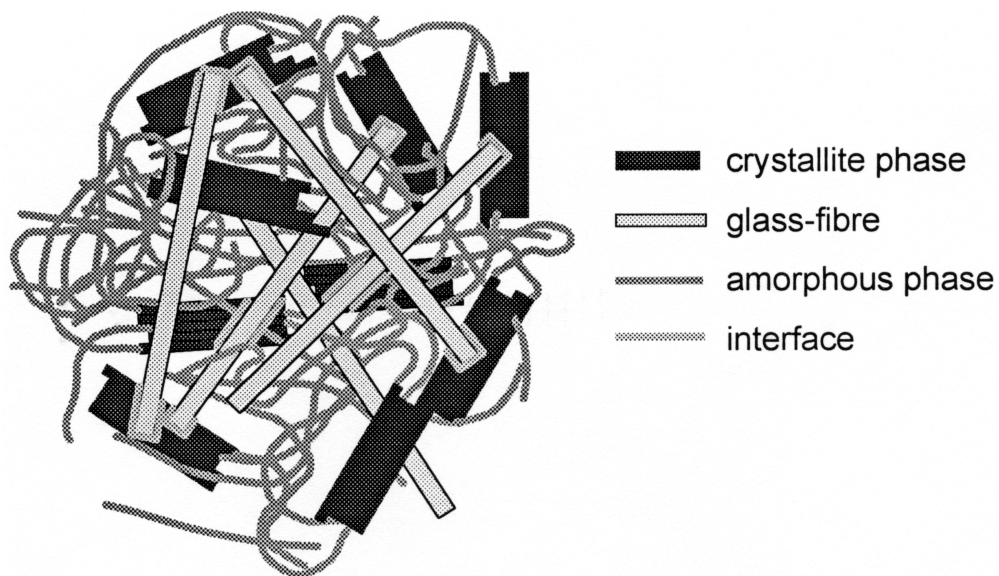


Figure 7. Glass-fiber reinforced polyolefins.

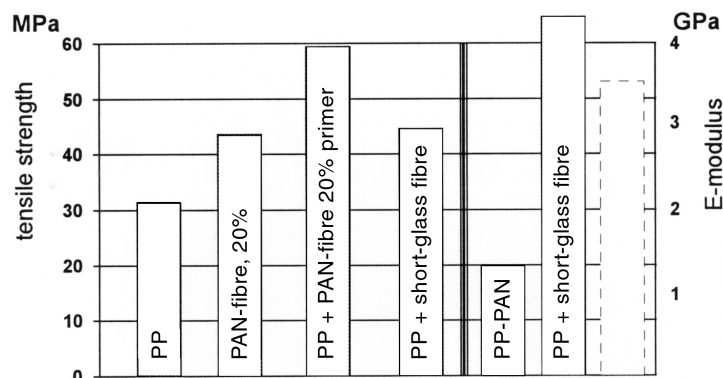


Figure 8. Tensile strength and E-modulus of PAN-fiber reinforced PP and at primer addition.

demonstrates the good level of the tensile strength of PP reinforced e.g. with polyacrylonitrile (PAN)-fiber in comparison to the reinforced i-PP, with short glass fibers [7]. The tensile strength of the PAN-fiber reinforced i-PP brings chemical coupled higher values than with glass fibers. The toughness is always at a very good level.

However, the modulus of PAN-fibers is too low to increase the modulus of the reinforced i-PP remarkably. This is a clear target for developing new synthetic fibers with a low price, good adhesion properties nucleation ability, and with a higher modulus in comparison to i-PP. Combining the fiber development with other properties, e.g. the decreasing of the flammability, the material has a good chance to come into a new horizon of reinforced i-PP's.

A new generation of reinforced i-PP's with special particles in nanometer (Nm) dimensions are now under development. Nanocomposites, on the basis of special swellable layer silicate minerals as e.g., "BENTONITE" in polyamides, polymethylmethacrylate, and epoxides, lead to an increase of the E-moduli and the notched impact strength in comparison to the well-known filled materials [8]. Montell and General Motors published in the Montell-News Release [9] a new Nm-based family of thermoplastic olefins with extended properties for the automotive industry. Nm-based i-PP-compounds give the chance to new PP-materials with high stiff and high impact properties.

Future Products by Metallocene (MC-) Catalysts

An important influence of the molecular structure of i-PP lies in the copolymerization of propylene with different comonomers. The new MC-catalysts, in comparison to the (Ziegler-Natta) ZN-catalysts, the copolymers of higher (α -olefins) bring a distinct reduction of the melting point with low contents of the comonomers due to their stronger randomness distribution. These materials are now well known in the film and bottle market as unimodal or bimodal grades.

A new way to increase the modulus of i-PP is the copolymerization of propene with cyclic olefins. The MC-catalysts make these copolymers possible, whereas, the ZN-catalysts are not specific enough to copolymerize cyclic olefins. So, the developer of stiff polypropylenes now have the possibility of playing with the balance between the crystallinity and the content of cyclic molecules as e.g. norbornen.

The potential for new products with stiff cyclic comonomers in i-PP can be predicted in the future when we are able to produce block-copolymers of the stiff segments between the segments of homo- or copolymers of the i-PP. But, this needs a further generation of MC-catalysts.

Figure 9 demonstrates the possibilities of better properties of i-PP's, which are produceable with the new MC-catalysts. The narrower molecular

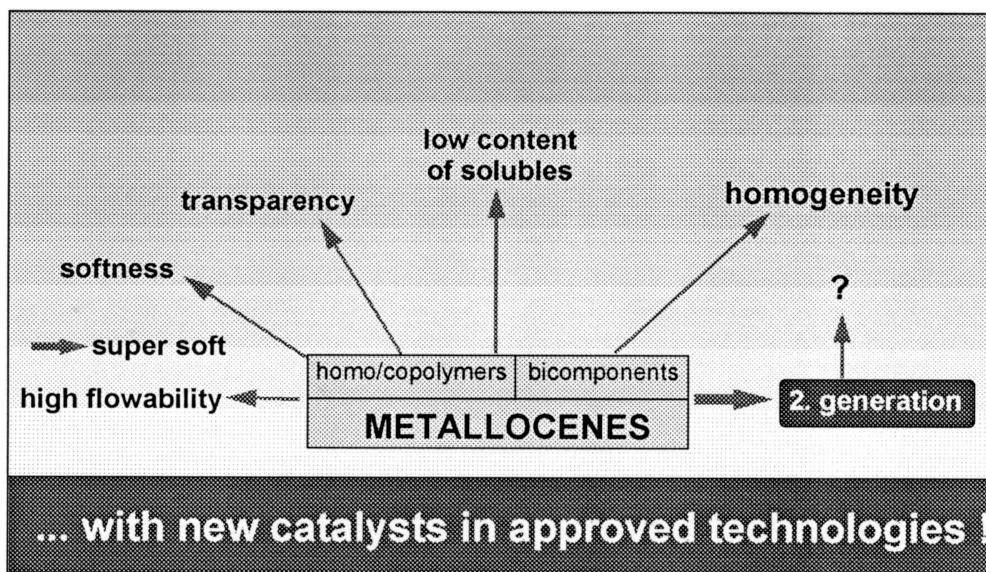


Figure 9. Designing the future of PP.

weight distribution of PP brings a low content of solubles and from this, less smell and flavor impairment.

The MC-catalysts allows us to produce i-PP's with higher flowability and better transparency. The properties of the heterophasic i-PP's, the block copolymers of EPR, combined with a random copolymer, the soft random heterophasic copolymers (RAHECOs), and with a homopolymer, the stiff heterophasic copolymers (HECOs) can be increased with MC-catalysts by better compatibilities in the interface.

There is an enormous potential in the possibility of producing a bimodal grade in one reactor by using a dual MC catalysts, that means two different active components on one support. The homogeneity of this material by growing and entangling the two macromolecules, different in length, side by side at the same time, is higher than in comparison with all materials from the conventional bireactor technologies. The growing of the macromolecules in each other leads to an interpenetrating structure, as it is schematically shown in Figure 10.

If the polymer chains are long enough, the entangling leads to a good homogeneity of this material, and fully new properties of this material can be predicted.

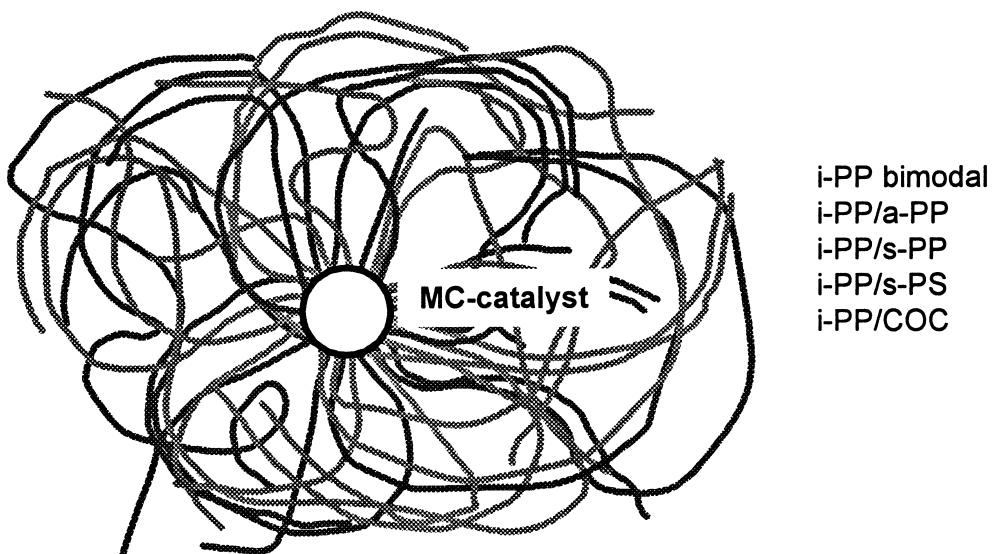


Figure 10. Interpenetrating polymers from dual MC-catalysts.

A catalyst, where one component is active to polymerize propene to s-PP and the second component-at the same time under analogous conditions- active to polymerize the propene to i-PP, leads to an interpenetrating material of the two, thermodynamically immiscible polypropylenes. This presents the possibility of coming to a new generation of materials with properties which cannot yet be estimated. The predicted material properties as e.g., high impact properties at low temperatures depend on the possibilities of the separation of the elastomeric phase.

Another future material is an interpenetrating of the macromolecules of PE or PP with syndiotactic polystyrene (s-PS) at dual catalysts, where one active center polymerizes the propene or ethene to the polyolefin macromolecule and the second center the styrene to s-PS. The high melting point and stiffness of s-PS molecules lead to an increase of the thermal and mechanical properties. The combination with the good processabilities of the polyolefins leads to a new material with extended properties.

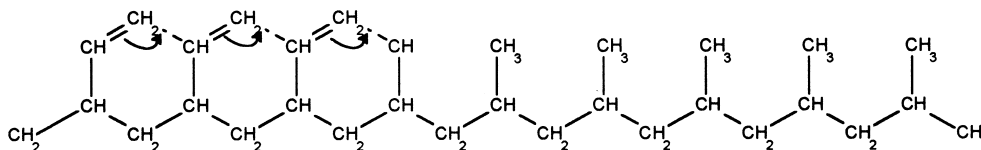
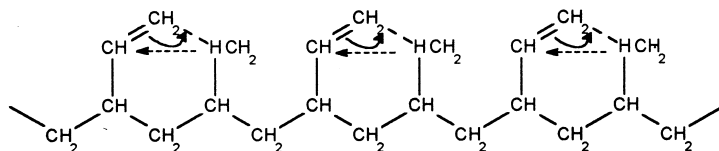
The scientists now work in a renaissance of new polymers from the well-known and cheap butadiene. Butadiene is, like ethylene and propylene, a product of the cracker-technology by the pyrolysis of benzene with the amount of approximately 20% of the propene content. The demand for butadiene has been reduced in the last years and occasionally are hydrated to other applications. New block-copolymers of olefins with cis 1,2 butadiene gives us the chance to develop new, stiff and impact materials by cyclization reactions of the butadiene units as shown in Figure 11/Equation 1. A cyclization of alternating isotactic propene/1,2 butadiene copolymers seems possible, as Equation 2 demonstrates schematically.

Super Soft PP

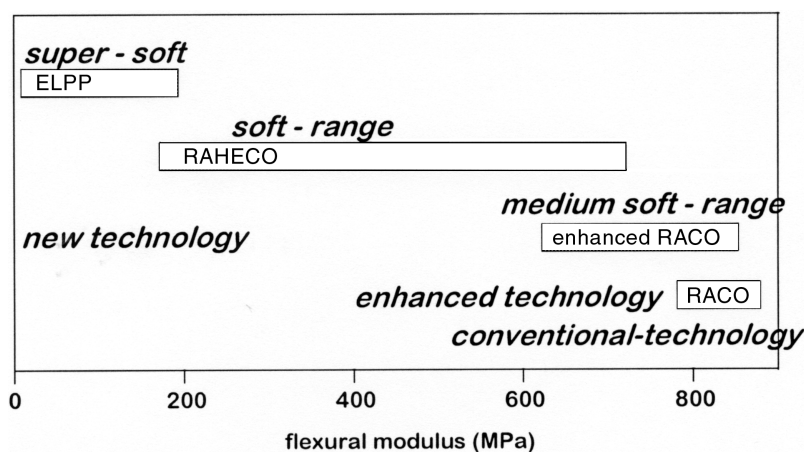
The soft random heterophasic copolymer, discussed in the section “Future Products by Metallocene (MC-) Catalysts”, the so-called RAHECO’s out of the enhanced technologies in Figure 12 are not the end of the story of soft PP.

A special super soft, elastomeric PP, the so-called ELPP with a modulus below 100 Mpa, is a high molecular weight block-copolymer with a narrow molecular weight distribution with about 90% atactic sequences and 10% isotactic propylene sequences.

The ELPP is to be produced with a special tetra neophyl zirconium catalyst from Du Pont [10]. Analogous products are now under development with MC-catalysts. MC-catalysts gives us the chance to produce this material with low tackiness and in conventional technologies [11].

block i(cis)1,2 butadiene**alternating i(cis) 1,2 butadiene / propene-copolymers****Figure 11.** New challenges with butadiene / propen copolymers.

It is further important that super soft PP's with the same molecular structure as i-PP are thermodynamically miscible with i-PP so that the whole range of stiffness and elasticity as transparent PP-applications from the mixtures can be produced by mixing the components in an extruder.

**Figure 12.** Soft-PP ... range of Borealis.

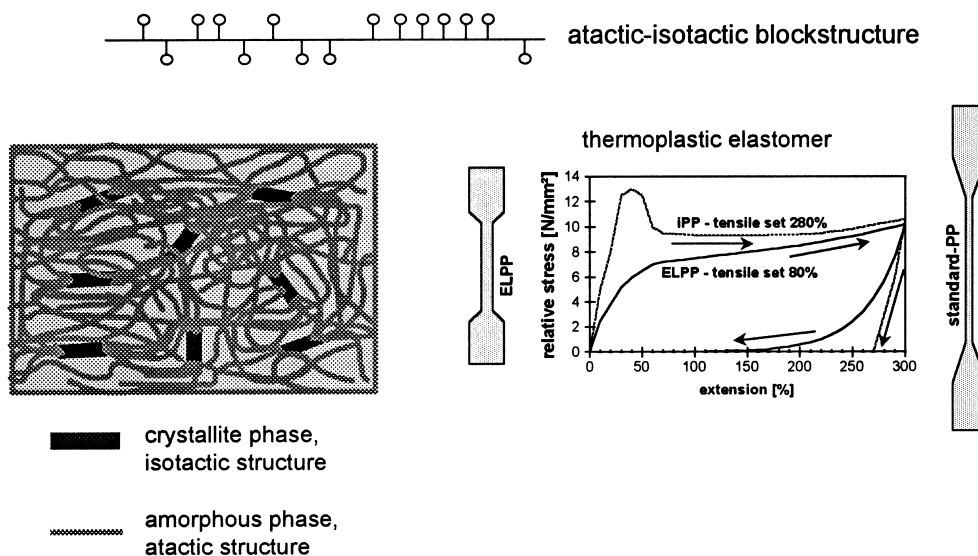


Figure 13. Two steps towards supersoft PP ==> getting competitive to high-value products.

In comparison to the high molecular atactic PP (HHW a-PP), the mystery of the super soft ELPP is that its short isotactic sequences crystallize, and lead to a physical crosslinking of this material. The result of that is the good elasticity of the material (Figure 13).

Table 2 presents an overview of properties of different soft polyolefins. One interesting fact is that the melting points of the super soft polypropylenes

TABLE 2. Properties of ELPP Compared to other Soft Polyolefins

		MC-LLDPE	conv. aPP	HMW-aPP	ELPP
MFR 230°C/2,16kg	g/10min	0,8 *	18	0,1	0,1-20
Melting point	°C	60	148	150-155	155-163
Density	g/cm ³	0,9	0,9	0,8	0,87-0,9
Tens. modulus	MPa	11	50	5	6-30
Tens. strength	MPa	7	6	2	3-8
Elong. at break	%	500	550	2000	750-1100
Hardness	Shore A	74	77	55	56-85
Haze	%	?	10	18	4-10

* MFR at 190°C/2,16 kg

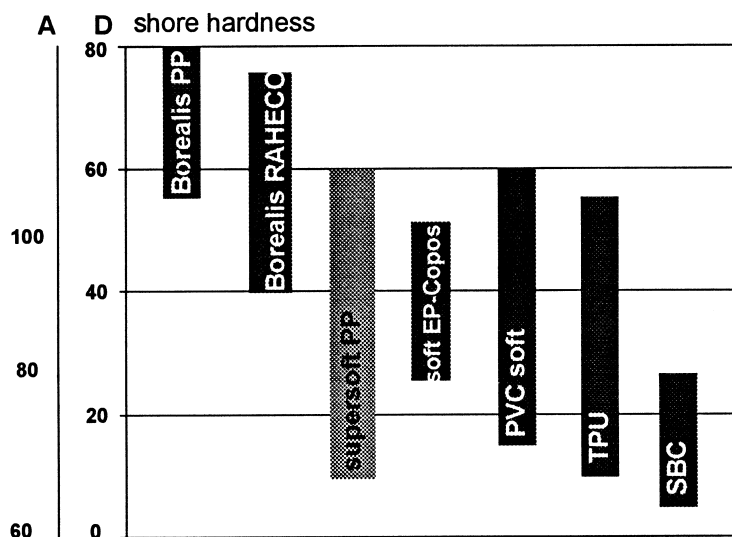
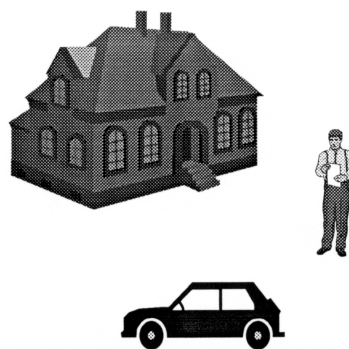


Figure 14. Supersoft PP.

are $> 150^{\circ}\text{C}$, whereas the super soft polyethylenes as LLIDPE have melting points at 60°C . Further extended properties of the super soft PP's are a high elongation at break and a low hardness. The supersoft PP shows in Figure 14 a good chance to substitute in the market application from PVC soft, TPU's and soft EP's, because of their positive environmental impact and the lower price. The possible applications of these materials are shown in Table 3. It was estimated that the

TABLE 3. Applications Discussed with Borealis' Customers for Super-Soft PP

- medical films and pouches
- sanitary films
- soft film for packaging, domestic use etc.
- soft bottles and mouldings for cosmetics etc.
- sealing profiles
- soft tubing and cable insulation
- modification of TPE's
- roofing and construction membranes
- damping elements for furniture
- flexible flooring
- bitumen modification



demand for such products is about 500.000 to/y on the European market. The high potential of substitution depends also on the lower costs of the ELPP in comparison to the more expensive conventional materials.

Products from the Post Reactor Technology

An important technology part of all i-PP technologies is the homogenization of the product and the distribution of stabilizers and other additives in the melt after the reactor in the granulation step. The process conditions in the granulation steps are very complex, depending on the rheological properties and the inhomogeneity of the products from the reactor and the needed additives. In most of the production plants, a special degradation technique to decrease the molecular weight by a radically induced splitting of the i-PP macromolecules to grades with lower viscosities is included in the melt extrusion steps.

In the following part of the paper, the development of enhanced polypropylenes in this so-called “post reactor technology” will be presented. Therefore, the importance of the knowledge of the basic reactions, which are going on in the process by radical reactions, will be discussed.

The modification of the i-PP macromolecules by radical reactions is a well-known technology process. The research work was started by the investigation of this process in detail by studying the elementary reactions. This brought us to the based idea to develop new materials by radical modification of i-PP in addition of radically polymerizable monomers. From this idea, we developed a technology with a new generation of high melt strength (HIVIS) i-PP's which is now the leading product in Europe [12]. The target was the conversion of a linear i-PP structure into a longchain branched (LCB) material as shown in Figure 15.

10 years ago, Montell started the development of LCB-i-PP by using an electron beam radiation process. Montell found the very intelligent way by radical reactions below 80°C. If one looks at the PP-degradation reaction in dependence on the temperature (Figure 16) an equilibrium between degradation and build up reaction at 65°C can be demonstrated. At the elementary mechanism as shown in Figure 17 there is, after the H-abstraction, in a second step the β -scission. However, in the third step a recombination of the two radicals to a bimodal long-chain branched i-PP can arise.

At higher temperatures, the degradation reaction is faster and the recombination reaction is so dramatically reduced, that a low viscosity material is the result.

Our experiences with the grafting of different monomers during the radical reactions onto PP leads us to an idea for our own new way to the LCB-PP.

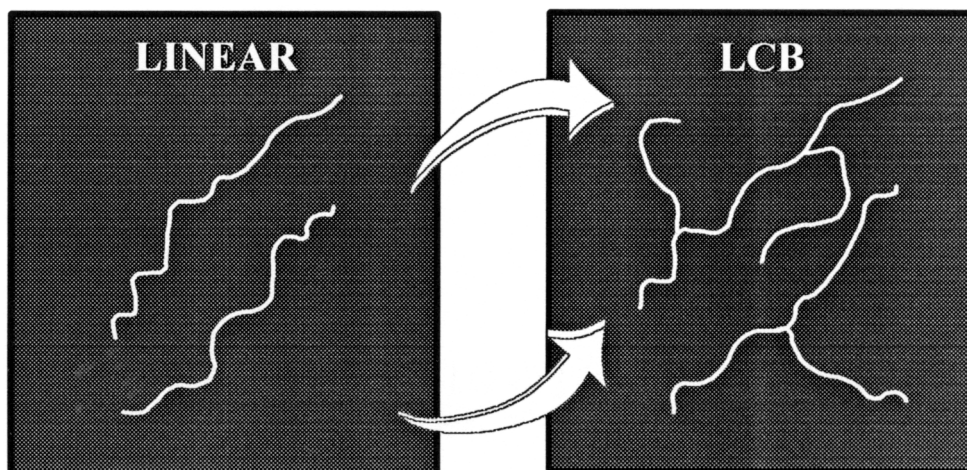


Figure 15. Producing long-chain branched PP.

Monomers with a high resonance stability are added side by side with the peroxide onto the *i*-PP powder from the reactor. By increasing the temperature, the built up oxyradicals from the peroxid split a proton from the tert. C-atom of the PIP-macromolecule chain and the tert. C-radical was the first reaction, product. The second reaction steps are side by side with a part of β -scission reaction, the addition of monomers as styrene or butadiene onto the tert.-C-radicals, and the

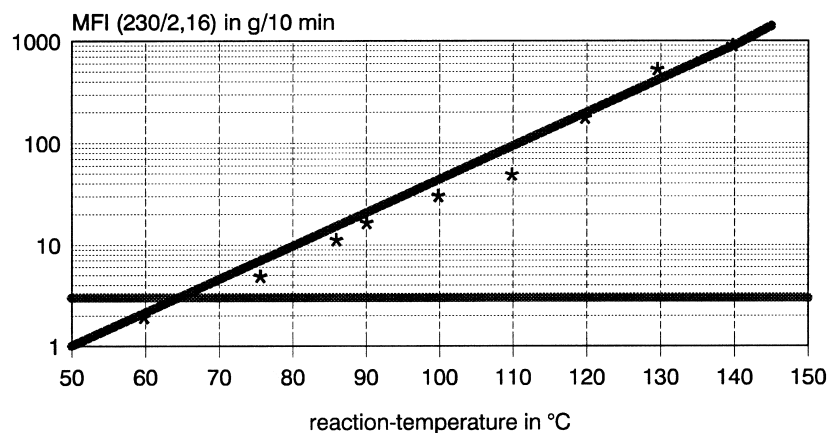


Figure 16. PP-degradation in dependence of the temperature.

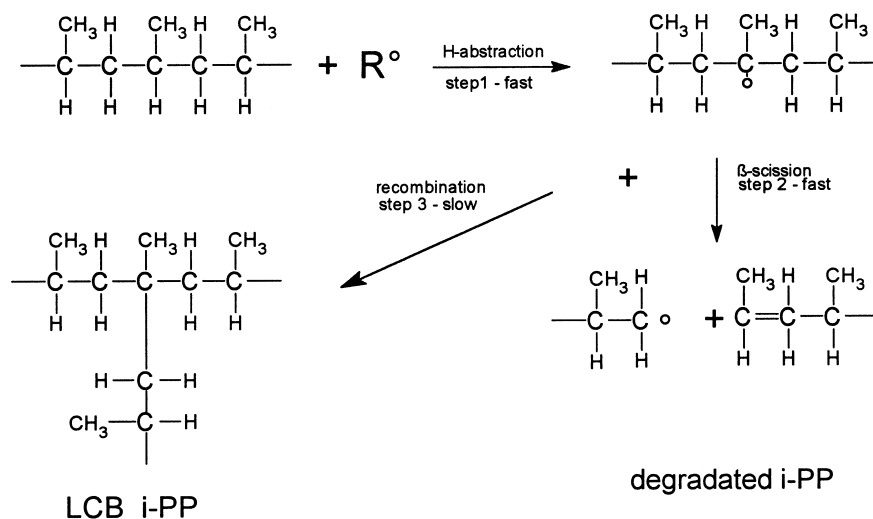


Figure 17. LCB-i-PP by radical reactions on i-PP below 80°C.

β -scission reaction formed primer C-radicals. The formed monomer radical stops the β -scission reactions, and increases the stability of the radicals to higher temperatures. The reason for that is the higher resonance stability of these monomer radicals and the following low transfer rate to the PP macromolecules. The reason for the increasing of the stabilization of the radicals is that the live time of the monomer radicals is even higher than the live time of the mainly tert.C-radicals. So, the decomposition, transfer and recombination reactions to form long chain branched PIP's can be transferred to higher reaction temperatures. The development of the so-called "Daploy technology" enables the grafting and recombination reactions at 160-180°C, that means in the intermediate range from the solid state in the melt phase in a special extruder. Figure 18 demonstrates the reaction mechanism with monomer additions, β -scissions and recombination reactions of the grafted comonomer. The result of the radical modification and recombination is a bimodal molecular weight distributed i-PP and a long-chain branching of the high molecular part with typical high melt strength (HMS) properties [13].

The quality of the HMS-PP is measurable with the rheotens-equipment measurements. The force or melt strength is measured by an increased drawing of the melt strand of the PP out of a special die (Figure 19). We have investigated the different PP-grades from the polymerization plants. In Figure 20, the melt

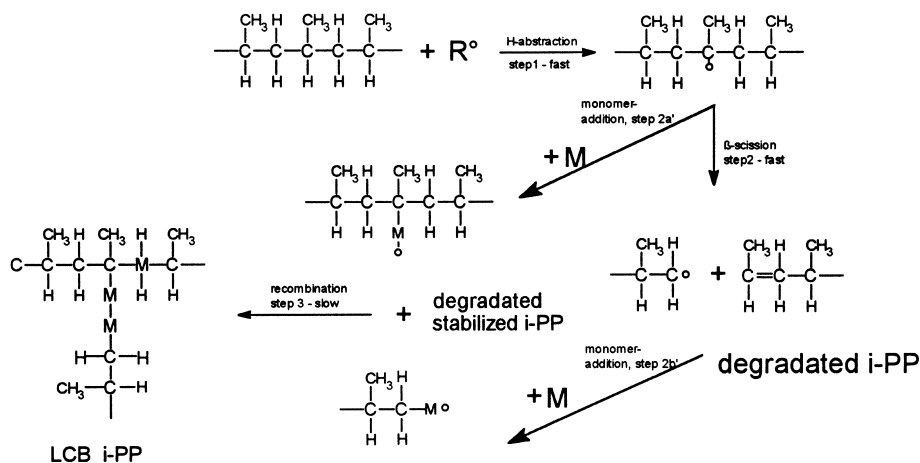


Figure 18. LCB-i-PP by radical reactions on i-PP at higher temperatures.

strength of different i-PP grades from the polymerization plant are presented. It is remarkable that the melt strength depends on the molecular weights of the i-PP. Materials with the highest molecular weight, that means lowest MR, have the highest melt strength. At the same MFI, the heterophasic copolymers (PP HECO) show a higher melt strength than the homopolymers (PP HOMO). The reason for

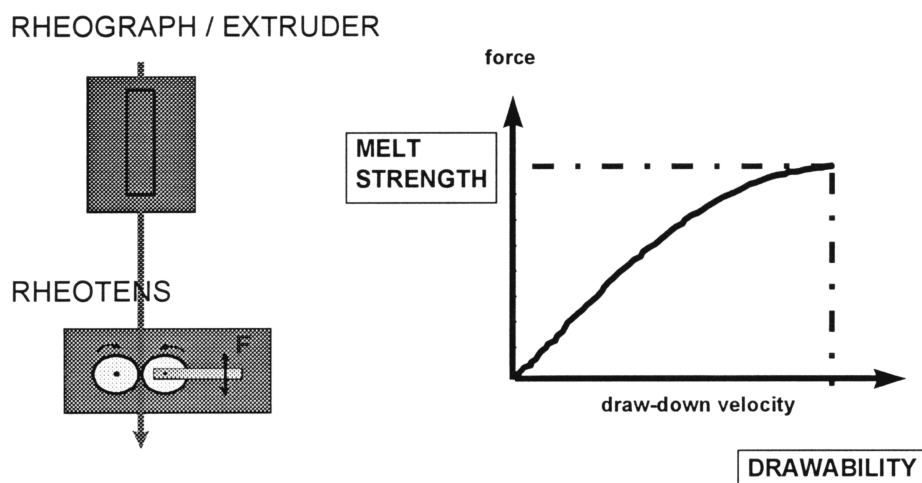


Figure 19. Principle of rheotens measurements.

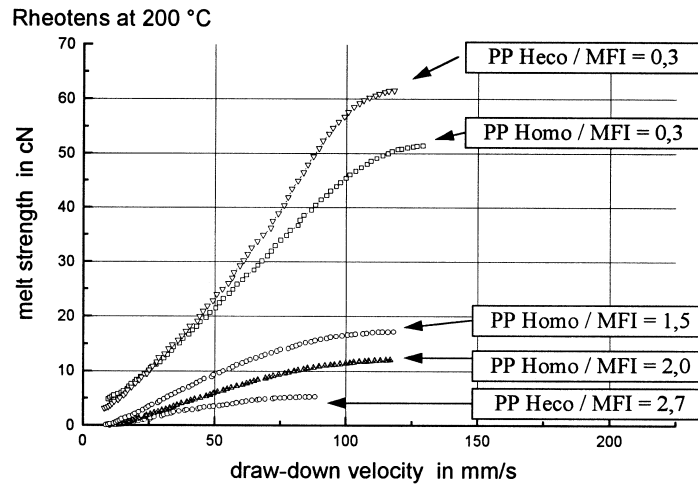


Figure 20. Strength and drawability of polyolefin melts.

that is the increased force of EPR-particles and interface adhesion between the EPIR and the i-PP phase.

Figure 21 shows that Borealis' bimodal i-PP 7148/7150 R with a MFI of 0,52 has the highest melt strength with 90 cN while the other grades are in the normal force below 30 cN. All polymerization grades have a relatively low elasticity of the melt and they break at a velocity between 80 and 120 mm/s.

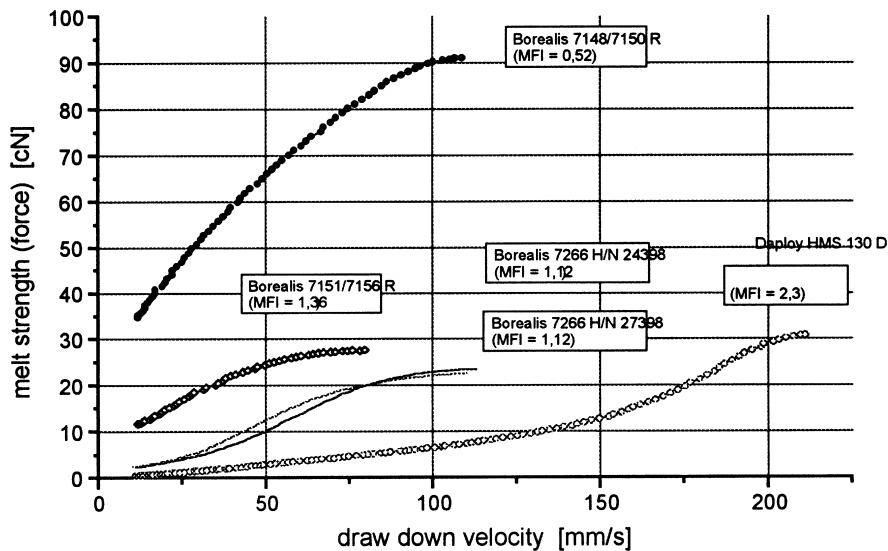


Figure 21. Melt strength and drawability at 200°C.

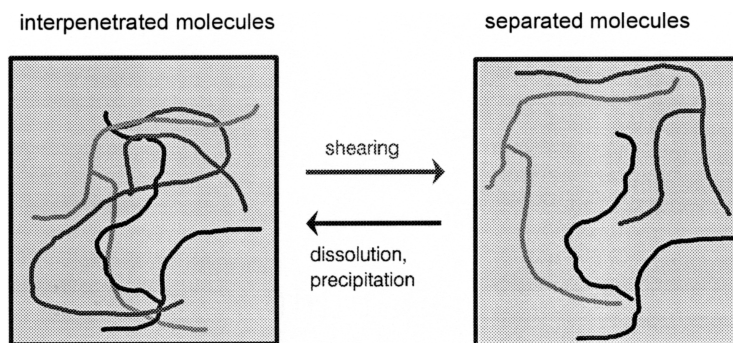


Figure 22. LCB-PP-entanglements.

In comparison to the normal linear i-PP grades, the HMS-PP shows a draw-down velocity, more than double, and an increasing of the melt strength during the drawing process. The increase of the melt strength during the driving, the so-called melt hardening process, is typical for all long-chain-branched PO's. In our example, a LCB-PP with a MFI of 2,3 which has unbranched a melt strength of 7 cN and a drawdown velocity of max. 100 mm/s reached values of 31 cN and 230 mm/s.

The high melt elasticity of the HMS-PP depends on our investigations from the entanglements of the long-chain branched as shown in Figure 22 (left side). By shearing the long-chain branched PIP during an extrusion process we found a reduction of the complex-viscosity and the G' and G'' values as demonstrated in Figure 23. In addition, the melt strength of the extruded material was decreased roughly. This can be explained by a break of the macromolecular chains or by an disentanglement of the long-chain branched macromolecules (Figure 22 right side) during the shearing in the extruder. To prove this effect, we solved the disentangled long-chain branched material in an aromatic solvent and precipitated it again. The precipitated i-PP leads again to the original high melt strength. That means that we could bring the material back to the original melt strength as it is schematically demonstrated with the below arrow in Figure 22, and a break down mechanism of the PP-chains can be excluded.

Figure 24 demonstrates the potential application fields of HMS-PP.

With the “Daploy” technology, there was an additional development of a range of new transparent thermoplastic elastomers, and grafting of new alloys by polystyrene and polymethylmethacrylate onto the polypropylene main chain. These results are presented in different papers [14].

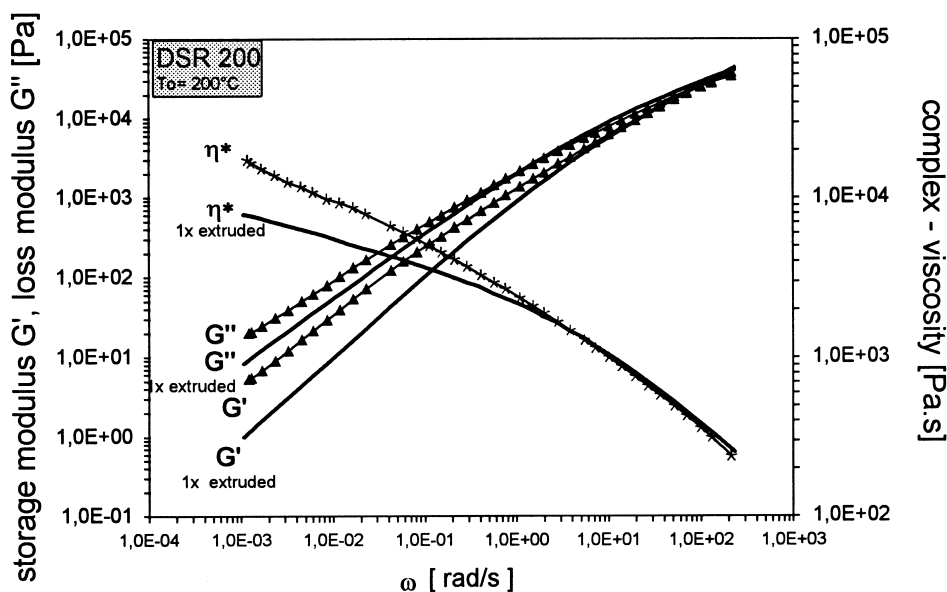


Figure 23. Dynamical rheology of HMS-PP.

At this time, the potential of this new technology at this time is bold, and needs additional research work based on experiments.

The basic research of these new developed materials was done in cooperation with universities and institutes. An external R&D cooperation network is very successful in transferring the know-how of the basic research of the universities and institutes in a short time to the market relevant product. From this viewpoint, Figure 25 is presents an overview of the different steps from the demands of the market and, on the other hand, from new basic results of our partners over an ideas generation, to products with new extended properties within the PD&I project status. PD&I is the Borealis project organisation to bring new basic results of outstanding products on the market.

CONCLUSION

The highest growth of the polypropylene consumption will continue in the next 10 years in Asia, Far East, and Middle East, while Europe shows a linear growth rate of the PIP consumption. The reduced margins in the PIP-com-

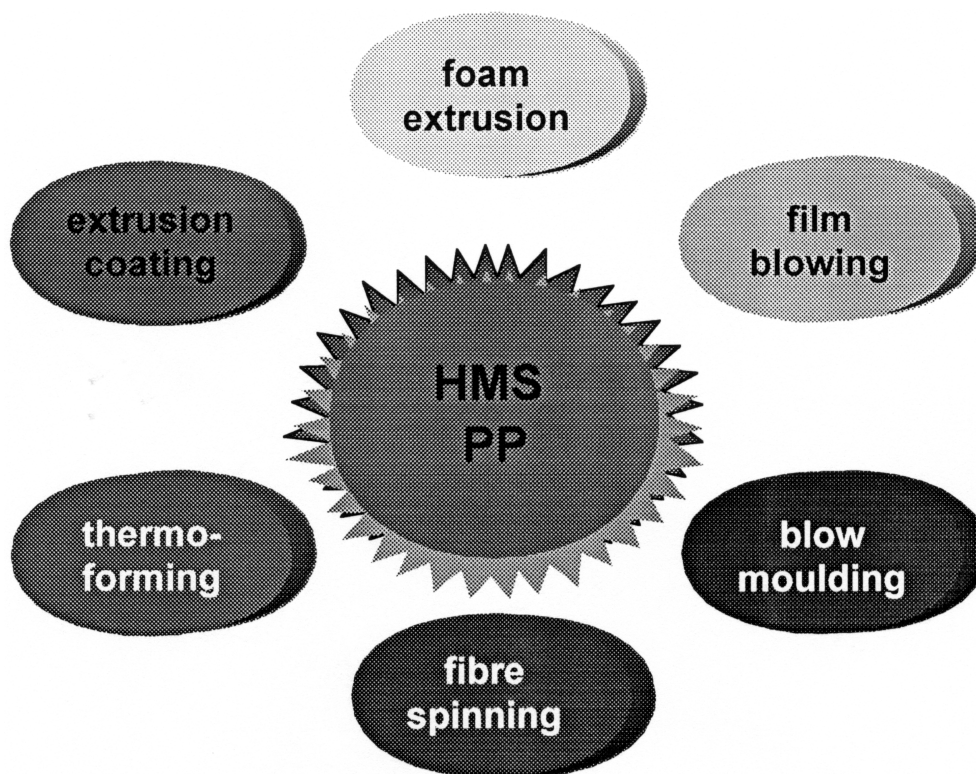


Figure 24. Potential applications of HMS-PP.

modity business and in search of new applications lead the producer to scoop out the full potential of the PIP-structure to develop higher value properties for the market. Nano and synthetic fiber composites on one side, and (α - or β -nucleation agents on the other side, open new property potentials. PP's from MC-catalysts of the first generation are in the market. Dual MC-catalysts and the second generation of catalysts for new materials with outstanding properties are now under development. The possibilities for new materials is being discussed. An interesting new market is presented for the new super soft PP's.

The chemical modification of i-PP powder after the polymerization step with radical reactions and additionally in combination with ionic mechanism will be a future development area. New technology concepts of combined polymerization and modification reactors with steered residence time distributions will follow the developments in the catalyst and chemical reaction progress. In reflection of the international development and the cost/research potential situation,

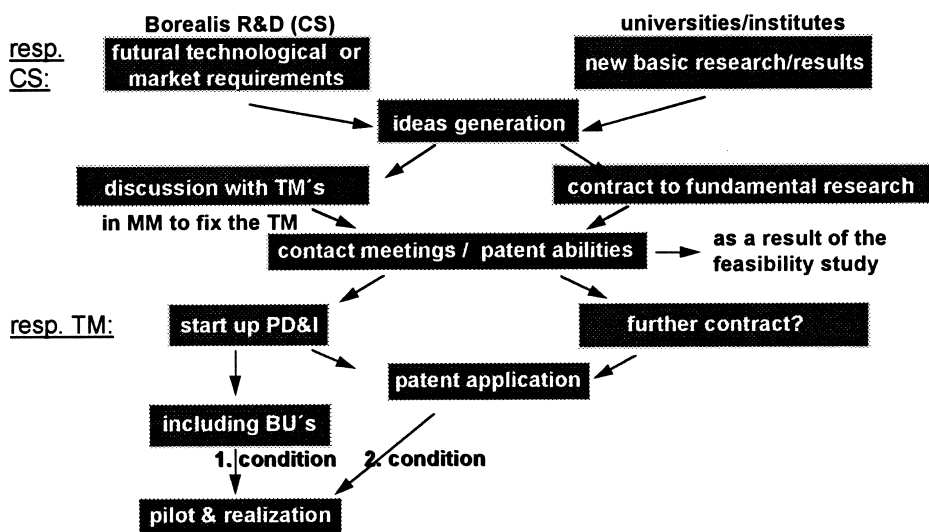


Figure 25. External R&D co-operations in new directions.

the special PP's will play an important role in the future market of the successful international

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