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Mehrkomponentenspritzguss von Kleinstbauteilen mit Silikon, Seite 10

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Efficient Method for Characterizing the Service Lifetime of Rubber Seals

Rubber · Seal · Lifetime · Crack · Initiation

Seal applications require rubber compounds capable to endure numerous cycles of deformation without crack initiation. A lab testing equipment named Intrinsic Strength Analyzer (ISA™) has been used to investigate the crack initiation energy of different rubber compounds typically used for seal applications. Four compounds were investigated. The compounds were based on two different types of ethylene-propylene-diene-monomer (EPDM), varied quantity of carbon black (CB) and varied types and quantities of curing agents. On hand of the compounds analyzed could be shown first of all, the intrinsic strength is an indicator for service lifetime of seals. Secondly, the predominant effect of varied rubber type on the crack initiation energy compared to the lower influence of other chemicals used has been observed.

Effiziente Methode zur Charakterisierung der Gebrauchsdauer von Gummidichtungen

Gummi · Dichtung · Lebensdauer · Rissinitierung

Dichtungsanwendungen erfordern Gummimischungen, die geeignet sind, zahlreiche Deformationszyklen ohne Rissentstehung auszuhalten. Das Labortestgerät ISA wurde eingesetzt, um die Rissentstehungsenergie verschiedener Gummimischungen zu untersuchen. Die Gummimischungen basierten auf zwei verschiedenen Ethylenpropylendienkautschuken (EPDM) mit variierenden Anteilen an Ruß und variierenden Typen und Anteilen von Vernetzungschemikalien. Einerseits konnte an analysierten Gummimischungen gezeigt werden, daß die intrinsische Festigkeit ein Indikator für die Gebrauchsdauer der Dichtungen ist. Andererseits, ist der vorherrschende Effekt der variierten Kautschuktype auf die Rissentstehungsenergie im Vergleich zu einem geringeren Effekt der übrigen eingesetzten Chemikalien beobachtet worden.

Figures and Tables: By a kind a approval of the authors.

Motivation

In seal applications predominantly rubber material is used. Seals are exposed to static and dynamic load conditions while their service lifetime. The behaviour of the rubber material is significantly influencing the service life. To guarantee safe operation and long service lifetime it is important to estimate the durability of these products and how long a seal will last under its planned service conditions. In case of high dynamic and varied loading conditions the traditional test fatigue crack growth analysis is the preferred method to estimate the resistance of the material against the crack propagation [1-4]. This methodology gives a measurement of crack growth rate vs. given strains and strain rates and helps to predict the lifetime of rubber in a wide field of load levels. For seal applications in foreground is an exposure to load conditions which are characterised by lower and stable levels of deformations. Therefore, the knowledge of the energy level inducing cracks in rubber is of a greater importance for seals. This endurance limit is a material constant unique for each rubber compound and called the intrinsic strength. [5-7] It equals to the lowest tearing energy in a fatigue crack growth characteristic well known as Paris-Erdogan plot up to which no crack develops. [6, 8]
Thus, it is a mandatory challenge to find a methodology which estimates what the energy level is under which definitely no crack arises.

If a look is taken in nano-scale on an arising crack in rubber material, the polymer chain will rupture in direction of the main strain respective highest stress. A schematic visualization of a loaded rubber seal is shown in Fig. 1, right side. Furthermore, the process of rupture of rubber chains is shown in Fig. 1 left side schematically. From Fig. 1 it is very clear that due to applied load the chains rectangular to the load direction are strained. At an energy above the intrinsic strength the polymer chain orthogonal to the main strain respective stress direction will crack. The rupture of chain will proceed as long as the stress is higher as the intrinsic strength. [5-7] Thus, all the chains behind the crack tip are ruptured and in front of the crack tip they are strained. The stress decreases with increasing distance from the crack tip. The sketch in Fig. 1 is very simplified, whereas in reality the material is amorphous, the polymer chains are chaotically oriented and entangled as well. The cracking process is much more complicated only the principle is shown in the drawing.

However, the intrinsic strength defines the threshold of energy density to rupture a polymer chain. It is a material constant and independent from any load scenario. A rubber product operated at any load form with an energy level below its specific intrinsic strength can be assumed to be operable indefinitely without cracking the polymer chains or destroying the polymer network respectively [6, 7, 9].

Consequently, suitable rubber material can be developed to fulfil the specific requirement of a seal by knowing about the influence and the right choice of rubber ingredients. It can be concluded, the higher the intrinsic strength of a material the higher resistance against frac-

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ture processes. Fig. 1, right side illustrates in a schematic sketch, where the stress peaks will appear according to the given load and where the fatigue process will be initiated most likely. The rubber compound showing a high parameter of intrinsic strength would have enhanced resistance against crack initiation and thus service lifetime.

The aim of this work was to compare the archived ISA results with practical experiences of a seal producer at one hand to improve the methodology. On the other hand, to investigate the influence of rubber compound formulations and different curing systems typical for rubber compounds used for seal application on the intrinsic strength of rubber compounds. Finally, showing a possibility to improve the quality and service lifetime of seal application at real-life conditions. The methodology itself and the determined results will support the design of new rubber compounds for seal applications with focus on prediction of its durability. The work utilizes a new, commercially available instrument called Instrumented Intrinsic Strength Analyzer (ISA, Coesfeld GmbH & Co. KG, Dortmund, Germany), whereas the test methodology is based on the theoretical background of the cutting method published by Lake and Yeoh [5].

Experimental Details

Two different types of ethylene-propylene-diene-monomer rubber (EPDM) were used in this study. These polymers were filled with respectively 55 phr and 60 phr of N 550 type carbon black (CB). The compounds reinforced with 60 phr of carbon black additionally contain processing oil and plasticizers as well. Finally, two varied peroxide curing systems were applied. All chemical components have been sourced by. Dätwyler Schweiz AG, Switzerland. Table 1 lists the complete formulations of the rubber compounds, whereas the detailed description of the chemical components cannot be disclosed due to confidentiality. The experiences on the durability of the investigated compounds are given by Dätwyler Schweiz AG after the investigation as well.

The Rubber compounds were prepared using a two-stage mixing procedure described in Table 2, where both steps were performed in an internal mixer (SYD-2L, Everplast, Taiwan). The final batches were stored for 24 hours before curing.

1

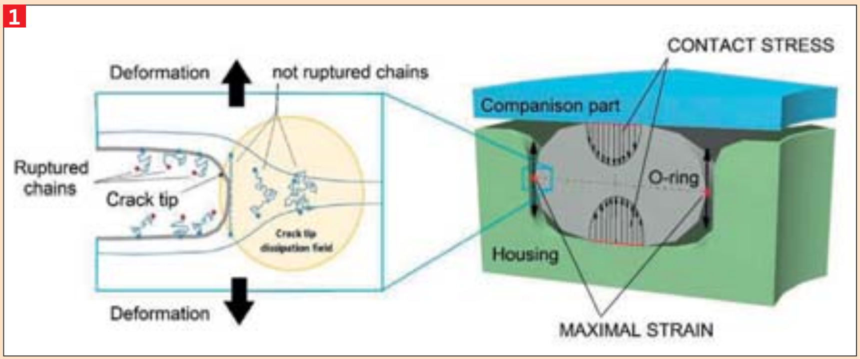

Fig. 1. Visualization showing the typical deformation of a crack tip and association of ruptured and non-ruptured chains at the vicinity of the crack tip for a seal application.

2

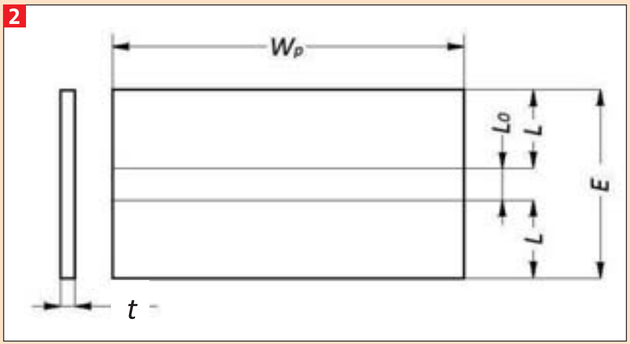

Fig.2. Simple plane pure-shear sample for ISA analyses.

1 Rubber Formulations in phr

Ranking	1	4	3	2
Chemical component	A6	B6	A5	B5
EPDM A	100.00	----	100.00	----
EPDM B	----	100.00	----	100.00
Carbon black N 550	60.00		55.00	
Processing oil	5.00		----	
Plasticizers	3.00		0.50	
ZnO	3.00			
Antioxidant 1	0.70			
Antioxidant 2	0.70			
Stearic acid	0.75			
Co-agent 1	0.32		----	
Co-agent 2	----		1.00	
Peroxide 1	----		4.00	5.00
Peroxide 2	3.00	3.60	----	
Sum	176.47	177.07	165.65	166.65

The curing characteristics were determined using a moving die rheometer (MDR 3000 Basic, MonTech, Germany) according to ASTM D 6204. The curing parameter t_{90} for all compounds analyzed is listed in Table 3.

Standard 150 mm x 150 mm sheets with 1.5 mm thickness were cured at t_{90} in a compression mold using a hydraulic press (LaBEcon 300, Fontijne Presses, Netherlands) heated to 175 °C. From the prepared sheets, samples with pure-shear dimensions schematically shown in the Fig. 2 were cut. The total length of test specimens is $E = 50$ mm, where $L = 20$ mm are used each side to fix the sample in the clamping system. Thus, the length $L_0 = 10$ mm defines the effective strained area of the sample. The width of sample was $W_p = 100$ mm and the thickness, $t = 1.5$ mm.

Rubber samples were tested at 25°C with ISA^{TM} operated with testing methodology developed by Endurica LLC, USA,

2 Compounding process

First stage					
Parameter	Time [s]	Encore	Ventilationtime [s]	RPM [r/min]	Temperature [°C]
Start	0	Rubber + chemicals	-	30	-
1.ventilation	60	-	10	25	-
2.ventilation	120	-	10	20	-
Mixing	240	-	-	20	max. 130
Tempering [°c]	T_{Body} [°C]: 50				
Second stage					
Start	0	Master batch + peroxides	-	15	-
1.ventilation	60	-	10	15	-
2.ventilation	120	-	10	15	-
Mixing	190	-	-	15	max. 110
Tempering [°c]	T_{Body} [°C]: 50				

3 Curing behavior

Compound	t_{90} [min]
A5	5.42
A6	8.21
B5	5.18
B6	8.38

which is based on the approach of Lake and Yeoh. [5] The photograph of ISA™ is shown in the Fig. 3 and the measurement principle is in detail explored in Fig. 4.

The samples firstly were preconditioned with a cut on one side with positioning its tip in the pure-shear region of the sample. [10] In the next step the cutting edge of a sharp blade is brought into contact with the crack tip without force. During the test the sample is exposed to a range of deformations from 0 to 50 %. It is stepwise deformed and kept in each position to equilibrate for 5 minutes. After equilibration at each step the blade is forced to cut the material continuously in three constant rates (10 mm/min, 0.1 mm/min, and 0.01 mm/min). While cutting after each step and equilibration, the energy density, *w* is noted as well as the cutting force f at each cutting rate. To maintain a constant rate of cutting the cutting speed has to be controlled.

3

Fig. 3. Photograph of Intrinsic Strength Analyzer.

4

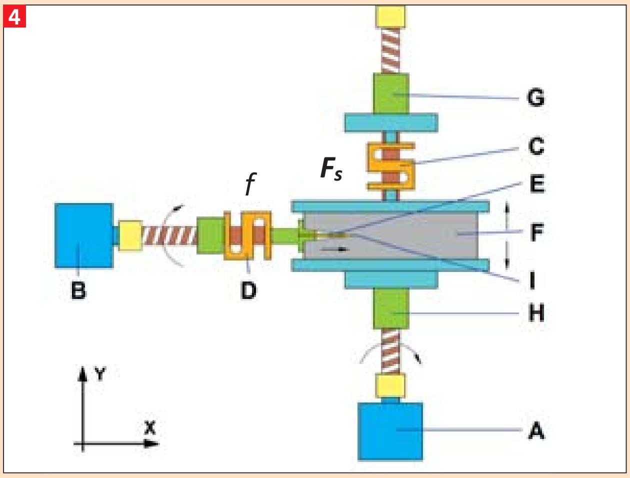

Fig. 4. Measurement principle, where: A – actuator of the axis Y; B – actuator of the axis X; C – loading cell of the axis X; D – loading cell of the axis Y; E – razor blade; F – test specimen; G – upper clamping system of test specimen; H – bottom clamping system of test specimen; I – razor blade tip.

As the sample has pure-shear dimensions, the tearing energy, *T* of the sample is calculated as the strain energy density, *w* multiplied by the length of non-deformed sample length, L_0.

$$T = w \cdot L_0 \tag{1}$$

The cutting blade plays an important role. The sharpness and the cutting edge have to be constant all the time of cutting to maintain a sufficient repeatability. Therefore, special blades produced by Lutz Blades, Germany were used. As the cutting energy has an additional contribution to the total energy release rate in driving the crack tip. The cutting energy, *F* results in division of cutting force, *f* by sample thickness, *t*:

$$F = \frac{f}{t} \tag{2}$$

The sum of the tearing energy, *T* and cutting energy, *F* for straining and cutting per step of strain and cutting rate is the required cutting energy for each strain/cut combination.
At the end an algorithm searches the minimum of energy. The resulting value is the minimal cutting energy labelled as intrinsic cutting energy, $S_{0,c}$ to cut the sample and is given as follow:

$$S_{0,c} = T + F \tag{3}$$

As the intrinsic cutting energy has a proportional correlation to the intrinsic strength, T_0 for a specific rubber compound it can be written as:

$$T_0 = b \cdot S_{0,c} \tag{4}$$

where the proportional factor, *b* is of a value between 0.12 and 0.18 observed in tests of different rubber types [5, 6]. The value of b depends on the rubber type, cutting friction between blade and rubber as well as blade´s tip sharpness.

4 **Intrinsic strength for all analyzed materials**

Compound	Intrinsic cutting energy $S_{0,c}$ [J/m²]	Proportionality constant *b* [-]	Intrinsic strength T_0 [J/m²]
A5	594.04	0,105	62.37
A6	682.46		71.66
B5	665.55		69.88
B6	497.20		52.21

An exemplification of the methodology to determine $S_{0,c}$ from the data measured using ISA™ is given in Fig. **5**, where the cutting energy, *F* is plotted against the tearing energy, *T* and the line with the slope -1 is corresponding to Eq. 3. This line is drawn as tangent to the lowest point of the data measured and consequently the intrinsic cutting energy, $S_{0,c}$ can be determined from the point, where the line intersects the X axis.

Results and Discussion

The plot of the cutting energy, *F* versus tearing energy, *T* is shown in Fig. **6**. The evaluation of the intrinsic cutting energies, T_0 is listed in Table 4. The lowest intrinsic cutting energy, $S_{0,c}$ = 497.20 J/m² was found for the compound B6, whereas the highest intrinsic cutting energy, $S_{0,c}$ = 682.46 J/m² exhibits the compound A6, both compounds reinforced with the 60 phr of CB . These two compounds differ in the varied type of elastomer as well as the compound B6 contain about 0.6 phr of the peroxide 2 more. Thus, it could be concluded that the type of elastomer has a significant influence on intrinsic cutting energy, whereas the higher amount of peroxide 2 could support the decreasing trend of intrinsic cutting energy for the elastomer of the type B. From the group of compounds reinforced with 55 phr of CB the lowest intrinsic cutting energy, $S_{0,c}$ = 594.04 J/m² was determined for the compound A5, whereas the higher intrinsic cutting energy, $S_{0,c}$ = 665.55 J/m² exhibits the compound B5. In the case of this group the rubber type has marginal influence, whereas the missing content of the processing oil, lower amount of plasticizers as well as the applied type of co-agent for peroxide curing and peroxide make the both materials more uniform compared to each other.

It is evident that the instrument ISA™ as well as the methodology deliver a clear ranking under given rubber materials with reference to its lifetime by determining the intrinsic cutting energy, $S_{0,c}$. Thus, the methodology based even on evaluating the intrinsic cutting energy, $S_{0,c}$ is an effective and reliable indicator for the expected lifetime of rubber seal.

Moreover, intrinsic strength, T_0 is a linear proportional to intrinsic cutting energy, $S_{0,c}$ but the proportional factor, *b* is only known as to be roughly between 0.1 and 0.2. The knowledge of the exact value of T_0 is more an academically question. It defines the tearing energy, *T* under which no crack initiation occurs. In other words, T_0 is the energy given in the Paris-Erdogan plot, where the graph intersects the x-axis. [9]

In the paper from Lake and Yeoh [5] 6 different rubber compounds were used, for which the values of the intrinsic strength were known. The proportional factor, *b* varied between 0.12 and 0.18. To estimate the intrinsic strength, T_0 with a good bounding above a factor *b* of 0.1 was used in our work. Using this constant, the intrinsic strength T_0 for the analyzed material was calculated and the estimated values of endurance limits are listed in Table **4**. Thus, the ranking of the materials from the highest to the lowest endurance limit is as follow: A6 > B5 > A5 > B6. From the results, it is visible that for seal application the compound A6 would be a material, which withstands the largest initiation energy before cracking.

Thus, a seal produced from material A6 would have the longest service lifetime and improved long-term durability amongst the seals produced with the other investigated compounds.

Conclusion

Experimental investigation of the influence of varied rubber formulations on the intrinsic strength of compounds used for seal applications has been performed. The complex methodology itself and the determined results have been used to support the development of new rubber compounds for seal applications in terms of prediction of their service lifetime and durability. Using this methodology, it was

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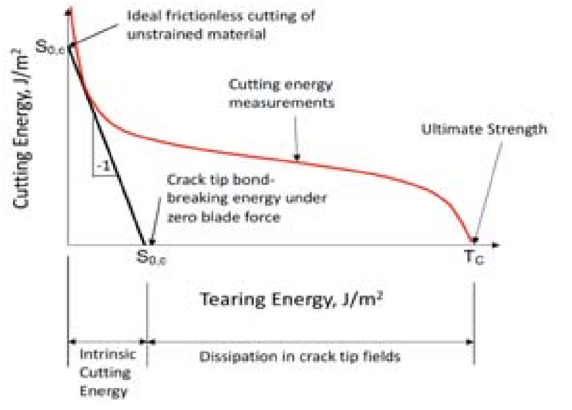

Fig. 5. Method used to determine $S_{0,c}$ from *F* versus *T* data.

6

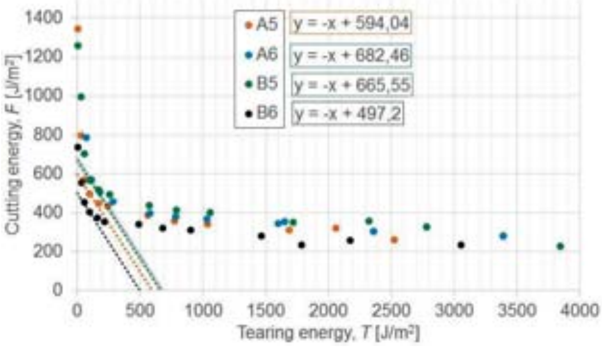

Fig. 6. Cutting energy, *F* versus tearing energy, *T* behaviors from which $S_{0,c}$ values are determined.

possible to estimate the fracture initiating energy of rubber compounds and select the right formulation in order to improve a compound's resistance to crack initiation and consequently enhance the service lifetime.

ISA™ is a simple tool to estimate efficiently and with low effort the endurance limits of rubber compounds. Within this study four commercial rubber compounds varying in formulations were studied. Based on the observed results it seems the polymer type has the most dominant effect on the level of the intrinsic strength compared to other ingredients varied in this study. This can be related to the molecular structure, molecular weight distribution, the content of ethylene and others. A study of the effect of various polymers on the intrinsic strength should be elaborated further in detail in a subsequent study.

This approach promises to optimize rubber based seal products in terms of durability. Furthermore, it leads to shorter development times, and as well to reduction of the time to market. It saves costs by minimizing resources, time and efforts on otherwise necessary extensive prototype testing.

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Dichtungslösung für eine neue Duroplastabdeckung im Projekt LeiMot

DICHTUNGEN Eine maßgefertigte Dichtungslösung von Dätwyler, entwickelt in Zusammenarbeit mit dem Aachener Engineering-Spezialisten FEV, ist nun Teil eines finalen Motorprototyps im Forschungsprojekt LeiMot (Leichtbaumotor). Ziel des Projektes ist es, das Gewicht der entscheidenden Komponenten - Zylinderkopf und Kurbelgehäuse - eines bestehenden Verbrennungsmotors der neuesten Generation (ICE) um bis zu 30% zu reduzieren. Durch das Ersetzen von Standardkomponenten und -teilen aus Metall durch Alternativen aus Faserverbundwerkstoffen und additiver Fertigung reduziert das Projekt nicht nur das Gesamtgewicht des Motors, sondern verbessert auch Betriebseffizienz und -verhalten sowie Wärmemanagement und reduziert Geräusch, Vibration und Rauigkeit (Noise, Vibration, and Harshness; NVH), um zukünftige Anforderungen an Komfort, Sicherheit und Nachhaltigkeit zu erfüllen. Dätwyler Produktmanager Rolf Figi kommentierte: „Wir waren begeistert vom Vorschlag unseres Engineering-Partners FEV, uns an diesem Projekt zu beteiligen. Die Anforderung bestand darin, eine Dichtungslösung für eine neue Kunststoffabdeckung zu entwickeln, und unser Co-Engineering-Ansatz hat sich hierfür als perfekt geeignet herausgestellt. Das LeiMot-Team verfügte über eine bereits vorhandene Simulation, die wir intern optimieren konnten, um eine Dichtungslösung mit bestmöglicher Flüssigkeits- und Wärmebeständigkeit zu empfehlen. Es ist das erste Mal, dass für diesen Zweck ein Kunststoffbauteil eingesetzt wurde.“

Nach Erhalt der ersten Simulationsdaten von der FEV führte der Dichtungshersteller eine eigene Simulation durch und machte Empfehlungen bezüglich der Geometrie des Bauteils, um die Dichtung zu verbessern. Dabei mussten viele Faktoren sorgfältig berücksichtigt werden, einschließlich der Tatsache, dass Kunststoffkomponenten eine höhere Vibrationswahrscheinlichkeit aufweisen, da sie nicht so fest fixiert werden können wie eine Metall-auf-Metall-Konstruktion. Die Dichtung musste deshalb auch in der Lage sein, alle potenziellen Vibrationen zu absorbieren, um übermäßigen Lärm und Haltbarkeitsprobleme zu vermeiden. Dätwyler nutzte seine Materialkenntnisse ebenfalls, um sicherzustellen, dass das gewählte Verbundmaterial in Bezug auf Temperatur- und Medienresistenz optimiert wurde.

Das Projekt tritt nun in die finale Prototyp-Phase ein, in der bis zu fünf Motoren komplett gefertigt werden. Das geringere Gewicht des Leichtbaumotors infolge der Verwendung von Verbundwerkstoffbauteilen wird sich direkt auf Kraftstoffverbrauch und thermische Effizienz auswirken. Zudem sorgen die optimierte Struktur und die Möglichkeit, Bauteile wie die Kunststoffabdeckung zu entkoppeln dafür, dass NVH-bedingte Probleme minimiert werden. Andreas Minatti, Head of Business Development bei Dätwyler, fügte hinzu: „Der Mobilitätssektor bewegt sich kontinuierlich in Richtung Elektrifizierung. Dabei darf aber nicht vergessen werden, dass der Verbrennungsmotor noch viele Jahre eine große, wenn nicht gar führende Rolle spielen wird. Genau diese Tatsache macht das LeiMot-Projekt so spannend und wichtig. “ ■

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