

Technical

Peroxide-cured HFP boosts cure rate, demolding

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Executive summary

Fluoroelastomers having a monomer composition of tetrafluoroethylene (TFE), hexafluoropropylene (HFP) and vinylidene fluoride (VF2) with an incorporated cure site monomer that allows peroxide crosslinking have grown in commercial significance in the past 20 years since the introduction of Viton GF in 1980. While these FKM terpolymers show improved water, steam and acid resistance, the need to peroxide crosslink them has caused processing problems such as mold sticking and poor hot tear resistance.

This paper will introduce a new generation of peroxide-cured HFP-containing fluoroelastomers, which use a new, unique improved cure site monomer system. These ICSM containing products give much faster cure rates, low mold fouling and improved hot-demolding properties. In addition, they provide improved compression set resistance, even when not postcured, and enhanced aqueous media resistance. Processing and end-use data will highlight the utility of these new type fluoroelastomers.

TECHNICAL NOTEBOOK

Edited by Harold Herzlich

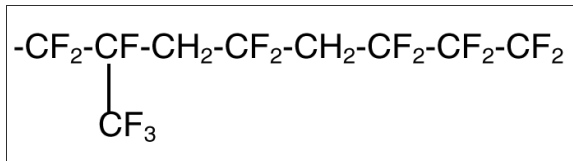
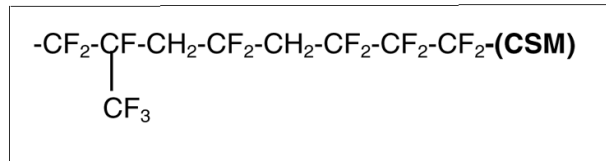
Fluoroelastomer terpolymers having a structure of hexafluoropropylene (HFP), tetrafluoroethylene (TFE) and vinylidene fluoride (VF2) have been used since the 1960s in a variety of industrial and automotive applications. The general chemical structure of these polymers is shown in **Fig. 1**.

In the mid-1970s a breakthrough in cure systems became available to the fluoroelastomer chemist, an FKM terpolymer with incorporated cure site monomer (CSM) that was sensitive to a peroxide/coagent cure system (**Fig. 2**).

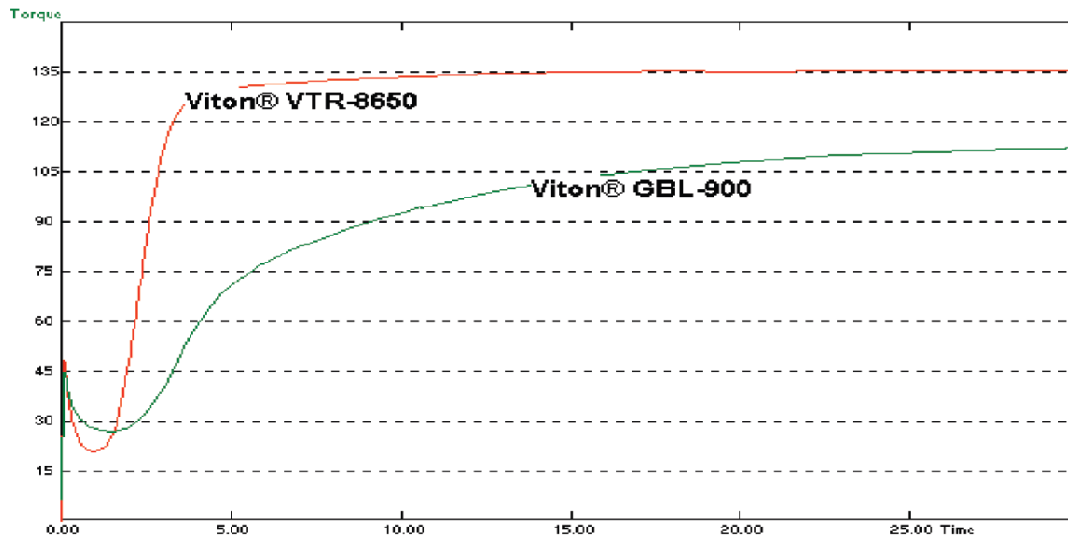
These peroxide-cured types of FKM were introduced at the San Francisco Rubber Division/ACS meeting by Finlay, MacLachlan and Hallenbeck^{1,2} in 1976, and in 1980, Bauerle and Finlay³ introduced an improved ~70-percent fluorine version of this type of FKM called Viton GF. Later, 68-percent fluorine versions of these elastomers called GBL900 and GBL200 were introduced by Tabb and Stevens⁴ at the Los Angeles Rubber Division/ACS meeting in 1985.

These new peroxide-cured types of fluoroelastomer had several advantages in end-use properties, specifically in the area of fluids resistance. The peroxidecured polymers do not depend on strong metal bases such as magnesium oxide to dehydrohalogenate the backbone at the VF2-HPF site to initiate a double bond for the crosslinking reaction, rather they crosslink through a cure site monomer by use of a coagent that is activated by a peroxide. These peroxide-cured FKMs are known to have improved water, steam, acid and amine additive resistance when compared to conventional bisphenol-cured types of fluoroelastomers.

While the end-use properties of peroxide-cured FKM are quite impressive, the processability of compounds suffers from the use of this cure system. Mold sticking and reduced hot tear strength are two problems that can be observed when processing peroxide-cured FKM. These problems led to more production scrap and higher part costs for peroxidecured FKMs.

Fig. 1. FKM terpolymer.**Fig. 2. Peroxide curable FKM terpolymer.****Fig. 3.**

New "APA" Technology Polymers	Fluorine Content	Gum Mooney (ML @ 121°C)	Comparable Commercial Peroxide Cured FKM
VTR-8600 (GF-600S)	70.2%	65	Viton® GF, GF300
VTR-8650 (GBL-600S)	68.5%	65	Viton® GBL200, 900

Fig. 4. ODR @ 162°C, 3° arc, 100 range, 30-minute clock.

Advanced Polymer Architecture technology

Knowing both the advantages and problems with peroxide-cured FKM, the research and development group at DuPont Dow Elastomers L.L.C. has undertaken an extensive development program to reshape our peroxide-cured FKM product line. The result of this program is a new generation of peroxidecured FKM that utilizes a new, improved cure site monomer (ICSM), and is polymerized in a way that permits a high degree of control of molecular weight distribution and end group chemistry. These new Advanced Polymer Architecture peroxide types of fluoroelastomer show dramatically improved processing as evidenced by a faster cure rate, much improved mold release and better hot properties. In addition to processing advantages, the Advanced Polymer Architecture products provide significant end-use property advantages such as low compression set, with or without a postcure, lower water swell and improved acid resistance.

The purpose of this paper is to introduce these new HFP-containing terpolymers made with DuPont Dow's Advanced Polymer Architecture technology. The polymers are described in **Fig. 3**. Both of the polymers are a nominal 60-65 Mooney (ML 1+10 @ 121°C) and originally were developed for compression molding. Data on processing and end-use properties will be presented to illustrate their performance.

Supply

At this point in time, VTR-8600 and VTR-8650 are being manufactured commercially at a new DuPont Dow facility in Dordrecht, Netherlands. This factory started manufacturing in the third quarter of 2002. Once these products are established in the marketplace, the VTR designation will be upgraded to commercial names Viton GF-600S (for VTR-8600) and Viton GBL-600S (for VTR-8650).

Experimental

Several lab studies were run to compare the properties of VTR-8600 and VTR-8650 with commercial peroxidecured grades. Much of the initial testing was done on a standard 30 MT Black lab recipe cured with a TAIC coagent/45-percent active 2,5-dimethyl 2,5-bis (tbutyl peroxy) hexane peroxide cure system.

Compound and vulcanizate properties have been completely characterized using standard ASTM test methods. Tests performed include rheological tests, original physical properties, heat resistance, fluid resistance, compression set resistance and low temperature properties.

To complement the standard ASTM tests, additional evaluations were carried out using DuPont Dow test methods that will be described in the paper. These evaluations included mold release testing, capillary rheometry, permeation and stress relaxation testing. Compounds were mixed both in a BR lab Banbury and on a two-roll mill.

Fig. 5.

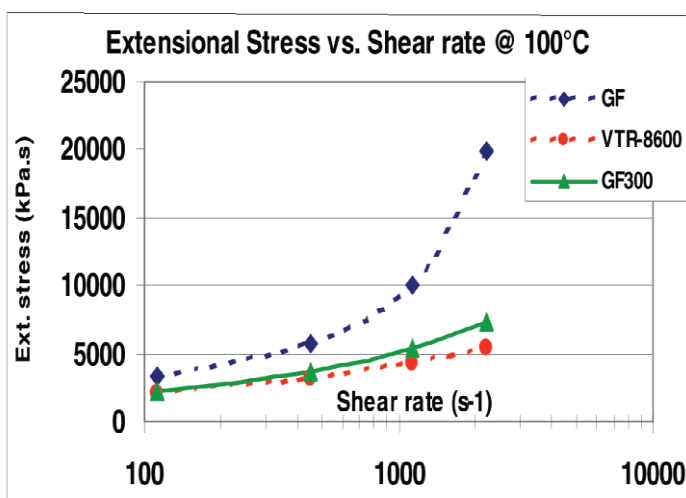
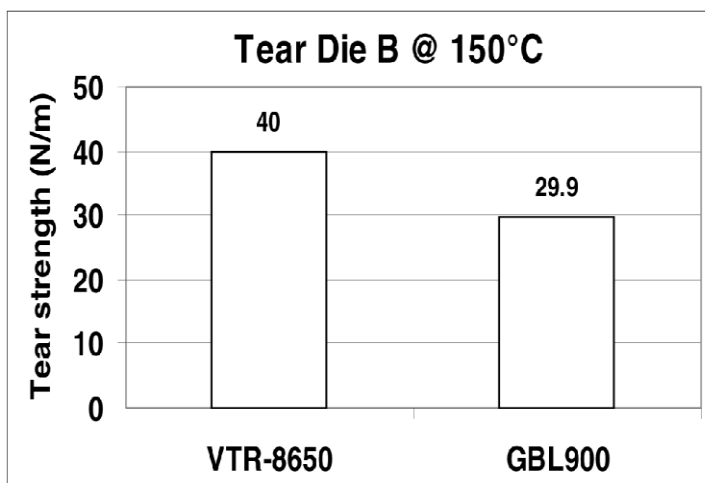


Fig. 6.



Results and discussion

Processing evaluations

The initial evaluations were done using the standard test recipe mixed in a BR internal mixer. No significant mixing differences were seen between the control compounds and the developmental VTR-8600 and VTR-8650. In the case of the higher fluorine types like GF and VTR-8600, the use of a slightly higher load factor (70 percent) seemed to help the batch come together faster.

Rheology

Rheology measurements such as gum polymer Mooney, Mooney Scorch, ODR and MDR can be reviewed in **Table I**. The data in **Table I** show that VTR-8600 gum tested out at 60 Mooney (ML 1+10 @ 121°C) and the VTR-8650 tested at 64 Mooney. Mixed compounds based on VTR-8600 and VTR-8650 showed a little shorter 2-point rise time and lower viscosity than the controls. A dramatic difference in cure rate was seen between the Advanced Polymer Architecture types and the control types as is demonstrated in **Fig. 4**.

The trends of faster cure speed and higher state of cure seen on the ODR were repeated on a moving die rheometer, the MDR2000, when run at 177°C. It was felt that the faster cure rate seen with VTR-8600 and VTR-8650 was a very desirable attribute for processability. One of the reasons peroxide-cured types of fluoroelastomer stick in the mold is the fact that the coagents, commonly TAIC, can homopolymerize in the presence of peroxide. If the fluoroelastomer cures too slowly, the TAIC has time to migrate out of the polymer, homopolymerize and stick to the mold surface. With the cure speed demonstrated by the Advanced Polymer Architecture types, the likelihood of keeping the TAIC in the vulcanized compound matrix, and not bleeding out, is much higher, reducing the possibility of mold sticking.

Another unique attribute of the Advanced Polymer Architecture types is the way they flow and shear-thin in capillary rheometer testing even though they are 60 Mooney compression molding grade polymers. Testing was done on a Rosand capillary rheometer at 100°C.

Fig. 5 shows the extensional stress vs. shear rate relationship for GF, GF-300 and Advanced Polymer Architecture VTR-8600. Viton GF is seen to develop much higher extensional stress than either GF-300 or VTR-8600, particularly at higher shear rates. Molding and extrusion processes invariably contain regions of high shear so **Fig. 5** strongly implies that VTR-8600 would flow much more easily than GF and similar to 30 Mooney GF-300.

“Hot” tensile properties for demolding

To demold parts successfully, especially parts with complex shapes, a rubber material needs adequate tensile strength and elongation at the hot demolding temperature. Lack of hot tear strength always has been a common complaint about peroxide-cured fluoroelastomers.

Testing was done to compare the hot properties of Advanced Polymer Architecture VTR-8600 and VTR-8650 to current commercial peroxide-cured FKM. Slabs were cured at 177°C and then they were tested with no postcure at high temperature to simulate the condition they would be in during demolding. A summary of this data can be seen in **Table I**.

In **Fig. 6**, 68-percent fluorine VTR-8650 and GBL900 are compared in a Tear Die B test run in an environmental chamber at 150°C. VTR-8650 demonstrated considerably more hot tear strength than the commercial GBL900, some 33 percent higher. This indicates that VTR-8650 will be much easier to demold without tearing than GBL900.

O-ring demolding tests

DuPont Dow has devised a test to simulate the molding performance of fluoroelastomers. This demolding test is the removal of small O-rings from a simulated production O-ring mold. The mold and test protocol used are described in a Rubber Division/ACS paper.⁵ Parts are demolded with a controlled, light stream of air only. The mold self-deflashes itself so the only thing removing the O-rings is air. The number of O-rings stuck to the mold is the measure of demoldability.

In **Fig. 7**, demolding data are given on two 70-percent fluorine types, GF and VTR-8600. The GF was run for 10 heats with an average 98 percent of the Orings sticking while the VTR-8600 was run 50 heats with an average sticking rate of 21 percent.

These demolding results clearly show that peroxide-cured VTR-8600 (GF-600S), made with APA technology, gives much less mold sticking than the conventional peroxide-cured FKM GF.

Physical and end-use properties

The physical properties for Advanced Polymer Architecture technology VTR-8600 and VTR-8650 are shown in **Table I**. In general, VTR-8600 and VTR-8650 have a good balance of physical strength and high elongation at break either with a press cure only (no postcure) or with a full postcure.

The modulus at low stress (10-percent elongation) is similar to current peroxide-cured FKM, but the 100-percent modulus is lower and the elongation higher, especially after postcure. This difference in modulus profile would be advantageous for parts, such as O-rings, which must be elongated for part installation.

FKM parts sometimes can be split by overcompression in service. Normal compression seals are compressed (squeezed) about 20 percent, and in a standard ASTM compression set test or a stress relaxation test, the seal is deflected 25 percent. However, in service seals mistakenly can be compressed 35 percent or more and this compression can cause a

standard peroxide-cured FKM seal to split under that compressive force. Finished parts having the higher elongation, lower modulus profile shown by Advanced Polymer Architecture technology VTR-8600 or VTR-8650 indicate that seals made from these materials would be less likely to split from overcompression, thus making a more robust seal from an end user prospective.

Table I. Comparison of VTR-8600 (GF-600S) and VTR-8650 (GBL-600S) to commercial GF and GBL.

	70% fluorine types			68% fluorine types		
	GF	VTR-8600	GF300	GBL900	VTR-8650	GBL200
Mooney, ML 1 + 10 @ 121°C on gum polymer						
	64	60	29	88	64	26
<u>Compound Number</u>	A44-03	A44-04	A44-06	A28-01	A28-02	A28-03
Viton® GF	100	-	-	-	-	-
Viton® VTR-8600	-	100	-	-	-	-
Viton® GF300	-	-	100	-	-	-
Viton® GBL900	-	-	-	100	-	-
Viton® VTR-8650	-	-	-	-	100	-
Viton® GBL200	-	-	-	-	-	100
Zinc Oxide	3	3	3	3	3	3
MT Black (N990)	30	30	30	30	30	30
Diak 7 (TAIC)	3	3	3	3	3	3
Luperox 101XL45	3	3	3	3	3	3
Total phr lab	139	139	139	139	139	139
Mooney Scorch @ 121°C						
Minimum, MU	45	31	19	49	33	19
2 Pt. Rise (min)	19.3	17.6	21.9	15.9	16.4	20.3
5 Pt. Rise (min)	23.0	19.2	23.3	18.7	18.0	23.5
10 Pt. Rise (min)	29.3	20.5	24.3	21.6	19.4	26.2
MDR 2000 @ 177°C, 0.5 Degree Arc, 100 Range, 12 Minute Clock						
M-L (dNm)	2.2	1.9	0.8	2.8	2.1	0.9
ts-2 (min)	0.6	0.4	0.4	0.6	0.4	0.5
t'50 (min)	1.0	0.6	0.6	1.1	0.6	0.7
t'90 (min)	3.3	0.9	1.1	3.3	0.9	1.9
t'95 (min)	4.2	1.2	1.5	4.2	1.2	2.7
M-H (dNm)	14.3	31.7	23.8	18.8	28.0	17.7
Physical Properties @ R.T. - Original (Cure 7' @ 177°C - no postcure)						
M-100, MPa	4.0	3.6	3.4	4.7	2.9	3.1
T-B, MPa	11.5	14.4	12.1	15.4	13.7	13.1
(T-B, psi)	1666	2087	1759	2227	1985	1900
E-B, %	307	341	312	233	345	305
Hardness, A, pts	71	71	70	67	68	66
Physical Properties @ R.T. - Original (Cure 7' @ 177°C - Postcure 16 hrs @ 232°C)						
M-100, MPa	7.3	5.3	5.8	7.3	4.2	4.8
T-B, MPa	18.9	21.4	21.8	20.9	20.5	21.0
(T-B, psi)	2733	3102	3167	3023	2970	3039
E-B, %	184	323	258	174	301	247
Hardness, A, pts	74	76	75	73	73	74

Table I continued.

Compound Number	A44-03 GF	A44-04 VTR-8600	A44-06 GF300	A28-01 GBL900	A28-02 VTR-8650	A28-03 GBL200
	70% fluorine types			68% fluorine types		
Compression Set , Method B , Plied						
22 Hrs @ 200°C						
- No postcure	39	12	18	21	12	24
- PC 2 hrs @ 232°C	33	11	15	-	-	-
- PC 16 hrs @ 232°C	25	15	18	14	15	23
336 Hrs @ 200°C						
- No postcure	76	29	44	54	25	58
- PC 2 hrs @ 232°C	73	27	40	-	-	-
- PC 16 hrs @ 232°C	57	33	41	39	30	47
Low Temperature Testing						
TR-10, °C	-4.8	-4.9	-	-15.1	-15.2	-14.9
Tg (midpoint), °C	-7.0	-6.9	-	-15.8	-17.9	-17.0
Physical Properties @ R.T. - Heat Aged 70 Hrs @ 250°C In Oven						
M-100, MPa	7.8	4.5	5.3	8.1	3.7	4.7
(% Change,M100)	7%	-15%	-7%	10%	-12%	-2%
T-B, MPa	19.8	17.6	18.9	19.1	19.6	18.8
(% Change,T-B)	5%	-18%	-14%	-8%	-4%	-10%
E-B, %	195	356	294	168	357	272
(% Change,E-B)	6%	10%	14%	-4%	18%	10%
Hardness, A,pts	75	77	75	73	73	73
(Pts Change)	1	1	0	0	0	-1
Physical Properties @ R.T. - Heat Aged 70 Hrs @ 275°C In Oven						
M-100, Mpa	4.8	3.1	3.1	6.6	2.9	3.2
(% Change,M100)	-34%	-43%	-46%	-11%	-30%	-35%
T-B, Mpa	14.8	8.1	8.2	14.5	12.5	11.8
(% Change,T-B)	-22%	-62%	-63%	-30%	-39%	-44%
E-B, %	295	416	394	180	423	308
(% Change,E-B)	60%	29%	53%	3%	40%	25%
Hardness, A,pts	74	76	74	74	73	71
(Pts Change)	0	0	-1	1	0	-3
Fluid Immersions - Volume Swell - Tested for 168 hrs @ temperature noted						
Water (distilled), @ 100°C	8.4	3.9	-	8.9	4.3	8.4
Fuel C, @ 23°C	2.4	2.5	-	3.9	4.0	4.2
85/15 Fuel C/Methanol, @23°C	8.3	8.0	-	23	23	22
Methanol, @ 23°C	4.7	3.7	-	53	56	52
Physical Properties @ R.T. - Aged 168 Hrs @ 150°C In ASTM #105 Oil (5W/30)						
M-100, MPa	7.4	5.3	5.6	7.9	4.4	5.5
(% Change,M100)	32%	13%	7%	8%	6%	14%
T-B, MPa	9.6	10.8	10.9	9.7	10.0	10.3
(% Change,T-B)	-41%	-44%	-45%	-54%	-51%	-51%
E-B, %	124	175	161	113	182	154
(% Change,E-B)	-55%	-45%	-44%	-35%	-39%	-38%
Hardness, A,pts	77	76	77	75	76	76
(Pts Change)	3	0	2	2	3	2
Volume change, %	1.4	0.8	1.7	0.9	0.8	0.9

Compression set resistance

Compression set is another key property for any fluoroelastomer product. With a lower modulus and a higher elongation, one might think VTR-8600 and VTR-8650 are simply vulcanized to a lower state of cure. If this were the case, however, the resistance to compression set would be poorer. Compression set was tested on plied discs for 22 and 336 hours at 200°C, and the data are shown on **Table I**. The data show that Advanced Polymer Architecture technology VTR-8600 or VTR-8650 have very good compression set resistance. The other interesting aspect of this data is that the compression set values are quite low with no postcure.

When this trend of low compression set with no postcure was noted, it was decided to try a short, intermediate postcure to see what effect was noted. When experimenting with different hot air postcure conditions with VTR-8600 and VTR-8650, it was noted that most of the benefit of postcuring these new products occurs with a two-hour postcure at 232°C in an air-circulating oven. This short postcure improves the physical properties and slightly improves the compression set. A long 16-hour postcure improves the physical properties but does not improve the compression set resistance beyond what already is seen after two hours.

It would appear that products based on Advanced Polymer Architecture technology can be used with no postcure if the physical properties are sufficient, or with a short two-hour postcure if optimal physical properties are needed.

Stress relaxation resistance

While the compression set properties of VTR-8600 (GF600-S) and VTR-8650 (GBL600-S) are impressive, a more refined test of a compressed seal is stress relaxation. Stress relaxation is a test requirement on many new automotive specifications being developed for fluoroelastomers and its utility and procedure has been the subject of numerous technical publications. Examples of these publications on stress relaxation include a *Rubber & Plastics News* technical article by K. Smith in 1994⁶ and a paper by D. Kotz at the Detroit Rubber Group in 1995.⁷

To test stress relaxation, an experiment was run in a Shawbury-Wallace test jig. Testing was run at 150°C in an air-circulating oven per ISO test method 3384. In this case, VTR-8650 was compared to GBL900 using the same compound recipes found in **Table I**. The testing was run out to a total of 2,000 hours with the GBL900 being taken off test after 1,200 hours.

The results of the test are shown in **Fig. 8**. The data show the Advanced Polymer Architecture technology VTR- 8650 did quite well in this test with a retained sealing force of ~45 percent after 2,000 hours. These data coupled with the compression set results that were run at 200°C indicate VTR-8600 and VTR-8650 should make very good, long lasting compression seals.

Heat aging resistance

Another attribute expected with a high performance material like a fluoroelastomer is heat resistance to temperatures in excess of 200°C. This property was tested and is documented in **Table I** at test temperatures of 250°C and 275°C. VTR-8600 was compared to GF and GF300 while VTR-8650 was compared to GBL900 and GBL200.

The 250°C heat age data show VTR- 8600 and VTR-8650 hold up quite nicely to this exposure. The general trend seen is the Advanced Polymer Architecture technology polymers VTR-8600 and VTR-8650 tend to lose some tensile strength while gaining elongation as they are heat aged. This same trend follows in the 275°C heat age testing.

Fluids resistance

The fluids resistance of VTR-8600 and VTR-8650 are documented in **Table I**. Testing was done in aromatic hydrocarbon fuels such as Fuel C, M15 Fuel, 100- percent methanol and in test oils such as ASTM #105—a 5W/30 motor oil with a very aggressive amine stabilizer package. In addition to this set of fluids, testing was run in hot distilled water at 100°C.

The trends are as expected for the fuels and the methanol. VTX-8600 and GF exhibit low swell in fuel and methanol with VTR-8600 showing slightly lower swell most likely due to its slightly higher fluorine content than GF. VTR-8650 shows quite similar results to GBL900 and GBL200 in fuel and methanol. The most interesting results are the water swell of the Advanced Polymer Architecture technology polymers. VTR-8600 and VTR-8650 show less than half the swell of the commercial GF and GBL type materials in water. The value of less than 5 percent swell in water was obtained using zinc oxide as the acid acceptor as is seen in the recipe on **Table I**. This trend could be quite significant

for parts which need resistance to hot aqueous fluids without requiring the use of a lead-based acid acceptor to obtain low volume swell.

VTR-8600 and VTR-8650 also were tested in ASTM #105 oil, an aggressive simulated 5W/30 motor oil. The results of these tests are shown in **Table I**. The percentage of change of tensile strength and elongation for VTR-8600 and VTR-8650 are quite similar to the commercial types of peroxide-cured fluoroelastomer, but since VTR-8600 and VTR-8650 start with a higher elongation, they still have a higher ultimate elongation after this demanding exposure.

Water and acid resistance

An additional fluid aging experiment was run using mixed compound of VTR- 8600 and VTR-8650 based on the recipes in **Table I**. In this experiment immersions were done in hot distilled water, 96-percent sulfuric acid and nitric acid.

VTR-8600 and VTR-8650 repeated their tendency to swell less in water, and also showed lower changes in modulus and tensile strength than GF and GBL200, respectively. Both VTR-8600 and VTR-8650 showed good resistance to 96-percent sulfuric acid at 70°C, and VTR-8600 showed improved resistance to aggressive nitric acid compared to GF. VTR-8600 showed lower swell, less hardness change and less modulus change after 168 hours at 70°C in nitric acid.

Permeation resistance of VTR-8600

Permeation resistance to fuels and fuel/alcohol blends is important for high fluorine fluoroelastomers. An experiment was run comparing VTR-8600 to GF and GF300 using the modified Thwing Albert cup method, ASTM E96- 66, described in SAE paper 2001-01- 1127 by Stevens.⁸

Permeation testing was done in three fuels, Fuel C, E10 Fuel, which is a 90- percent Fuel C/10-percent Ethanol blend, and M15 Fuel, which is an 85- percent Fuel C/15-percent Methanol blend. The test results shown in **Fig. 9** are in units of gms-mm²/day.

The permeation results show that VTR-8600 is similar to the other 70-percent fluorine types tested. The VTR- 8600 shows somewhat lower permeation rates than GF in oxygenated fuels E10 and M15, but slightly higher rates than GF300.

Conclusions

A new generation of peroxide-cured fluoroelastomers has been introduced. Advanced Polymer Architecture technology polymers VTR-8600 and VTR-8650 show many processing and end-use advantages when compared to peroxidecured polymers that currently are available. These advantages include:

Fig. 7. Demolding data.

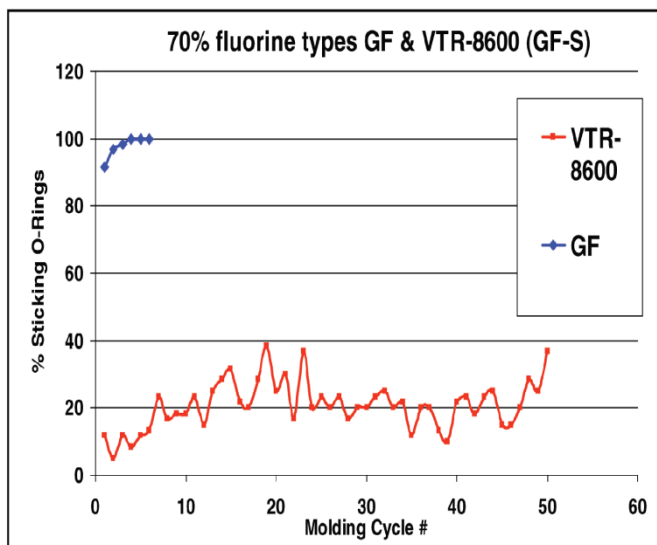


Fig. 8.

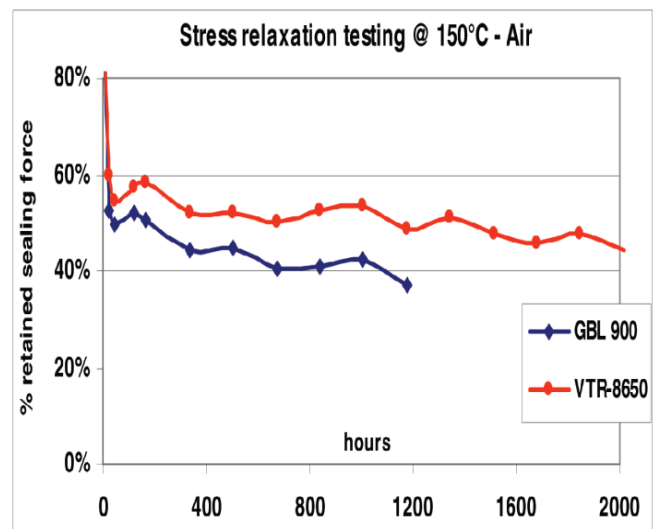


Fig. 9.

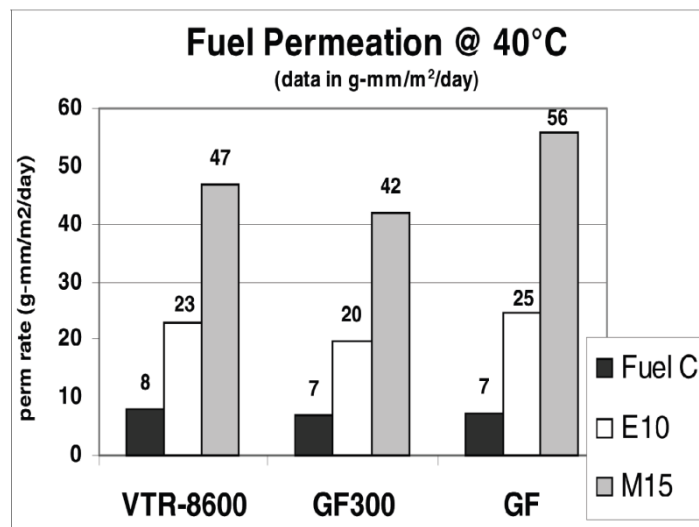


Fig. 10. Ingredients list.

Name	Ingredient type	Supplier
Armeen® 18D	process aid	Akzo Nobel
Diak™ 7	coagent (TAIC)	DuPont Dow Elastomers LLC
Elastomag® 170	metal oxide (MgO)	Morton International Inc.
Luperox® 101XL-45	peroxide	ATOFINA Chemicals, Inc
Struktol® WS-280	process aid	Struktol Company of America
Varox® 130XL	peroxide	R.T. Vanderbilt
Viton® GF, GF300	fluoroelastomer	DuPont Dow Elastomers LLC
Viton® GBL200, 900	fluoroelastomer	DuPont Dow Elastomers LLC
VPA 2	process aid	DuPont Dow Elastomers LLC

Viton® fluoroelastomer is a registered trademark of DuPont Dow Elastomers LLC

Processing advantages for new APA polymers VTR-8600 (GF-600S) and VTR-8650 (GBL-600S)

- faster curing;
- better mold flow/improved rheology/ less shear heating (scorch safety under shear);
- better hot tear and tensile strength;
- better hot modulus vs. strain profile helps demolding;
- better mold release;
- lower scrap due to molding problems; and
- low or no postcure capability.

End-use advantages for new APA polymers VTR-8600 (GF-600S) and VTR-8650 (GBL-600S)

- more robust physical properties for seal installation and use (higher elongation);
- lower compression set/better longterm sealing;
- improved stress relaxation (longterm sealing force retention);
- low or no postcure capability—parts can be bonded to thermoplastic carrier and do not need to be postcured;
- improved water resistance/lower water swell without the use of a lead oxide;
- improved nitric acid resistance; and
- competitive fluid, permeation and additive resistance compared to current peroxide-cured fluoroelastomers.

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