Technical

EPDM solution for automotive dense weatherstrip

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Executive summary

Because of its outstanding UV stability, thermal stability, hot air-aging, ozone and excellent polar fluid resistance, EPDM rubber is the material of choice for manufacturers of various automotive rubber parts, including weatherstrip. Dow's new Advanced Molecular Catalyst and polymerization process technology advancements enable the tailored design of unique EPDM molecular architectures, including composition and molecular weight distributions.

These unique microstructural features enable Nordel-brand EPDM end-users to develop formulations that meet both process requirements and finished product specifications in many automotive applications.

This report summarizes new Nordel EPDM product developments for dense weatherstrip applications; an efficient Design of Experiment-based formulation approach for developing compounds with balanced processability and physical properties; and Nordel EPDM technical solutions for automotive dense weatherstrip applications.

Ethylene propylene diene terpolymers (EPDM) is the third largest synthetic rubber and is widely used in applications such as transportation, infrastructure, industrial goods, and appliance.

Although many EPDM grades are still produced using conventional Ziegler–Natta catalysts, molecular catalysts for solution phase EPDM production have drawn increasing attention and have grown rapidly. The shift from Ziegler–Natta catalysts toward molecular catalysts is motivated by the desire for improved properties because of the enhanced control over polymer microstructure that molecular catalysts provide, as well as the ability to drive toward higher efficiencies and sustainable production.[1-4]

TECHNICAL NOTEBOOK
Edited by John Dick

Dow's Advanced Molecular Catalysts have much more efficient comonomer and termonomer incorporation compared to conventional Ziegler–Natta catalysts, thus allowing for the production of copolymers with any desired level of comonomer and termonomer.[5] For example, higher ENB (>8.5 percent) content is easily achieved with AMC for EPDM at both high and low ethylene content.

For the rubber end-user, Dow's AMC technology can efficiently produce EPDMs with ultra-high molecular weight, higher diene content and increased levels of long chain branching compared to conventional metallocene catalysts resulting in faster mixing and crosslinking.

The fully saturated molecular structure provides EPDM rubbers with outstanding weatherability, UV stability, and low/high temperature resistance. Therefore, EPDM rubber has been established as the primary polymer choice for automotive sealing applications. Automotive body sealing (both dense and sponge weatherstrip) represents one of the largest applications, accounting for about 20 percent of the total worldwide consumption of EPDM.

A typical manufacturing process of automotive dense weatherstrip includes the following steps:

- Compound formulating and mixing;
- Profile extrusion;
- Curing;
- Surface coating if necessary;
- Cutting and shaping;
- Connecting the final assembly; and
- Finishing, packing and shipping.

It is well known that the productivity of an automotive weatherstrip is predominately determined by the extrusion speed and cure speed of the EPDM compound. For a given die geometry and head temperature, and a given screw speed, EPDM molecular structure has significant influence on extrusion speed. Long chain branching and broader MWD improves the shear thinning of the EPDM compound, thus reducing the high-shear viscosity at extruder die. On top of high extrusion speed, the cure kinetics should match the high extrusion speed, which is determined by the diene type of content of the EPDM. In general, EPDM with high diene content for fast cure and high cure state is preferred.

Nordel-brand 6565 XFC EPDM based on AMC technology is a high diene, amorphous EPDM grade designed for extruded applications. It is ideally suited for solid and foamed extruded profiles requiring fast cure conditions. The combination of high molecular weight, low crystallinity, high ENB content and long-chain branched structure allows for a good balance of extrusion processability, extendibility, low-temperature compression set and load deflection performance.

The product features improved mixing, extrusion and cure characteristics, suitable for Class A surface manufacturing with improved shape stability of the green compound.

The basic material specifications of Nordel 6565 XFC are listed in **Table 1**.

Depending on the final application requirement, EPDM compound formulation must be optimized for compound cost, processability and other critical physical properties. A Design of Experiment approach for developing compound formulations with balanced processability and physical properties was adopted in this study.[6] Once the formulation or recipe is defined, key steps to determine the final performance of weatherstrip are compound mixing, extrusion and curing.[6-8]

The main objective of this paper is to demonstrate the formulation development and validation of Nordel 6565 XFC-based compounds for dense weatherstrip for three different hardnesses with a novel nitrosamine-free cure system.

The paper is divided into two parts:

- A Design of Experiment approach developed for predicting the properties of a dense weatherstrip compound, for example, hardness; and
- Industrial scale validation of N-nitrosamine-free solutions based on a model predicted formulation with Nordel 6565 XFC.

Materials and experiments

Table 2 shows the formulations used in the DOE study. In this study, the ethylene content was varied by blending N4470 with N6565; the content of Sunpar 2280 oil content was varied from 80 to 160 phr; the content of N550 carbon black was varied from 130 to 250 phr.

For large scale compounding and extrusion runs, the following three formulations predicted from the DOE model, shown in **Table 3**, were used for 60 Shore A, 70 Shore A and 80 Shore A hardness targets, respectively. A novel nitrosamine-free cure system was adopted in the proposed formulations.

The larger scale mixing and extrusion run has been carried out on an industrial production scale process including mixing, extrusion and curing.

A 135-liter net volume rubber internal mixer with intermeshing rotor design and hydraulic ram control was used in this evaluation. However, any of the

Table 1: Basic material specifications of Nordel 6565 XFC EPDM.

ASTM & ISO Properties[1]			
Property	**Nominal Value**	**Unit**	**Test Method**
Specific Gravity	0.862	g/cc	ASTM D297
Mooney Viscosity (ML 1+4 at 125°C)	65	MU	ASTM D1646
Ethylene Content	55.0	wt%	ASTM D3900
Ethylidene Norbornene (ENB) Content	8.5	wt%	ASTM D6047
Ash Content	< 0.1	wt%	ASTM D296
Molecular Weight Distribution	Broad		Dow Method
Propylene Content	36.5	wt%	ASTM D3900
Residual Transition Metal	< 10	ppm	Dow Method
Volatile Matter	< 0.40	wt%	Dow Method

[1]Typical properties: these are not to be construed as specifications

Table 2: Lab scale dense weatherstrip compound formulation for DOE.

Formulations	
EPDM	100
N-660 Carbon Black	130~250
Omya 2T-FL	60
Talc	20
Zinc Oxide	5
Stearic Acid	1
PEG 3350	2
Sunpar 2280	80~160
CDPA	1
MTI	2
BASE TOTAL	401~601
Desical P	6.00
Sulfur	1.00
DPTT-70	1.50
TMTD-75	0.90
ZDBC-75	0.20
MBT-80	0.30
MBTS-75	1.00
CURE TOTAL	**10.9**

Table 3: One-pass Nitrosamine free dense weatherstrip formulation.

	60 Shore A	70 Shore A	80 Shore A
Material	**phr**	**phr**	**phr**
NORDEL™ 6565 XFC	100	80	50
NORDEL™ 4770		20	50
N550	134	136	146
Calcium Carbonate	60	60	60
Talc	20	20	20
ZnO	6	6	6
Stearic Acid	1	1	1
PEG 4000	2	2	2
Sunpar 2280	105	90	86
AO405	1	1	1
AO 60	2	2	2
CaO Dispersion (95%)	5.00	5.00	5.00
Sulfur (80% dispersion)	1.25	1.25	1.25
MBT (MBT-80)	0.30	0.30	0.30
MBTS (MBTS-70)	1.00	1.00	1.00
ZAT (ZAT-70)	0.80	0.80	0.80
TBzTD (TBzTD-70)	1.10	1.10	1.10
ZnBEC (ZBEC-70)	0.30	0.30	0.30
ZnBPD (50%)	1.00	1.00	1.00
Grand Total	**441.8**	**428.8**	**434.8**

Table 4: One pass mixing procedures.

Step	Command	Logic	Time (s)	Temp (°F (°C))	Rotor RPM
1	Raise Ram	None			38
2	Charge Black/White/Oil	None			38
3	Auto Charge EPDM	None	20		38
4	Close Hopper Door	None			38
5	Lower Ram	TEMP	0	176 (80)	38
6	Raise Ram	None	10		30
7	Auto Charge Curatives	None	20		18
8	Close Hopper Door	None			18
9	Lower Ram	TEMP		203 (95)	18
10	Raise Ram	TIME	10		18
11	Lower Ram	TEMP		221 (105)	18
12	Discharge Warning	TIME	3		18
13	Open Drop Door	TIME	45		20
14	Close Drop Door	None			20

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The authors

Sharon Wu is a research scientist at Dow Chemical Co. She currently is responsible for driving the Nordel-brand EPDM product and technology innovation pipeline for various thermoset applications.

Wu joined Dow Elastomers product research group in 2008 after she received her doctorate in polymer materials and engineering from Georgia Institute of Technology. Her areas of focus are structure-property relationships of polyolefin elastomer products, and new processes and methods to produce unique elastomer products with differentiated properties.

Wu

She is the author or co-author of 15 refereed journal publications, 26 patent applications (15 granted), and 30 conference proceedings.

Greg Li is a research scientist in North America Packaging & Specialty Plastics Technical Service & Development at Dow. His responsibilities include the development of Nordel EPDM products, formulations, and application and processing developments.

Li joined Dow Elastomer R&D in 2011. Prior to joining Dow, he worked at the Technical Foams Division of Armacell L.L.C. for three years as a research scientist and plant process chemist, where he developed elastomeric closed cell and open cell foams for the automotive industry.

Li

He received a bachelor's in engineering from Tianjin University, a master's from the Institute of Chemistry (Beijing), Chinese Academy of Sciences, and a doctorate from the University of Toronto. Li performed post-doctoral research at Case Western Reserve University.

Santosh Bawiskar is a principal scientist at Dow. He has more than 20 years of experience in material development, compounding and processing of elastomers. He graduated with a doctorate in polymer engineering from the University of Akron and an MBA from Clarion University.

Bawiskar

Bawiskar serves on the board of the TPE Special Interest Group of the Society of Plastics Engineers.

Rennisha Wickham received a bachelor's degree in chemistry from Virginia State University and a doctorate in chemistry from the University of Maryland. Her research background is in the synthesis of end-group functionalized low molecular weight precision polyolefins and the development of new analytical tools for their characterization.

Wickham

Wickham joined Dow in 2012 and has held both product development and application development roles within the R&D organization. In 2017, she transitioned to a customer manager role within the Dow Packaging & Specialty Plastics' commercial organization, where she leverages her R&D and sales background to strengthen Dow's innovation portfolio and secure long-term relationships.

Wenzhao Yang is a statistician in the North America Core R&D unit at Dow. Yang's key responsibilities include supporting, enhancing and promoting sound experimental design strategies and statistical data analysis techniques within Dow.

Yang

She joined Dow Core R&D in 2014. Yang has been involved in causal inference and optimization for formulation and product development. She holds a master's degree in statistics and a doctorate in applied statistics and agriculture from Michigan State University.

Lena Nguyen is the global product technology leader for Nordel EPDM at Dow. She has been with the company for more than 20 years and has worked in various businesses, including Dow Building Solutions, Dow Automotive Systems and Dow Elastomers.

Nguyen

Her work involves development and commercialization of Nordel EPDM products to serve the rubber industry in various applications.

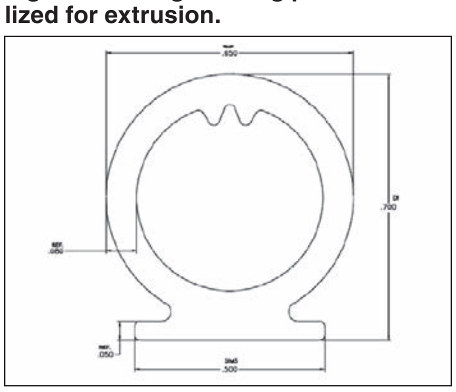
rubber mixing equipment should work with the appropriate mixing procedures and mixing conditions, including:

- Rubber internal mixer: Intermeshing rotor design;
- Rubber internal mixer: Tangential rotor design;
- Rubber kneader; and
- Two roll mill

The following mixer settings were utilized for one-pass mixing of the proposed formulations:

- Fill factor = 0.74 for 60 shore A compound formulation;
- Fill factor = 0.72 for 70 shore A compound formulation; and
- Fill factor = 0.70 for 80 shore A compound formulation.

Fig. 1: An omega testing profile die utilized for extrusion.



The detailed mixing procedures/steps are illustrated in **Table 3**.

Mooney viscosities of all the mixed compounds were measured using an Alpha Technologies Mooney Viscometer following ASTM D1646. Compound cure kinetic profiles at 180°C were measured using an Alpha Technologies MDR 2000 moving die rheometer following ASTM D5289-15.

All three dense profile compounds were extruded on a 3.5-inch rubber cold feed extruder with a 10:1 L/D ratio to form the desired profile shape as shown in **Fig. 1**.

The extruded profile was continuously cured on a continuous vulcanization line that was a combination of multiple hot air ovens and microwave ovens. High quality dense profiles with smooth skin were successfully produced via the above mentioned process.

In this particular study, the CV line consisted of a total of three 20-foot ovens. The first and third ovens are hot air ovens with adjustable speed, temperature and air velocity. The second oven is a 20-foot microwave oven with three adjustable power outputs (maximum power 6kW, 2kW for each zone) as well as adjustable speed, temperature and air velocity.

Table 5: Extrusion and curing conditions.

	Unit	60 Shore A Formulation	70 Shore A Formulation	80 Shore A Formulation
Extruder				
Screw Temperature	F (°C)	150 (66)	150 (66)	150 (66)
Barrel# 1 Temp	F (°C)	175 (80)	175 (80)	175 (80)
Barrel# 2 Temp	F (°C)	175 (80)	175 (80)	175 (80)
Barrel# 3 Temp	F (°C)	175 (80)	175 (80)	175 (80)
Speed	RPM	17.9	17.9	17.9
Hot Air Oven # 1				
Oven length	ft	20	20	20
Temperature	F (°C)	450 (232)	450 (232)	450 (232)
Speed	fpm	17.0	17.0	17.0
Microwave Oven				
Oven length	ft	20	20	20
Temperature	F (°C)	450 (232)	450 (232)	450 (232)
Microwave	Kw	0.8	0.8	0.8
Speed	fpm	18.9	18.9	18.9
Hot Air Oven # 2				
Oven length	ft	20	20	20
Temperature	F (°C)	450 (232)	450 (232)	450 (232)
Speed	fpm	19.3	19.3	19.3

Results and discussion

Design of Experiment

One goal for this study was to identify optimal EPDM formulations for multiple key properties through DOE planning and statistical modeling. Based on previous research, three interested input factors were considered in the DOE plan: EPDM ethylene content (55 phr to 70 phr), Sunpar 2280 content (80 phr to 160 phr) and N660 carbon black content (130 phr - 250phr).

An optimal design was generated using JMP-brand Pro 14.2.0 with 18 formulations including three center points and three replicates. Replicates were added to ensure good reproducibility for the key metrics. The design chosen was comparable to a response surface design in a number of experiments and capability of estimating main and second order polynomial effects.

For example, a Box-Behnken design requires 15 runs but it adds up to 18 runs with three replicates. Optimal design is usually chosen when there are constraints on factors or on randomization. Based on prior knowledge, no significant quadratic effect of EPDM ethylene content for the key metrics were expected. Hence, the proposed model in the optimal design excluded quadratic effects on EPDM ethylene content.

All properties had a good model fit for the proposed model as most models were significant with R2 > 0.9 shown in **Table 6**. For hardness, 99 percent variation can be explained by the main and second order polynomial effects of the input factors (except quadratic effect of EPDM ethylene content).

For targeted 60 Shore A, 70 Shore A and 80 Shore A EPDM compound for dense profile, the JMP model predicted the blend ratio of N6565 and N4770, the oil content and the CB content as shown in **Table 3**.

Mixing

Mixing curves for all the different hardness compounds are included in the graph shown in **Fig. 2**. Good temperature control and fast mixing cycles (<3 minutes) can be achieved with proposed formulations and mixing procedures. Good mixing power (250 kW) also was achieved, enabling good filler dispersion.

As the compound hardness increases, the filler and oil incorporation rate slows down. The ram did not reach the fully seat position until the later stage of the mixing process. Therefore, special care was needed to optimize the mixing process for 80 Shore A compound with higher amount of filler and less oil.

Compound properties

The compound Mooney viscosity and

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Table 6: Predicted model evaluation results.

	Hardness	*Tensile*	*Elongation*	*Tear Strength*	*Compound ML1+4 (100°C) for processability*	*Compression Set 22 hrs @70°C*
Significance (Yes or No)	Yes	Yes	Yes	Yes	Yes	Yes
P (<0.05)	<.0001	0.0004	<.0001	0.001	<.0001	0.0005
RSq (>0.80)	0.99	0.92	0.97	0.90	0.95	0.92

Maplan benefiting from lean production process

By Patrick Raleigh
European Rubber Journal

DUESSELDORF, Germany—A lean manufacturing set-up is proving its worth for Maplan in adapting to the current business climate, according to CEO Wolfgang Meyer.

For 2019, the Austrian injection equipment maker expects revenues to drop back to 2017 levels thanks to the global economic slowdown, officials said at the recent K 2019 show in Duesseldorf. The decline follows the Austrian company's best ever business year in 2018, when it delivered consolidated sales of about $55.7 million and built around 350 machines.

Maplan reported sales as nearing $50 million for 2019, and since has set a goal of growing sales to about $80 million by 2021: $33 million in Europe, $22 million in America, $11 million in Asia and the remainder coming from aftersales.

Since February, however, there has been a marked drop-off in orders, largely reflecting a decline in automotive vehicle production in China and some signs of slowdown in Europe and the U.S., Meyer said.

About half of Maplan's business is in the automotive industry—its offerings for the sector largely include horizontal machines for sealing products and vertical machines for anti-vibration parts, Meyer said.

Among its other markets, the company supplies molders serving the construction, infrastructure and consumer goods industries, he said, adding that Maplan is adapting to current market conditions.

In 2016, Maplan relocated to a specially designed headquarters facility in Kottingbrunn, Austria, near Vienna, that uses an automated flow assembly production system to reduce throughput times by about 30 percent.

According to Meyer, a rubber injection molding machine typically requires 600-800 parts, and conventionally, up to 30 percent of the build time can be used in searching for parts.

At Kottingbrunn, a model of every part in stock is fed into Maplan's ERP system, which automatically orders the parts for the different assembly steps. Suppliers are notified by RFID when parts need to be restocked.

"We originally aimed to have 70 percent of our machines (built this way). At the moment, 90 percent of our machines are running over the line," Meyer said.

The operation further is supported by a component factory that was opened in 2018. It is located about 80 miles away in Malacky, Slovakia. Maplan produces electric and hydraulic assemblies as well as mid-size components and sheet-metal parts there.

The flexibility of the overall production set-up gives Maplan "greater efficiency, room for maneuverer, and cushions it from market declines," according to the Meyer.

Capacity freed up in the main plant is being used for product development, including the addition of "360-degree turnkey systems, ergonomic features and automation system upgrades," he said.

The program has, for example, included a complete overhaul of the company's vertical axis machine series, which has also been expanded with additional size levels.

"There is really a lot of novel features within the range, which now goes from 90 (metric tons) up to 900 tons, all with 20 percent steps in increase in clamping pressure," Meyer said.

The next big step in the company's strategy is to deliver "complete 360-degree solutions," Meyer said.

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curing speeds are listed in **Table 5**. Nordel 6565 XFC EPDM grade was designed to have a faster cure rate in order to enable fast extrusion and fast curing speed. The compound target cure speed, for example ts2 at 180°C, was approximately 0.7 minutes. Because of the higher filler loading and less oil extension, the higher hardness compound has higher compound Mooney viscosity than that of the softer compound.

The physical properties of cured compounds are shown in **Table 6**. All three compounds met the hardness target with high tensile strength and low compression set properties.

In summary, in order to control the compound hardness, several formulation adjustments were adopted to meet the target properties. The formulation adjustments are blending of semi-crystalline EPDM grade, adjusting filler loading, and adjusting oil content in the formulation.

The 80 Shore A compound contains 50 phr of semi-crystalline EPDM grade (Nordel 4770), higher carbon black loading level and less oil content. As the compound hardness increases, the compound tensile strength, the compound compression set and TR 10 increase as well.

The extruded and cured profile parts were tested for compression load deflection (CLD) and compression set. The results are summarized in **Table 7**. All the extruded parts with different hardness exhibited good surface quality and low compression set.

Conclusions

The combined impact of Dow's AMC and solution polymerization process upgrade enables product design upgrades to EPDM that feature broader MWD, increased LCB level and finer molecular architectural control. Nordel 6565 XFC has significantly improved melt strength and fast cure speed compared to Nordel 5565 because of a broader MWD, increased LCB level and ENB level.

This work summarizes the formulation development and industrial scale validation of Nordel 6565 XFC-based solution for dense weatherstrip for three different hardness with a novel nitrosamine-free cure system.

Using a DOE approach, EPDM formulations for dense weatherstrip compounds with different shore A targets were developed, followed by an industrial scale validation of N-nitrosamine-free solutions based on model predicted formulations with Nordel 6565 XFC.

It is concluded that the use of a DOE is a powerful and effective tool to develop EPDM compound formulations to predict the processing and application requirements. Nordel 6565 XFC was utilized to successfully produce dense weatherstrip profiles with various compound hardness targets via a one-pass mixing process to enable flexibility in processing.

Nordel 6565 XFC-based nitrosamine-free solutions developed for 60-80 Shore A automotive dense weatherstrip applications show excellent heat aging, compression set and ozone resistance that exceed the OEM specifications.

Fig. 2: One-pass mixing curves.

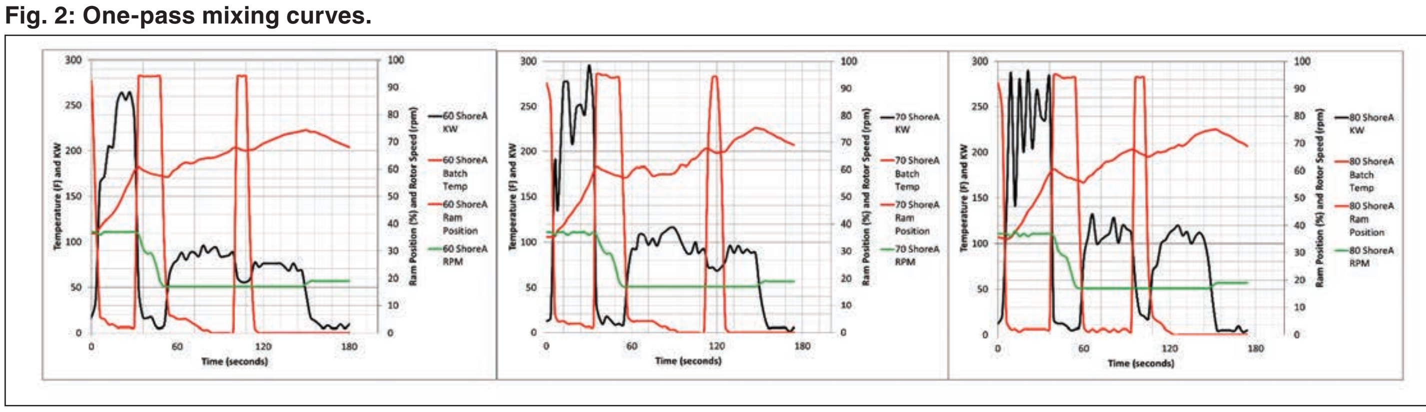

Table 7: Compound Mooney and MDR results.

Test Conditions	Properties	Units	60 Shore A Formulation	70 Shore A Formulation	80 Shore A Formulation
ML 1+4@ 100°C		MU	50.7	68.2	83.7
MDR @ 180°C For 30 min	ts1	min	0.7	0.6	0.6
	ts2	min	0.8	0.7	0.7
	t30	min	1.1	1.1	1.1
	t70	min	11.9	10.6	10.8
	t90	min	19.1	19.0	18.3
	ML	lb-in	1.5	2.0	2.4
	MH	lb-in	8.7	10.1	11.2

Table 8: Compound cured physical properties.

Test Conditions	Properties	Units	60 Shore A Formulation	70 Shore A Formulation	80 Shore A Formulation
Original Properties	Hardness	Sh.A	66	73	78
	Tensile strength	MPa	9.2	9.5	11.0
	Elongation at break	%	601	554	458
	100% Modulus	MPa	2.2	2.9	3.5
	300% Modulus	MPa	5.2	6.3	8.2
	Tear strength	N/mm	30	32	37
Aged Properties (70 hrs at 70°C)	Hardness	Sh.A	67	75	79
	Tensile strength	MPa	8.9	9.8	9.5
	Elongation at break	%	578	513	492
Compression Set (22h@70C)		%	14.6	16	20
TR-10		°C	-39	-34	-30
Ozone Resistance			No Crack	No Crack	No Crack

Table 9: Extruded dense profile physical properties.

Properties[1]	Units	60 Shore A Formulation	70 Shore A Formulation	80 Shore A Formulation
CLD(1)	lbf	4.9	8.1	13
	(kgf)	(2.2)	(3.7)	(6)
CLD(2)	lbf	9.4	15	24
	(kgf)	(4.3)	(6.8)	(10.9)
Compression set(3)	%	12	13	12
Compression set(4)	%	41	36	39

Notes:

(1) Compression Load Deflection (CLD) (1): Compress 25% on a 100 mm profile. The profile was pre-flexed three times at 40% deflection

(2) Compression Load Deflection (CLD) (2): Compress 40% on a 100 mm profile. The profile was pre-flexed three times at 40% deflection

(3) Compression Set (3): Compress 50% deflection for 22 hrs at room temperature on 100 mm piece and recovery for 24 hours.

(4) Compression Set (4): Compress 40% deflection for 22 hrs at 70C on 100 mm piece and recovery for 2 hours.

Acknowledgments

We would like to thank Kristel Blackwell for the compounding support at the Dow Elastomers Thermoset Rubber Lab in Lake Jackson, Texas.

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