THE INFLUENCE OF DIFFERENT DIE GEOMETRIES ON THE EXTRUSION PROCESS OF HIGH-CONSISTENCY SILICONE RUBBER

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Abstract

Silicone Rubber and especially High-Consistency Silicone Rubber (=HCR), are typically processed in the extrusion process. Due to the high requirements in terms of the material properties and the geometric dimensions, a fundamental knowledge of the whole process including experiences in tool design are essential.

In this study, HCR with different Shore-hardnesses are extruded on a vertical silicone extrusion line with various breaker plates with different length to diameter ratios (=L/D-ratio) in order to analyze the influence on the whole extrusion process.

It has been shown that soft materials, regardless of die geometry, achieve higher throughputs compared to harder compounds. Increasing counter pressure, e.g. due to longer die lengths, reduces the volume flow rate per revolution and reduces the throughput. Tools with a small L/D ratio achieve the highest throughput. With regard to die design, it can be seen that dies with a smaller L/D ratio have clear advantages: due to their short length, they represent a smaller pressure consumer. As a result, the dwell time in the extruder is shorter and the risk of scorch is reduced. The absolute value of the swelling behavior is larger, but can be predicted with high accuracy. Shorter tools also show less flow instabilities.

Introduction

Rubbers are an often amorphous polymer which crosslinks to an elastomer by chemical crosslinking. Rubbers are divided into natural rubber and synthetic rubber [1, 2]. Silicone rubber belongs to the group of synthetic rubbers and, due to its unique property profile, offers broad application possibilities in a large number of industrial sectors. Silicone rubbers are usually processed by injection moulding, pressing or extrusion [3, 4]. In terms of volume, extrusion accounts for the largest share [5, 6]. Silicone rubber is used as a sealant or adhesive in the construction industry. In the electronics industry, the high thermal and chemical resistance of many components has an effect. In the medical technology, automotive or food industries, silicone rubbers are often used as media-promoting components in the form of hoses or profiles [7 to 10]. All applications have in common that high demands are made on the material as well as on the geometric dimensions and thus on the processing process.

Rauwendaal et al [11] stated that despite the growing industrial importance of silicone rubber, little literature can be found on the basic understanding of the extrusion process.

Lehnen [12] diagnosed that "problems" during processing are not solved by optimizing the process, but primarily by modifying the recipe. Depending on the branch of industry, changes in the material recipe can have far-reaching consequences, for example with regard to compliance with legal regulations. In contrast, the optimization of the manufacturing process requires less time and money. In addition, a material change usually implies a tool adaptation or complete redesign. This in turn represents an additional expense.

Objectives

A profound understanding of the extrusion process, including experience in tool design, helps to reduce rejects in the field of high-consistency silicone rubber (=HCR) extrusion due to optimized and thus timely improvement processes. In the field of tool design, the running-in effort can be minimized by reducing iterations. "Problems" during processing can be solved promptly and without material changes by adjusting the process parameters with the help of a profound understanding of the process. As a result, ecological and economic resources are conserved. The goal of this study is the analysis of the influence of different materials and tool designs on the extrusion process of HCR. In addition to the analysis of the pressurethroughput characteristics as a function of different die geometries, the scorch risk is to be evaluated on the basis of the dwell times determined. The influence of the tools on the swelling behavior will also be examined.

Experimental

All extrusion tests were carried out on a vertical silicone extrusion line EEK 32.12 S - 4/90SIR of Rubicon Gummitechnik und Maschinenbau GmbH/Germany. As shown in Figure 1, the silicone extruder stands elevated on a platform and enables vertical extrusion from top to bottom with the aid of a deflection tool. The advantage of this arrangement is that the not yet cured material does

not collapse and stick together after leaving the die due to the lack of dimensional stability. This is particularly advantageous for large-volume hollow sections [4, 13].

The feed roll of this smooth-bore extruder is fed with strip-shaped HCR. The double-flight screw with a diameter of 32 mm has a constant depth. There is no compression. The extruder has a length of 12 D [14]. The extruder and the deflection tool, which carries the actual forming tools, are actively cooled with water. The extrudate leaves the deflection die at an angle of 90° downwards and is guided through an infrared tunnel (=IR tunnel). A sag control is installed at the lower end of the tunnel, which enables a defined sag to be set. By adjusting the sag, the downstream conveyor belt pulls off the extrudate faster or slower and thus controls the height of the sag. The greater the sag, the more material - the uncured part of the extrudate - is drawn into the length; thus reducing its outlet cross section [4].

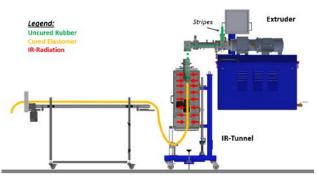


Figure 1: vertical Silicone Extrusion Line from Rubicon Gummitechnik & Maschinenbau GmbH/Germany

All extrusion tests were carried out under laboratory conditions. Care was taken to ensure proper feeding of the extruder: During the tests there was sufficient material in the feed section - any relining was avoided. The raw data of the extruder were checked for correctness before each evaluation: A constant screw speed with a constant die should also lead to a largely constant pressure above the test batch. An overview about the dies used in this study is shown in Figure 2.

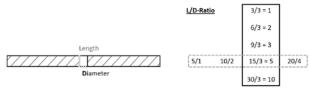


Figure 2: Overview of the breaker plates used incl. L/D-ratio information

Materials

All HCRs investigated in this paper are commercially available types from various material manufacturers.

Hardness grades between 10 and 60 Shore A were selected in order to identify and quantify the influence of different hardness and resulting filler contents on the processing properties. The manufacturers do not specify the exact material composition.

Table 1. Selected properties of the employed materials

Material	Hardness [Shore A]	Density $\left[\frac{\mathbf{g}}{cm^3}\right]$	Curing System [-]
ELASTOSIL® R 401/10	13	1,07	Peroxide
Synersil 109011	40*	1,10	Platin- Cured
ELASTOSIL® R 401/60	61	1,15	Peroxide

^{*=} self-determined values

Characterization

The breaker plate is widely used in the rubber sector and has also proven itself for processing silicone rubber [3, 13, 15]. An elementary disadvantage is a high input effort in an iterative process paired with reworking on the tool. Various aperture tools with different L/D ratios are used for the analysis of suitable tool technology.

The geometric dimensions of the extrudate were measured and recorded after leaving the die. A non-contact measuring system consisting of an ODAC 33 Trio - a triaxial measuring head from Zumbach Electronic AG/ Switzerland - and the ODAC Online 32 software from IPC/Germany was used.

Results and Discussion

Tools primarily affect the design of the extrudate geometry. However, they also have an impact on the extrusion process, as they influence, for example, the counterpressure or the dwell time of the material in the extruder. Therefore, the influence of dies with different L/D ratios on the extrusion process will be identified and analysed in the following. The following investigations refer to the extrusion of a rotationally symmetrical full profile with different outer diameters.

Pressure-Throughput Characteristic

Figure 3 and Figure 4 show the pressure-throughputcharacteristics of the extruder as a function of various dies and high-consistency silicone rubbers. The solid lines represent the so-called die characteristics and the dotted lines the extruder characteristics [16].

The more rigid an extruder, i.e. the pressure build-up is largely independent of the die and the resulting counterpressure, the more parallel the die characteristics are to the abscissa [17]. It becomes clear that the conveying be-

haviour here depends on the back pressure. With increasing Shore-hardness and green strength of the rubber, less material is conveyed at the same screw speed. The throughput decreases with increasing Shore-hardness (at the same screw speed) due to increasing green strength [15, 18]. The melt pressure was constant within the individual batches/operating points. The high gradient of the tool characteristics also shows that the influence of the screw speed on the melt pressure is lower for soft materials compared to harder silicone rubbers.

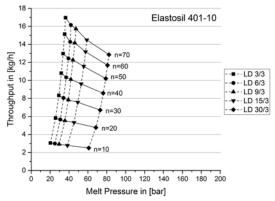


Figure 3: Pressure-Throughput Characteristic of Elastosil 401-10

Across all materials it becomes clear that the melt pressure increases with decreasing tool diameter. With increasing die length (and same diameter) the melt pressure decreases due to the increasing pressure consumption by the die [19, 20]. The extruder characteristics of Elastosil 401-10 and Synersil 109011 show a high linear correlation. The coefficient of determination per screw speed stage is >97%.

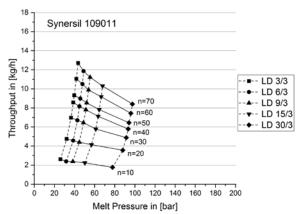


Figure 4: Pressure-Throughput Characteristic of Synersil 109011

The elementary function of extrusion according to Lehnen [12] is to achieve the highest possible throughput while maintaining high extrudate quality. Regardless of the extrudate quality, short die lengths achieve significantly

higher throughputs due to the lower (die) counter pressure [21]. Softer materials show higher throughputs with the same process parameters (and same screw geometry) compared to harder compounds [18]. The screw geometry must be adapted for harder compounds in order to improve material conveying and avoid relining. According to [22], the influence of the screw design on the conveying behaviour is greater compared to the material behaviour or the process parameters. According to [3, 17], deepcut screws are well suited to increase throughput.

Specific volumetric flow rate

The specific volume flow rate represents the specific volume per screw revolution and adjusts the often displayed specific throughput for density. The results for different tools for the Elastosil 401-10 are shown in Figure 5. The Elastosil 401-10 and the Synersil 109011 clearly show that the volume flow rate per revolution decreases with increasing speed. However, the total throughput increases with increasing screw speed.

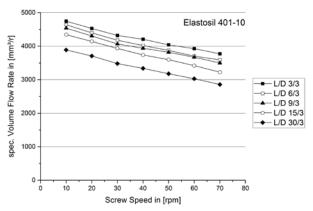


Figure 5: Specific Volume Flow Rate as a function of the Screw Speed for Elastosil 401-10

With the same L/D ratio and increasing tool diameter, the specific volume per revolution increases due to the increasing tool back pressure. With the same diameter and increasing tool length, the volume flow rate decreases due to the increasing back pressure. An almost linear relationship can be seen here. The Elastosil 401-60 shows a differentiated picture (compare Figure 6): The specific volume flow rate decreases up to a screw speed of 30 rpm. It then also increases with increasing screw speed. This indicates that the screw is not completely filled due to the highly viscous material [23]. The screw used here is not capable of conveying harder materials sufficiently and of constantly supplying the tool with material. Despite the incomplete screw filling, the pressure fluctuations across all tools for the Elastosil 401-60 are within the usual range (<3%).

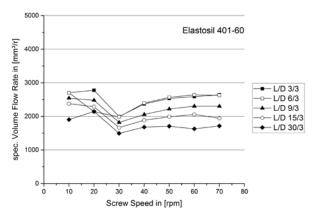


Figure 6: Specific Volume Flow Rate as a function of the Screw Speed for Elastosil 401-60

Dwell time and melt temperature to estimate the risk of scorch

The average dwell time is an important factor especially in extrusion: In the area of thermoplastic extrusion, thermal damage to the material caused by excessive temperatures in combination with long dwell times should be avoided [24]. In the area of elastomer extrusion, the material to be processed must not be cured in the extruder or in the die [25, 26].

For this reason, the average dwell time of Elastosil 401-60 was determined by means of colour tests as an example. For this purpose a masterbatch was added to the HCR and the time between the material being captured by the screw and leaving the tool was measured – similar to [27]. The mean dwell time (see Figure 7) follows the trend of the throughput in reverse logic. The lower the throughput, the higher the average dwell time in the extruder [3, 22]. The dwell time decreases as the die diameter increases. The dwell time increases with increasing tool length. Across all tools, the dwell time moves at a screw speed of 10 rpm at max. 600s and at 70 rpm at approx. 100s, with an average standard deviation of 1.5%.

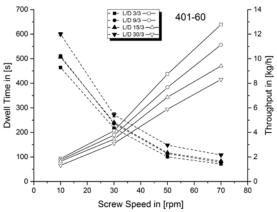


Figure 7: Dwell Time and Throughput as a function of Screw Speed

A well-founded estimation of the risk of scorch cannot only be carried out via the dwell time, but must always be carried out in combination with the corresponding melt temperature. For this reason, the melt temperatures after leaving the die were recorded and evaluated with the aid of an IR camera.

As an example, Figure 8 shows the results for a short tool (L/D ratio of 1) and Figure 9 shows the results by using the tool with the largest L/D ratio of 10. In all tests, the set-cooling and screw temperatures were kept constant (20°C). With increasing screw speed, the melt temperature increases across all materials and tools due to the dissipative heating by the screw [4, 28, 29]. The active cooling of the barrel and screw ensures that the melt temperature does not rise for all materials examined here above approx. 36°C despite the high speed and long residence time of the rubber in the extruder. On the basis of the values determined here for the average dwell time and maximum melt temperature, the risk of scorch of the HCR (with the peroxide crosslinking systems used here) in the extruder (with switched-on cooling) is negligible.

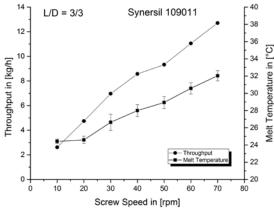


Figure 8: Throughput and Melt Temperature as a function of Screw Speed for L/D 1 with Synersil 109011

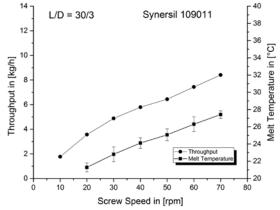


Figure 9: Throughput and Melt Temperature as a function of Screw Speed for L/D 10 with Synersil 109011

The effect of selected L/D-ratios on the geometric dimensions of the extrudate

The following results show the swelling behavior as a function of different L/D-ratios in the real extrusion process. With increasing L/D ratio and the same die diameter, the swelling decreases continuously. According to Michaeli [19], an extension of the tool length represents an enlargement of the parallel guide or the ironing zone, which promotes the reduction of reversible elastic deformations. The disadvantage of this design change is the increased pressure consumption by the tool [20]. A comparison of different materials also shows that the strand expansion decreases with increasing Shore hardness (and filler content) [6]. Within the investigated materials an almost linear correlation between strand expansion and shear rate can be seen. In the case of soft materials (here Elastosil 401-30 - Figure 10), the linear relationship is more than 94%, especially for the small L/D ratios (<3). The harder materials show comparable linearity values for all L/D ratios – see Figure 11.

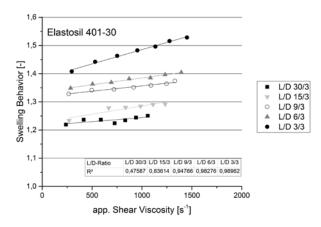


Figure 10: Swelling behavior of Elastosil 401-30 using dies with different L/D ratios

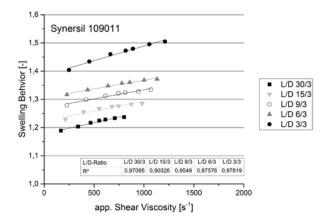


Figure 11: Swelling behavior of Synersil 109011 using dies with different L/D ratios

Conclusions

The goal of this study is the analysis of the influence of different materials and tool designs on the extrusion process of HCR. It has been shown that soft materials, regardless of tool geometry, achieve higher throughputs compared to harder compounds. Increasing counter pressure, e.g. due to longer tool lengths, reduces the volume flow rate per revolution and reduces throughput. Tools with a small L/D ratio achieve the highest throughput. The analysis of the dwell times in connection with the melt temperature shows that the danger of premature curing is negligible.

With regard to die design, it can be seen that dies with a smaller L/D ratio have clear advantages: due to their short length, they represent a smaller pressure consumer. As a result, the residence time in the extruder is shorter and the risk of scorch is reduced. The absolute value of the strand expansion is larger, but can be predicted with high accuracy. Shorter tools also show less flow instability.

The processing of different HCR types shows clear differences in the resulting process variables. Simple breaker plates are highly suitable for the processing of HCR.

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References

- [1] F. Röthemeyer and F. Sommer, Kautschuk-Technologie. Werkstoffe, Verarbeitung, Produkte, München, Hanser (2013).
- [2] M. Bogun, Untersuchungen zur kontinuierlichen Herstellung von Kautschukmischungen basierend auf Rubber/Filler-Composites am Doppelschneckenextruder, Doctoral Thesis, Halle (2005).
- [3] A. Limper, P. Barth and F. Grajewski, Technologie der Kautschukverarbeitung, München, Hanser
- [4] SKZ Würzburg: Fester und flüssiger Silikonkautschuk: Eigenschaften, Verarbeitung, Qualitätssicherung, Würzburg (1991).
- [5] S. Brockhaus, Theoretische und experimentelle Untersuchungen zum Temperatur- und Durchsatzverhalten von Kautschukextrudern, Doctoral Thesis, KTP Paderborn, Shaker (2017).
- [6] B. Crawford, A.P. Doherty, P.L. Spedding, W. Herron and M. Proctor, Viscosity of siloxane gum and silicone rubbers, Asia-Pacific Journal of Chemical Engineering, 5, 6 (2010).
- [7] Ceresana eK, Marktstudie Silikone, http://www.ceresana.com/de/marktstudien/kunststof fe/silikone/, last retrieved on: 09.10.2018.

- [8] BIW Isolierstoffe GmbH, Products, https://www.biw.de/silicon-extrusion/, last retrieved on: 09.10.2018.
- [9] Raumedic AG, Products, https://www.raumedic.com/de/impressum/, last retrieved on: 09.10.2018.
- [10] M + S Silicon GmbH & Co KG, Products, http://www.ms-silicon.de/ms-sil/team-ms-silicon/, last retrieved on: 09.10.2018.
- [11] C. Rauwendaal, P.J. Gramann, B.A. Davis, and T.A. Osswald, Polymer extrusion, München, Hanser (2014).
- [12] J.P. Lehnen, Beitrag zur Verarbeitung von Kautschukmischungen auf Einschnecken-Extrudern, Doctoral Thesis, RWTH Aachen (1970).
- [13] A. Colas, R. Malczewski, and K. Ulman, Silicone Tubing for Pharmaceutical Processing, https://pdfs.semanticscholar.org/5464/3ff1e7683844 c58cc733373038ab36bf909b.pdf, last retrieved on 16.10.2018.
- [14] rubicon Gummitechnik und Maschinenbau GmbH, Technical Documentation: Small Scale-Silicone Extrusion Line, Halle (2015).
- [15] T. Tylor, A processing guide to silicone rubber extrusions, https://www.thefreelibrary.com/A+processing+guid e+to+silicone+rubber+extrusions.-a021159375, last retrieved on 16.10.2018.
- [16] D. Schramm, Möglichkeiten und Grenzen einer verbesserten Prozessbeschreibung für Kautschukextruder, Doctorial Dissertation, KTP Paderborn, Shaker (2004).
- [17] T. Hallmann, Untersuchung des Prozessverhaltens neuartiger Einzugszonen-Konzepte für Kautschukextruder, Doctoral Thesis, Institut für Leichtbau mit Hybridsystemen, Aachen, Shaker (2013).
- [18] J. Dick and R. Annicelli, Rubber technology. Compounding and testing for performance, München, Hanser (2009).
- [19] W. Michaeli, Extrusionswerkzeuge für Kunststoffe und Kautschuk. Bauarten, Gestaltung und Berechnungsmöglichkeiten, München, Hanser (2009).
- [20] M.R. Kamal and H. Nyun, Capillary viscometry. A complete analysis including pressure and viscous heating effects, Polymer Engineering and Science, **20**, 2 (1980).
- [21] K. Westermann, Extrusion von physikalisch geschäumten Kautschukprofilen, Doctoral Thesis, RWTH Aachen, Mainz (2009).
- [22] A. Limper, Methoden zur Abschätzung der Betriebsparameter bei der Kautschukextrusion, Doctoral Thesis, RWTH Aachen (1985).
- [23] A. Limper and T. Wilhelmsmeyer, Prozessanalyse des Plastifizierens von Kautschukmischungen mittels Zahnradpumpen. Teil 1, KGK, **56**,1-2 (2003).

- [24] K. Kohlgruber, Der gleichläufige Doppelschneckenextruder. Grundlagen, Technologien, Anwendungen, München, Hanser (2016).
- [25] L. Köster, H. Perz and G. Tsiwikis, Praxis der Kautschukextrusion, München, Hanser (2007).
- [26] E. Brunnhofer and R.Sander, Das Extrusionswerkzeug. Leitfaden zur Gestaltung und Handhabung. Kunststofftechnik, Düsseldorf, VDI (2000).
- [27] W. Michaeli, Extrusion von Kautschukprofilen mittels Direktbegasung mit überkritischem Stickstoff als physikalisches Treibmittel, AiF- Abschlussbericht (2005).
- [28] A. Funk, Characterisation of solid silicone rubbers for the processing in the extrusion blow molding process, Doctoral Thesis, RWTH Aachen, Mainz (2015).
- [29] A. Limper and D. Schramm, Process Description for the Extrusion of Rubber Compounds - Development and Evaluation of a Screw Design Software, Macromolecular Materials and Engineering, 287, 11 (2002).