Process analysis for efficient rubber processing

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The IKV Rubber Technology group adopts a holistic approach. For the first time, the complete value chain of elastomer processing, from moulded part design via processing to extrusion and injection moulding, is brought together under one roof. The integrated machinery and equipment in the IKV’s technical centre represent a centre of competence for rubber technology. The technical centre for rubber consists of a fully-fledged mixing room with a laboratory roll mill, a laboratory internal mixer, a measuring mixer and a laboratory vulcanising press. In addition, the IKV’s extensive rubber testing facility at the Centre for Analysis and Testing of Plastics is available.

During rubber extrusion, a salt bath or a vulcanising unit combining hot air and microwaves can be situated downstream of an existing lab extruder with variable screw length. An online capillary rheometer is also available here for recording viscosity curves. Furthermore, the technical centre for rubber holds state-of-the-art injection moulding machines with clamping forces of 3,000 kN and 1,600 kN respectively for processing low and high viscosity elastomeric moulding compounds. The IKV Rubber Technology group combines research teams working in the areas of process and material modelling, mould technology and process development. This paper presents IKV research topics in rubber compounding and extrusion as well as elastomer injection moulding.

INFLUENCE OF MILLING ON THE PROPERTIES OF RUBBER COMPOUNDS

The process chain for the production of elastomeric parts starts with rubber compounding. Rubber compounding comprises all of the steps needed to prepare a rubber compound for processing by extrusion, compression moulding or injection moulding.

State of the art in the production of rubber compounds

To produce rubber compounds, crude rubber, active and inactive fillers, additives and crosslinking agents must first be processed into as homogeneous a mixture as possible. The composition of these raw materials, the so-called compound recipe, essentially determines the processing properties and the property profile of the finished product. However, the compound ingredients provided by the raw materials suppliers have properties that vary within a specific tolerance, which can have an impact on both the processing characteristics and the properties of the finished part. These raw material variations are found e.g. in the viscosity and molecular weight distribution of crude polymers and in the pellet hardness and specific surface area of carbon blacks.

The compounding ingredients are generally mixed in internal mixers and roll mills. These operate discontinuously, so even with consistent raw material properties they lead to property variations in the rubber compound since no two mixing operations will be exactly the same. Overall, this brings about batch variations in the rubber compounds which cannot be eliminated further along the process chain and can therefore lead to scrap (Figure 1). This entails considerable costs, particularly in times of rising raw material prices.

The compound properties can be divided into processing properties and material properties of the vulcanisate, which essentially characterise the eventual product properties. Both processing properties and component properties can be affected by the compounding process and depend primarily on the molecular weight of the polymer and on how well the fillers are distributed and dispersed. Variations can occur in the molecular weight, the molecular weight distribution...
and the degree of branching of polymers. Although a molecular weight distribution cannot be calculated directly from a Mooney viscosity, the Mooney viscosity of natural rubber (NR) takes qualitative account of molecular weight variation. The raw materials suppliers specify the Mooney viscosity of these polymers with a tolerance of ±5, and so they vary within a range of ten Mooney units.

With optimum process control, good filler dispersion is achieved with minimal degradation of the molecular weight. It is also extremely important that the filler dispersion and molecular weight of the polymer should be sufficiently similar in all rubber compounds for both the processing properties, especially for extrusion, and the eventual product properties to remain the same, as far as possible. With the variation in raw material properties, this represents a particular challenge.

Objective and results achieved

Although roll mills are widely used in rubber processing, the impact of the milling process on the rheological properties of the rubber compound and the mechanical properties of the end product is barely known, in contrast to the internal mixing process. However, by adjusting various parameters it has the potential to counteract and correct undesirable developments in compound properties, thereby reducing the quantity of scrap. Suitable milling parameters capable of affecting the properties of a rubber compound were determined so that a method could be set up in a subsequent step to enable the roll mill to have a direct and targeted influence on the compound in the production line. With the objective of discovering important process parameters and influences, the process parameters (nip width, friction, speed and milling time) were systematically varied while milling the rubber compounds with different crude rubber viscosities (Table 1).

By means of materials testing, differences were determined between the material properties of unmilled samples from the internal mixer and of the samples that had been milled as specified in the experimental design (Figure 2). The properties tested were the Shore hardness, Mooney viscosity, cure behaviour using a shearing disk viscometer (RPA) and the mechanical strength of the cured rubber compound on standard specimens in an accelerated tensile test. By evaluating the test results, it is possible to determine whether any significant influence of the different process parameters is detectable.

The rubber compounds for the investigations were produced in a laboratory internal mixer, type GK 1.5 E with PES-5 rotor geometry from Harburg-Freudenberger Maschinenbau GmbH, Freudenberg, Germany. The milling process was carried out using a laboratory roll mill of the 8” x 20” type from rubicon Gummitechnik und Maschinenbau GmbH, Halle/Saale, Germany. Natural rubber compounds with 40 phr carbon black and a sulfur-based curing system were produced for the tests. To investigate the effect on compound properties of varying polymer viscosity within a range of ten Mooney units, an SVR CV 60 (56 MU) and an SVR CV 60 (66 MU) were selected and processed with the same recipe and the same mixing specification.

The mixing process is divided into three phases. The crude rubber is first masticated for 2 min at a speed of 50 min⁻¹. At the start of the second phase, carbon black and minor chemical components are added to the internal mixer and incorporated into the masticated crude rubber over a period of 3 min at a speed of 50 min⁻¹. The added components raise the fill level in the mixing chamber, leading to a significant increase in power consumption. During this...
phase, the temperature in the mixing chamber rises to over 90°C. The hydraulic ram is pushed back by the mixing volume and only reaches its end position after about 50 s. The purpose of the final phase of compound production in the internal mixer is to incorporate the curing system. The curing chemicals sulfur and vulcanisation accelerators are fed into the internal mixer and, to avoid the risk of the compound starting to cure, the speed is reduced to 20 min⁻¹. As a result, the internal mixer’s power consumption decreases and the temperature in the mixing chamber drops to non-critical values of less than 80°C. Nevertheless, the duration of this phase is limited and it ends after 2 minutes with ejection of the lump of compound.

The diagram in Figure 3 shows the measured values from the Mooney test for the unmilled comparison samples of 16 compounds for each rubber specification. All of these unmilled rubber compounds were processed in the same way and should have approximately the same viscosity for each grade of rubber. However, variations in the batchwise mixing process and in the properties of the crude rubber, as a natural product, lead to some variation in the measured Mooney viscosity values. In the following diagram, therefore, for determining significances, the percentage difference between milled samples and comparison samples is evaluated using statistical tools, rather than the absolute measured values. It is interesting to note that the viscosity of the crude rubber in the compound has no significant effect on the viscosity of the unmilled rubber compound, as the curves for the two viscosity levels used (56 MU and 66 MU) are very close together.

The investigations into the milling process show that the results are affected in particular by variations in the nip and the milling time. Evaluation of the confidence intervals (Figure 4) shows that nip width and milling time have a highly significant influence on the Mooney viscosity of the rubber compound (green line). However, the combination AB and the speed (B) on its own are just outside the lower confidence bound of 95% (red line).

With a decreasing nip or increasing milling time, both the viscosity of the uncured rubber compound and the tensile strength of the cured rubber samples are reduced. No significant influence on hardness or curing characteristics can be observed. Similarly, the process parameters of friction and speed have no reliably detectable impacts on the measured results within the range of variation used for the test. Focusing on the parameters of nip width and milling time, the results obtained can be utilised to carry out further investigations. The recording of process data from the roll mill, such as its power uptake, may also be of interest with a view to further process automation.

Methods for extruding rubber are being developed and optimised. The key topic of hardware technology in rubber extrusion will be discussed below, with particular reference to the long-established technology of the pin-type extruder.

**IMPROVEMENTS IN THE MIXING CHARACTERISTICS OF PIN-TYPE RUBBER EXTRUDERS**

Together with Kunststofftechnik Paderborn (KTP), the IKV is currently involved in a research project which is systematically investigating ways of improving the mixing characteristics of a pin extruder by adjusting and varying the pin geometry.

**Basic principles behind the pin extruder**

Different types of extruders are employed in rubber processing. They are categorised as cold-feed or hot-feed extruders according to the feed method. Hot-feed extruders are characterised by their short overall length and are fed with preheated rubber strips. Nowadays, they
are only used for specific applications such as processing highly viscous compounds. Cold-feed extruders have been used successfully in the rubber processing industry since the 1970s. They have longer screws since there is no preheating unit. Because cold-feed extruders with a smooth barrel have to be very long in order to ensure good thermal homogeneity of the rubber compounds, there are various approaches for improving thermal homogeneity by means of additional elements in the screw channel. The use of pin extruders is currently state of the art. In a rubber extruder with a pin section, cylindrical pins protrude into the screw channel and the screw flights have corresponding cut-outs (Figure 5).

Use of a pin section improves the thermal homogeneity of the extrudate. Pin extruders are very versatile as they can process polymers with a wide range of viscosities. The improved mixing action is partly attributable to the rearrangement effects on the rubber compound at the cylindrical pins. The more pins there are in the barrel, the better the homogeneity achieved. At the same time, a very large number of pins in the barrel results in lower extruder throughput for the same speed because of the increased pressure required. Good homogeneity of the rubber compounds is important owing to the high specifications demanded of the eventual components. At the same time, high throughputs are essential for economic production. However, the more the output of a pin extruder is increased, the more the quality of the components will be reduced. The output efficiency of a pin extruder is therefore always limited by extrudate quality.

**Objective and results achieved**

The aim of the project with KTP is to provide a novel pin arrangement for a cold-feed rubber extruder for the economic production of rubber extrudates with improved homogeneity of the polymer material. The mixing action of the pins is to be improved so that fewer pins are needed in the pin barrel to achieve good extrudate quality. By reducing the number of pins, the throughput can be increased while maintaining good component quality. In addition, the new pin arrangement being investigated in this research project can be used as a low-cost upgrade to existing equipment. To make further improvements to the mixing characteristics of a pin extruder, it is very important to consider the flow and dispersion characteristics of the rubber compounds in the extruder, and particularly at the pins.

One of the factors in achieving this goal is to establish an evaluation method for determining dispersion quality, since the benefit of optimised pin geometries can only be quantified by objective and reproducible determination of dispersion quality. In general, the dispersion of a substance is categorised along a spectrum ranging from micro- to macro-dispersions according to the size of the particles. There are many methods for the analytical determination of the dispersion quality of filler particles in a rubber compound. These methods have various disadvantages, and so a suitable characterisation method must be chosen for each individual case. Figure 6 provides an initial overview, showing the most common methods in relation to the size of the filler particles [1].

One way of characterising a macro-dispersion is to prepare clean-cut surfaces of the compounds, which are then analysed by reflected light microscopy [2]. Clean-cut surfaces are prepared by cutting through the vulcanisate with a sharp blade to give a double surface impression of the filler agglomerates [3]. In the IKV’s investigations, an EPDM rubber compound with 50 phr of carbon black N326 is being studied in order to select suitable methods for determining dispersion. It can be seen clearly in Figure 7 that a double surface impression of the filler particles is indeed produced by the razor cut. According to the theory, the carbon black agglomerates always remain on one of the two cut surfaces. In other words, wherever there is an agglomerate, either an indentation or a protrusion appears. According to ISO 11345, for example, it is then possible to rate the specimens visually on a scale of 1 – 10 in terms of carbon black dispersion. The disadvantage of this method, however, is that it is time consuming and only qualitative. The German Institute for Rubber Technology e.V. (DIK) in Hanover has therefore developed a method of evaluating the cut surfaces. In this method, the image taken by the microscope is binarised using a grey threshold and then processed in terms of the number and area of the particles to give either histograms or a dispersion index. The challenge lies in adjusting the grey threshold for each specimen independently of the user.

Figure 8 compares the size distributions of carbon black agglomerates before and after
extrusion in order to quantify the dispersive mixing action of a pin extruder. Analysis was performed using the DIAS method. It is clear that significantly more agglomerates in the range greater than 4.5 µm are present before extrusion than after. The agglomerates that were broken down during extrusion are found in the histogram in the range below 4.5 µm, clearly illustrating the distributive action of the pins.

A confocal laser microscope is currently being used at the IKV to analyse the images of the clean-cut surfaces. Since the laser microscope produces topographic images of the cut surface, the objective is to provide a method in which the threshold value can be determined objectively on the basis of the topographic information. Based on the method that has been developed to evaluate dispersion quality, the improvement in dispersive mixing action achieved by varying the pin geometry can thus be validated over the course of the project. Having shown in this section how existing rubber extrusion equipment is being developed at the IKV, the next section will show how elastomer films or membranes can be produced much more efficiently by developing new equipment.

ENERGY EFFICIENCY IN INJECTION MOULDING

In the rubber-processing industry, the value added costs are determined by resource-intensive manufacturing processes. The heat energy needed for vulcanisation in particular represents a major part of the total energy requirements during manufacture. In contrast to thermoplastics processing, elastomers obtain their dimensional stability not by energy extraction through cooling but by input of heat, and therefore energy. However, in today’s world it is absolutely essential to use energy efficiently for environmental and economic reasons. Existing processes must therefore be analysed, evaluated and optimised in terms of their energy consumption.

One of the most important processes for making technical elastomer products (TEPs) is injection moulding. The injection moulding of elastomers is an energy-intensive process. Most of the energy is consumed by the drives for machine and tool movements and by the temperature control systems. To analyse the actual energy status precisely, in collaboration with Freudenberg Forschungsdienste SE & Co. KG, Weinheim, Germany, the actual energy requirements of the heating platens, material heating system, hydraulic pumps, control system and the overall system were recorded during mould heating and over 22 cycles for producing a butterfly valve seal (Figure 9).

In large-scale production, a twin cavity mould is heated to 180°C by heating platens mounted on the machine, resulting in heating times of about 1.5 h with a heating energy requirement of about 15 kWh. For an ongoing cycle the energy requirement was about 0.5 kWh, of which 44.37% was attributable to the heating platens, 26.67% to the drives, 21.19% to material heating and

![Figure 6. The most common methods for analysing filler dispersion [1]](image)

![Figure 7. Double fingerprint of a razor cut section of an EPDM specimen with carbon black N326](image)

![Figure 8. Size distribution of carbon black agglomerates before and after extrusion (1.5 – 100 µm)](image)
7.77% to the control system. Temperature control of material and mould therefore accounts for over 65% of the energy requirement at present and offers great potential for improvements in production efficiency.

**Dynamic temperature control to increase the initial melt temperature**

One way of reducing the energy requirement for temperature control in the injection moulding process is to raise the initial temperature of the melt [4]. An increased initial melt temperature results in a reduced heating time and therefore a shorter process cycle time. However, the initial melt temperature can only be raised in the plasticating unit to a level low enough to rule out premature curing. In collaboration with industrial partners, the IKV has developed a dynamic temperature control element to achieve a further increase in the initial melt temperature without premature curing. This temperature control element permits highly dynamic temperature control of the rubber melt before it enters the mould and is used instead of a cold runner feed system. The temperature profile during the injection moulding cycle provides for energy input by means of heat conduction during the injection phase. The wall of the temperature control channel is heated by means of a copper induction coil. To compensate for the large temperature gradient in the material from the outside to the inside due to the low thermal conductivity of rubbers, the flow channel in the temperature control element has a shear gap which is twisted and tapered in the direction of flow. This shear gap means that additional dissipative heating can be achieved together with homogenisation of the material temperature. After the injection phase, the temperature control element is cooled using an integral water cooling system so that the material in the temperature control element cannot cure.

**Study of the dynamic temperature control element**

The copper induction coil is powered during operation by an MFG 15 multi-frequency generator manufactured by eldec Schwenk Induction GmbH, Dornstetten, Germany. The generator has a maximum power output of 15 kW. The coil has to be actively cooled during operation to avoid overheating, leading to the surface temperature profile of the dynamic temperature control element shown in Figure 10. The large temperature differences between the inductively heated temperature control element and the cooled induction coil are clearly visible.

The heating characteristics of the dynamic temperature control channel depend on the generator output. The coil design means that this is limited to 60% of the maximum value. Figure 11 shows the heating and cooling behaviour of the system as a function of generator output. The high thermal dynamics with a high power output can be seen clearly, permitting the temperature control that is needed here and in particular preventing partial curing in the channel while the component is being vulcanised in the mould. The temperature was recorded by means of thermographic imaging at the midpoint of the element's surface.

The injection material flows through the temperature control element at a temperature of 50°C and a water-based system adjusts the temperature control element to 60°C. In these series of measurements, the temperature of the melt on emerging from the temperature control element is recorded by infrared camera. Figure 12 shows the melt exit temperature as a function of generator output. At high generator outputs, an increase of the melt exit temperature per cycle in large-scale production of a butterfly valve seal.
temperature to over 80°C is measured. The temperature control channel is twisted by 180° along the flow path and, in addition, the flow channel has a polygonal cross-section. As a result, the injection material is in rotation as it emerges. The fixed measuring point detects the protrusions and depressions in the strand of material, where there are localised differences in reflectivity which explain the periodic fluctuations in measured melt temperatures during the injection phase. The illustrated increases in initial melt temperature occur with a 30 s heating time of the temperature control channel during the vulcanisation phase in the mould. The associated energy requirement is illustrated in Figure 13. As the generator output – and thus the energy requirement – increases, the maximum and average temperatures of the injection material also rise. The energy required, in Wh, is in the double-digit range.

CONCLUSIONS

Injection moulding is an energy-intensive process in which a large part of the energy consumption is needed for controlling the temperature of material and mould. To reduce the amount of energy required, the initial melt temperature can be raised, resulting in shorter heating and process cycle times. With the aid of a dynamic temperature control element, the initial melt temperature can be raised without premature curing prior to entering the mould. Energy input by means of heat conduction and dissipation in a tapering, twisted shear gap enables temperature to be managed homogeneously and dynamically.

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Figure 11. Heating and cooling behaviour as a function of generator output

Figure 12. Energy requirement per cycle in large-scale production of a butterfly valve seal

Figure 13. Initial melt temperature as a function of energy requirement with 30 s heating phase

REFERENCES


