CERAMIC REINFORCED TECHNOLOGIES

CERFLON® and CERTEX™ FluoroCeramic Chemistry

CERFLON®
FLUOROCERAMIC TECHNOLOGY

Cerflon® Data Booklet

A Guide and Manual

VOLUME 3 | August 2015

Section II: CERFLON® Data Booklet

“Ceramic Reinforced Fluoropolymer” Patent Data

The “Ceramic Reinforced Fluoropolymer” US Patent 5,783,308 is a good place to start looking at the data as it was the basis for all of the research and development work going forward.

Following are the two test protocols and the data that was developed to support the patent claims. These are only excerpts and the whole content of all of the cited patents are included in the Appendix:

EXAMPLE 1:
The resistance to abrasion of four fluids were investigated in this example using the Ball on Cylinder Lubricity Evaluator (BOCLE) (ASTM D 5001). The ball on cylinder lubricity evaluator assesses the wear aspects of the boundary lubrication properties of a fluid. The fluid under test is placed in a test reservoir in which atmospheric air is maintained at 10% relative humidity. A non-rotating steel ball is held in a vertically mounted chuck and forced against an axially mounted steel ring with an applied load. The test cylinder is rotated at a fixed speed while being partially immersed in the fluid reservoir. This maintains the cylinder in a wet condition and continuously transports the test fluid to the ball/cylinder interface. The wear scar generated on the test ball is a measure of the fluid lubricating properties.

The first fluid tested was 100 weight percent of a 150 solvent refined paraffinic petroleum base oil. The second fluid tested was a combination of 98.0 weight percent of a 150 solvent refined paraffinic petroleum base oil and 2.0 weight percent of 10 wt % boron nitride/150 solvent refined paraffinic base oil dispersion. The boron nitride dispersion used was JB 33018. The resulting second fluid included 0.2 wt % boron nitride and 98.8 wt % 150 solvent refined paraffinic base oil. The third fluid was a stabilized dispersion of PTFE. The stabilized PTFE dispersion included 95.0 weight percent of a 150 solvent refined paraffinic petroleum base oil and 5.0 weight percent of a 20 wt % dispersion of PTFE in 150 solvent refined paraffinic base oil. The resulting third fluid included 1.0 wt % PTFE and 99.0 wt % 150 solvent refined paraffinic base oil. The fourth fluid consisted of a mixture including 93.0 weight percent of a 150 solvent refined paraffinic petroleum base oil, 5.0 weight percent of the PTFE dispersion, and 2.0 weight percent boron nitride dispersion to give a fourth fluid including 1.0 wt % of PTFE, 0.2 wt % boron nitride, and 98.8 wt % 150 solvent refined paraffinic base oil carrier.

The results of the BOCLE tests are set forth in Table I below:

TABLE I

Composition No.	Scuff Depth (MM)
1	0.72
2	0.635
3	0.58
4	0.53

These results indicate that a synergism is achieved by combining a dispersion of PTFE with a dispersion of boron nitride. Such a dispersion (composition 4) exhibited a 10 percent wear/friction improvement when compared to a PTFE dispersion alone, and 20 percent wear/friction improvement over a boron nitride dispersion alone.

EXAMPLE 2:

The Example demonstrates that Boron Nitride acts as a reinforcing agent in thin TEFLON® coatings to improve the ability of the TEFLON® to resist being worn away by abrasion.

A. Control Test

A clean stainless steel capillary die with internal capillary dimensions of 0.05 cm diameter times 2.54 cm length times 180 degree entrance angle was placed in an Instron capillary rheometer. A polyethylene with a melt index of one, commercially available as D7047 from Union Carbide, and containing 2.5% colloidal silica abrasive was extruded through the capillary at a temperature of 220 degree C. and at a shear rate of 700 s-.sup.1.

The pressure required to extrude the abrasive polyethylene through the capillary die remained constant at 0.5 MPa throughout the one hour duration of the test.

B. Capillary coated with Teflon, no Boron Nitride

The capillary die used in the previous example was thoroughly cleaned by burning out any residue at 800.degree. C. The capillary was coated with TE 5121A fluoropolymer manufactured by DuPont De Nemours, Wilmington, Del. which is a 3% by wt. solution of a TEFLON® fluoropolymer dissolved in a perfluorinated solvent. The coated capillary was dried for 4 hours at 200 degree C. and was placed in the Instron rheometer and the same polyethylene containing the abrasive was extruded through the capillary at a temperature of 220.degree C. and a shear rate of 700 s-.sup.1.

Result of Test B:

The pressure required to extrude the abrasive polyethylene through the capillary die was initially 0.2 MPa, which is less than half the pressure required to extrude the polyethylene through the capillary die in the previous example. This reduction in pressure is the result of the presence of the coating that allows the polyethylene to slip at the wall of the capillary and thus flow through the die more easily. The pressure remained constant at 0.2 MPa for about 6 minutes and then increased slowly to 0.5 MPa and then remained constant at this value, which is the same pressure as observed in the uncoated capillary. The slow increase in pressure up to the value observed for the capillary that was not coated with TEFLON® is due to the slow wearing away of the TEFLON® coating by the abrasive colloidal silica in the polyethylene.

C. Capillary coated with Teflon and Boron Nitride

The capillary die used in the previous examples was thoroughly cleaned by burning out any residue at 800 degree C. The capillary was coated with an admixture a 3% solution of TEFLON® TE 5121A fluoropolymer and 1% by wt boron nitride fine powder. The coated capillary was dried for 4 hours at 200.degree C. and was placed in the Instron rheometer and the same polyethylene containing the abrasive was extruded through the capillary at a temperature of 220 degree C. and a shear rate of 700 s-sup.1.

Result of Test C:

The pressure required to extrude the abrasive polyethylene through the capillary die was initially 0.2 MPa, which is the same as the capillary coated with only TEFLON®, but is less than half the pressure required to extrude the polyethylene through the uncoated capillary. This reduction in pressure is again the result of the presence of the Teflon coating that allows the polyethylene to slip at the wall of the capillary and thus flow through the die more easily. The pressure remained constant at 0.2 MPa for about 20 minutes and then increased slowly to 0.5 MPa after about one hour. The pressure then remained constant at 0.5 MPa, which is the same pressure as observed in the uncoated capillary. The slow increase in pressure up to the value observed for the capillary that was not coated with TEFLON® and boron nitride is again due to the slow wearing away of the TEFLON® and boron nitride coating by the abrasive colloidal silica in the polyethylene. Thus, the use of a boron nitride/TEFLON® coating dispersion significantly increased the time required for the abrasive to wear away the coating.

While the present invention has been described by means of specific embodiments, it will be understood that modifications may be made without departing from the spirit of the invention. The scope of the invention is not to be considered as limited by the description of the invention set forth in the specification and examples, but rather as defined by the following claims.

* * * *

Some interesting "Application Patents" that incorporate CERFLON®

Following is some background regarding a recent Xerox patent application (there is also an issued patent) that is a powerful statement for Cerflon®.

CRT worked on this project for over two years with the Xerox researcher/applicant, Tim Bender, Ph.D., who is now a Full Professor at University of Toronto. For credibility sake, following are Dr. Bender's credentials:

"Prof. Bender is an established expert in the field of organic electronic materials, specifically in their industrial application to organic photoreceptive devices. He has filed over 50 US Patents (17 currently issued) and authored or co-authored 17 peer reviewed papers in the fields of organic electronic materials and polymeric structures. While at Xerox he participated in and chaired several high level committees including a committee reporting directly to the Chief Technology Officer of Xerox Corp. (Global)."

The Xerox patent application cites fluoroceramic (Cerflon®) chemistry and does it quite often. They even use the Cerflon® trademark within the application. In the immediately following excerpts, references to Cerflon® and FluoroCeramic powders along with the results of testing are highlighted in blue. In the complete patent document there are simple comments throughout the application which are indicated by the yellow balloons adjacent to the PTFE, Boron Nitride (BN), PTFE/BN and Cerflon® references. The references to all of the above can be found on the following .pdf pages: 1, 5, 8, 11, 12, 15, 16, 19, 20, 23, 24 and 27. *Please note, these are the .pdf page numbers and not the patent application page numbers.* In the examples on .pdf pages 18 and 19 they show "Example 1" with Cerflon® and "Example 2" with Solvay Solexis FLUORLINK S-10 PFPE and no Cerflon®. Later in "Example 2" they indicate the favorable Cerflon® results! Not only did Cerflon® ($) perform better than the PFPE ($$$$) the SLA 2020 PTFE/BN dispersion is a far less expensive solution.

Following are excerpts from the XEROX 1-US 20080166643 patent application:

Overcoat layers, which may be useful for reducing torque in electrophotographic photoreceptors, are provided. The overcoat layers include a polymer matrix having a particulate inorganic lubricant and a particulate fluoropolymer uniformly dispersed therein. Also provided are electrophotographic photoreceptors that include a substrate and the overcoat layers, electrophotographic imaging apparatuses that include such photoreceptors, and methods for forming the photoreceptors.

[0082] The particulate inorganic lubricant and particulate fluoropolymer may be used individually or as composites or mixtures of particulate inorganic lubricants and particulate fluoropolymers. Such composites and mixtures are commercially available and include, for example, a commercially available line of particulate boron nitride and polytetrafluoroethylene (PTFE) from Acheson Colloidal Company, in which boron nitride, PTFE and mixtures thereof are available as dispersions in either alcohol or hydrocarbon. Particles size ranges for these particles are around 1 -5μ. Other commercially available colloidal dispersions include Colloidal PTFE* Emralon® 309 available as a dispersion in Anhydrous Isopropyl Alcohol 20% by weight, Colloidal Boron Nitride SLA 1720 available as a dispersion in Anhydrous Isopropyl Alcohol 20% by weight, Colloidal PTFE* SLA 1612 available as a dispersion in 150 Solvent Refined Paraffinic Petroleum Oil 20% by weight, Colloidal PTFE SLA 1614 available as a dispersion in 150 Solvent Refined Paraffinic Petroleum Oil 20%, by weight, Colloidal Boron Nitride SLA 1710 available as a dispersion in 150 Solvent Refined Paraffinic Petroleum Oil 10% wt, Cerflon® (PTFE/BN) SLA 2020 available as a dispersion in Anhydrous Isopropyl Alcohol 18% by weight and Cerflon® (PTFE/BN) SLA 2010 available as a dispersion in 150 Solvent Refined Paraffinic Petroleum Oil 18% by weight.

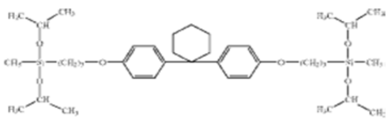
EXAMPLE 1:

[0130] An electrophotographic photoreceptor was prepared in the following manner. A coating solution for an undercoat layer comprising 100 parts of a zirconium compound (trade name: Orgatics ZC540), 10 parts of a si lane compound (trade name: Al 10, manufactured by Nippon Unicar Co., Ltd), 400 parts of isopropanol solution and 200 parts of butanol was prepared. The coating solution was applied onto a cylindrical Al substrate subjected to honing treatment by dip coating, and dried by heating at 150° C. for 10 minutes to form an undercoat layer having a film thickness of 0.1 micrometer.

[0131] A 0.5 micron thick charge generating layer was subsequently dip coated on top of the undercoat layer from a dispersion of Type V hydroxygallium phthalocyanine (12 parts), alkylhydroxy gallium phthalocyanine (3 parts), and a vinyl chloride/vinyl acetate copolymer, VMCH (Mn=27,000, about 86 weight percent of vinyl chloride, about 13 weight percent of vinyl acetate and about 1 weight percent of maleic acid) available from Dow Chemical (10 parts), in 475 parts of n-butylacetate.

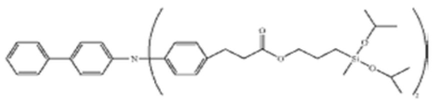
[0132] Subsequently, a 20µ thick charge transport layer (CTL) was dip coated on top of the charge generating layer from a solution of N,N'-diphenyl-N,N'-bis (3-methylphenyl)l,l-biphenyl-4,4'-diamine (82.3 parts), 2.1 parts of 2,6-Ditert-butyl-4-methylphenol (BHT) from Aldrich and a polycarbonate, PCZ-400 [poly(4,4'-dihydroxy-diphenyl-l-lcyclohexane), Mw=40,000] available from Mitsubishi Gas Chemical Company, Ltd. (123.5 parts) in a mixture of 546 parts of tetrahydrofuran (THF) and 234 parts of monochlorobenzene. The CTL was dried at 115° C. for 60 minutes. [0133] An overcoat layer formulation was prepared as follows:

[0134] Step 1. 5.8 parts of a compound of Formula (I-A) as shown below, 11 parts of a compound of Formula (II-G) as shown below, and 11 parts of methanol were mixed, and 2 parts of an ion exchange resin (Amberlist H15) were added thereto, followed by stirring for 2 hours.



Compound of Formula (I-A)

[0135]



Compound of Formula (II-G)

[0136] Step 2. 32 parts of butanol and 4.92 parts of distilled water were added to the mixture, followed by stirring at room temperature for 30 minutes. Then, the resulting mixture was filtered to remove the ion exchange resin.

[0137] Step 3. 0.180 parts of aluminum trisacetylacetonate (Al(AcAc)3), 0.180 parts of acetylacetone (AcAc), 2 parts of a polyvinyl butyral resin (trade name: BX-L, manufactured by Sekisui Chemical Co., Ltd.), 0.0180 parts of butylatedhydroxytoluene (BHT), 0.261 parts of a hindered phenol antioxidant (IrGANGX 1010), and 4.5 parts of CERFLON SLA-2020 (a commercially available isopropanol dispersion comprising 22 weight percent of particulate boron nitride and polytetrafluoroethylene composites, purchased from Acheson, Inc.) were added to the filtrate obtained in Step 2 and thoroughly mixed therein for 2 hours. The mixture was filtered through a 6µ glass fiber filter to obtain a coating solution for an overcoat layer. The coating solution thus prepared was applied onto a charge transfer layer by dip coating and dried by heating at 130° C. for one hour to form the protective layer having a film thickness of around 3µ thereby obtaining a desired electrophotographic photoreceptor.

EXAMPLE 2:

[0138] An electrophotographic photoreceptor was prepared in a similar manner as described in Example 1, except that the overcoat solution was further added in Step 3 with 0.06 part of FLUOROLINK S-10 (a perfluoropolyether purchased from Solvay Solexis, Inc.). The coating solution thus prepared was applied onto a charge transfer layer by dip coating and dried by heating at 130° C. for one hour to form the protective layer having a film thickness of around 3 µ, thereby obtaining a desired electrophotographic photoreceptor.

[0139] Photoreceptor Device: Comparative Example

[0140] A comparative example of electrophotographic photoreceptor was prepared in a similar manner as described in Example 1, except that no CERFLON SLA-2020 was added in the preparation of overcoat solution.

[0141] Evaluation of Electrophotographic Photoreceptor Performance:

[0142] The electrical performance characteristics of the above prepared photoreceptors such as electrophotographic sensitivity and short term cycling stability were tested in a scanner. The scanner is known in the industry and equipped with means to rotate the drum while it is electrically charged and discharged. The charge on the photoconductor sample is monitored through use of electrostatic probes placed at precise positions around the circumference of the device. The photoreceptor devices are charged to a negative potential of 500 Volts. As the devices rotate, the initial charging potentials are measured by voltage probe 1. The photoconductor samples are then exposed to monochromatic radiation of known intensity, and the surface potential measured by voltage probes 2 and 3. Finally, the samples are exposed to an erase lamp of appropriate intensity and wavelength and any residual potential is measure by voltage probe 4. The process is repeated under the control of the scanner's computer, and the data is stored in the computer. The PIDC (photo induced discharge curve) is obtained by plotting the potentials at voltage probes 2 and 3 as a function of the light energy. All the photoreceptors as prepared in Examples 1 and 2 showed similar PIDC characteristics as the control or Comparative Example device.

[0143] The electrical cycling performance of the photoreceptor was performed using a fixture similar to a xerographic system. The photoreceptor devices (Example 1, Example 2, and the comparative example) with the overcoat showed stable cycling of over 170,000 cycles in a humid environment (28° C, 80% RH).

[0144] The electrical testing results of the photoreceptors as measured above indicate that the addition of the particulate boron nitride and PTFE has minimal impact on the electrical characteristics of the photoreceptors.

[0145] The torque properties, measured in Newton-meter, of the photoreceptor are measured in the following manner. A photoreceptor was placed in a xerographic customer replaceable unit (CRU), as is used in a DC555 (manufactured by Xerox Corporation). The torque properties of the photoreceptor were measured before and after 500 prints with DC555. As a result, the photoreceptors as fabricated in Example 1 and 2 maintained a low torque before and after print test. As a comparison, the comparative photoreceptor displayed a low torque, but its torque increased more than 20% after the print test. The results show that the addition of the particulate boron nitride and PTFE in the overcoat offers torque improvement.

[0146] The image quality of the photoreceptors containing the composite overcoat was evaluated by print test using a printing machine equipped with the electrophotographic photoreceptor described herein under an ambient environment (for example, 23° C. and 65% relative humidity). No adverse impact on initial image quality and the image quality after 1,000 prints was observed.

EXAMPLE 3:

[0147] An electrophotographic photoreceptor having a backing layer (or anti-curling layer) was prepared in the following manner:

[0148] A photoconductor was prepared by providing a 0.02 micrometer thick titanium layer coated (the coater device) on a biaxially oriented polyethylene naphthalate substrate (KALEDEXTM 2000) having a thickness of 3.5 mils, and applying thereon, with a gravure applicator, a solution containing 50 parts of 3-amino-propyltriethoxysilane, parts of water, 15 parts of acetic acid, 684.8 parts of denatured alcohol, and 200 parts of heptane. This layer was then dried for about 5 minutes at 135° C. in the forced air dryer of the coater. The resulting blocking layer had a dry thickness of 500 Angstroms. An adhesive layer was then prepared by applying a wet coating over the blocking layer, using a gravure applicator, and which adhesive contains 0.2 percent by weight based on the total weight of the solution of copolyester adhesive (Ardel D100TM available from Toyota Hsutsu Inc.) in a 60:30: 10 volume ratio mixture of tetrahydrofuran/monochlorobenzene/methylene chloride. The adhesive layer was then dried for about 5 minutes at 135° C. in the forced air dryer of the coater. The resulting adhesive layer had a dry thickness of 200 Angstroms.

[0149] A photogenerating layer dispersion was prepared by introducing 0.45 parts of the known polycarbonate lupilon 200TM (PCZ-200) or Polycarbonate ZTM, weight average molecular weight of 20,000, available from Mitsubishi Gas Chemical Corporation, and 50 parts of tetrahydrofuran into a glass bottle. To this solution were added 2.4 parts of hydroxygalli- phthalocyanine (Type V) and 300 parts of Vs-inch (3.2 millimeters) diameter stainless steel shot. This mixture was then placed on a ball mill for 8 hours. Subsequently, 2.25 parts of PCZ-200 were dissolved in 46.1 parts of tetrahydrofuran, and added to the hydroxygalli phthalocyanine dispersion. This slurry was then placed on a shaker for 10 minutes. The resulting dispersion was, thereafter, applied to the above adhesive interface with a Bird applicator to form, a photogenerating layer having a wet thickness of 0.25 mil. A strip about 10 millimeters wide along one edge of the substrate web bearing the blocking layer and the adhesive layer was deliberately left uncoated by any of the photogenerating layer material to facilitate adequate electrical contact by the ground strip layer that was applied later. The charge generation layer was dried at 135° C. for 5 minutes in a forced air oven to form a dry photogenerating layer having a thickness of 0.4 micrometer.

[0150] The above photogenerating layer was overcoated with a charge transport layer prepared by introducing into an amber glass bottle 45 weight percent of N,N'-bis (3-methylphenyl)-N,N'-diphenyl-l,l-biphenyl-4,4'-diamine and 55 weight percent of Makrolon 5705®, a known polycarbonate resin having a molecular weight average of from about 50,000 to about 100,000, commercially available from Farbenfabriken Bayer A.G. The resulting mixture was then dissolved in methylene chloride to form a solution containing 15 percent by weight solids. This solution was applied on the photogenerating layer to form the bottom layer coating that upon drying (120° C. for 1 minute) had a thickness of 30 microns. [0151] The back of the above polyethylene naphthalate substrate was coated with an anti-curling layer of a polycarbonate comprising particulate boron nitride and PTFE. The coating solution can be prepared by dispersing 3 weight percent (solid content) of commercially available SLA2010 (purchased from Acheson, Inc.) and 97 weight percent of Makrolon 5705®, commercially available from Farbenfabriken Bayer A.G. in methylene chloride. The thickness of the layer after drying (120° C. for 1 minute) arranges from 5 to 25 microns. The improved backing layer is expected to offer lower friction and improved mechanical robustness. [0152] It will be appreciated that various of the above discussed and other features and functions, or alternatives thereof, may be desirably combined into many other different systems or applications.

Also that various presently unforeseen or unanticipated alternatives, modifications, variations or improvements therein may be subsequently made by those skilled in the art which are also intended to be encompassed by the following claims.

1. An overcoat layer for electrophotographic imaging members, comprising:

a polymer matrix;

a particulate inorganic lubricant; and

a particulate fluoropolymer;

Wherein the particulate inorganic lubricant and the particulate fluoropolymer are uniformly dispersed throughout the matrix.

2. The overcoat layer according to claim 1, wherein said particulate inorganic lubricant is selected from the group consisting of boron nitride, graphite, fluorinated graphite, oxidized graphite, molybdenum sulfide, and mixtures thereof.

3. The overcoat layer according to claim 1, wherein said particulate fluoropolymer is selected from the group consisting of poly(tetrafluoroethylene) (PTFE), poly(vinylidenefluoride), poly(vinylidene fluoride co-hexafluoropropylene), and mixtures thereof.

4. The overcoat layer according to claim 1, wherein said particulate inorganic lubricant comprises plurality of particles ranging in size of from about 0.05 to about 5μ, and said particulate fluoropolymer comprises a plurality of particles ranging in size of from about 0.05 to about 5μ.

5. The overcoat layer according to claim 1, wherein said particulate inorganic lubricant comprises a plurality of boron nitride particles ranging in size of from about 0.05 to about 5μ, and said particulate fluoropolymer comprises a plurality of poly(tetrafluoroethylene) particles ranging in size of from about 0.05 to about 5μ

6. The overcoat layer according to claim 1, wherein said particulate inorganic lubricant is present at from about 0.5 to about 10% by weight, relative to a total weight of the overcoat layer, and said particulate fluoropolymer is present at from about 1 to about 20% by weight, relative to a total weight of the overcoat layer.

And so on through Claim 30 **(See included full patent application in Appendix)**

The following Xerox patent also teaches that dry powders are an option

1-US 7734244 Xerox overview:

An image development method implemented on a substrate conditioning unit for the drying and compression of a print substrate in an electrophotographic imaging process. The conditioning unit includes a heating mechanism for maintaining the heat of compression rollers, belts or nip rollers for compressing the substrate by a determined distance between the conditioning rollers, or by direct contact between a compression roller and a nip roller, and having an abhesive layer to prevent adhesion of the conditioning unit to the print substrate.

The particulate inorganic lubricant and particulate fluoropolymer in the overcoat layer of embodiments may be present in any suitable amounts. However, in particular embodiments, the particulate inorganic lubricant may be present in amounts from about 1 to about 20% by weight, 25 relative to a total weight of the overcoat layer, and/or the particulate fluoropolymer may be present in amounts from about 5 to about 10% by weight, relative to a total weight of the overcoat layer.

The particulate inorganic lubricant and particulate fluoropolymer may be used individually or as composites or mixtures of particulate inorganic lubricants and particulate fluoropolymers. Such composites and mixtures are commercially available and include, for example, a commercially available line of particulate boron nitride and polytetrafluoroethylene (PTFE) from Acheson Colloidal Company, in which boron nitride, PTFE and mixtures thereof are available as dispersions in organic solvents of either alcohol or hydrocarbon. However, both inorganic and organic particulates are also commercially available as dry powder readily for coating solution dispersion. Particle size suitable for the present disclosure ranges from about 0.1 to about 5 µ.

Garlock Sealing Technologies (An EnPro Company (NYSE – NPO)

Boron Nitride filled PTFE, US Patent 7,811,490, October 12, 2010

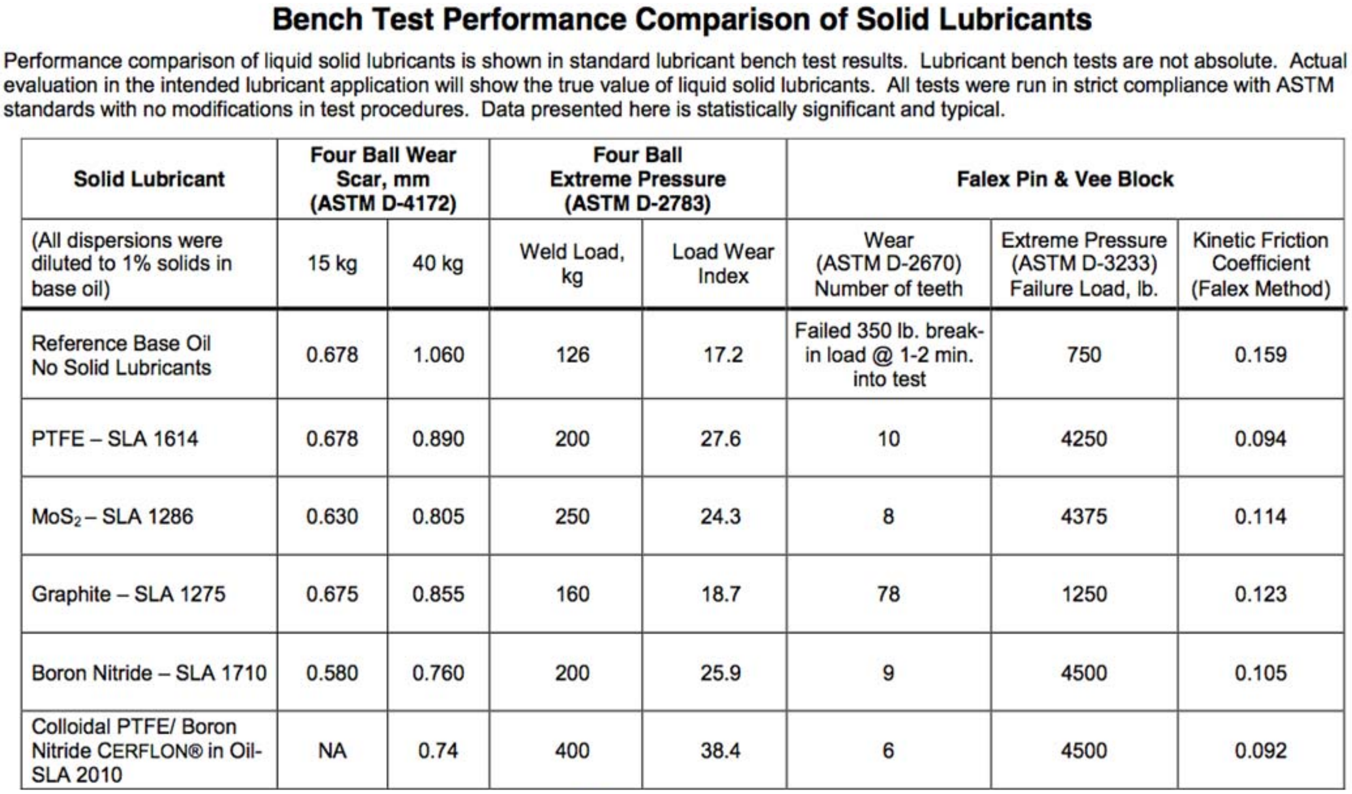
Ameet Kulkarni of Garlock Sealing Technologies developed a PTFE gasket filled with Boron Nitride particles. The Boron Nitride filled PTFE product is an alternative to graphite-filled PTFE by having improved sealability, greater resistance to permeation, and less contamination due to the white color of the Boron Nitride filled PTFE.

Abstract

A composition is provided that, in one aspect, may be used in the production of sealing materials, such as gaskets. The composition includes a polytetrafluoroethylene matrix and a Boron Nitride filler. In one aspect, the Boron Nitride filler may be provided as a hexagonal, close-packed, Boron Nitride filler that is homogeneously dispersed within the polytetrafluoroethylene matrix. In at least one embodiment, the composition is formed by combining quantities of polytetrafluoroethylene, Boron Nitride filler, hydrocarbon liquid, and solvent. The liquid and solvent may be removed through various processes prior to sintering the composition to form a full-density, Boron Nitride filled, polytetrafluoroethylene matrix that exhibits improved sealability, greater resistance to permeation, and less color contamination.

There is a subsequent continuation application dated 09/23/10, 1-US 2010_0331468 A1 - BN filled PTFE with a new set of claims.

FluoroCeramic Cerflon® Data



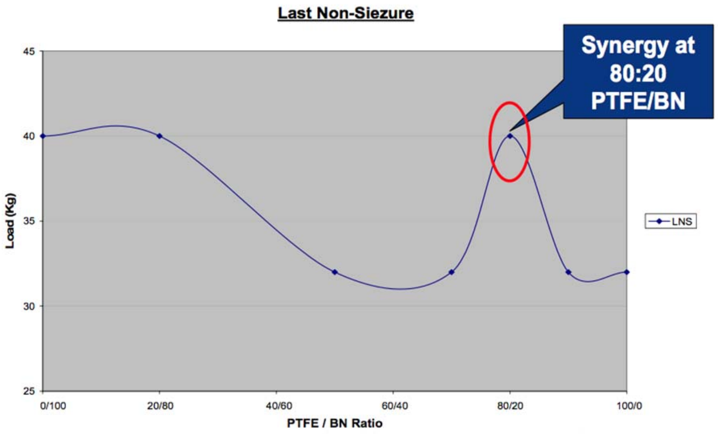
Bench Test Performance Comparison of Solid Lubricants

Performance comparison of liquid solid lubricants is shown in standard lubricant bench test results. Lubricant bench tests are not absolute. Actual evaluation in the intended lubricant application will show the true value of liquid solid lubricants. All tests were run in strict compliance with ASTM standards with no modifications in test procedures. Data presented here is statistically significant and typical.

Solid Lubricant	**Four Ball Wear Scar, mm (ASTM D-4172)**		**Four Ball Extreme Pressure (ASTM D-2783)**		**Falex Pin & Vee Block**		
(All dispersions were diluted to 1% solids in base oil)	15 kg	40 kg	Weld Load, kg	Load Wear Index	Wear (ASTM D-2670) Number of teeth	Extreme Pressure (ASTM D-3233) Failure Load, lb.	Kinetic Friction Coefficient (Falex Method)
Reference Base Oil No Solid Lubricants	0.678	1.060	126	17.2	Failed 350 lb. break-in load @ 1-2 min. into test	750	0.159
PTFE – SLA 1614	0.678	0.890	200	27.6	10	4250	0.094
MoS_2 – SLA 1286	0.630	0.805	250	24.3	8	4375	0.114
Graphite – SLA 1275	0.675	0.855	160	18.7	78	1250	0.123
Boron Nitride – SLA 1710	0.580	0.760	200	25.9	9	4500	0.105
Colloidal PTFE/ Boron Nitride CERFLON® in Oil-SLA 2010	NA	0.74	400	38.4	6	4500	0.092

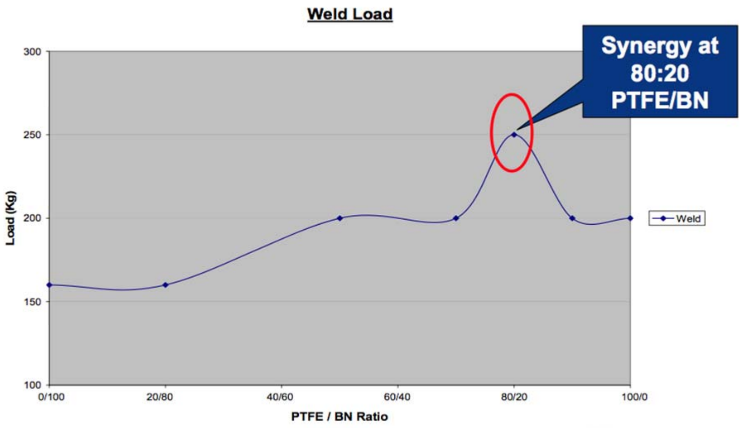
FluoroCeramic Cerflon® Ladder Studies

Four Ball 40Kg Wear



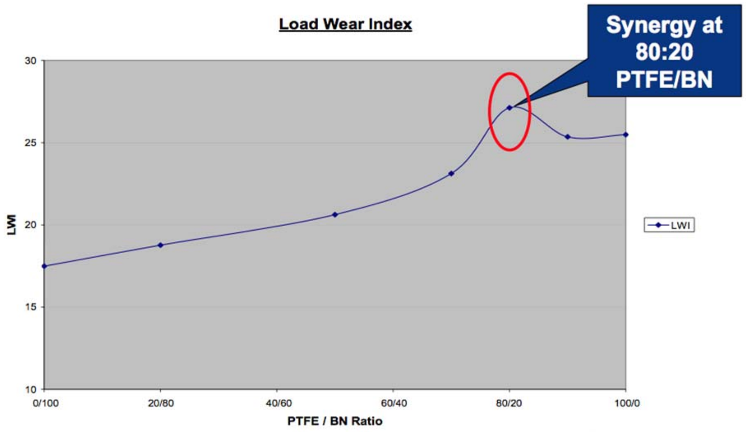
Dispersion Formulation Science

Acheson A member of the ICI Group



Dispersion Formulation Science

Acheson A member of the ICI Group



Dispersion Formulation Science

Acheson A member of the ICI Group

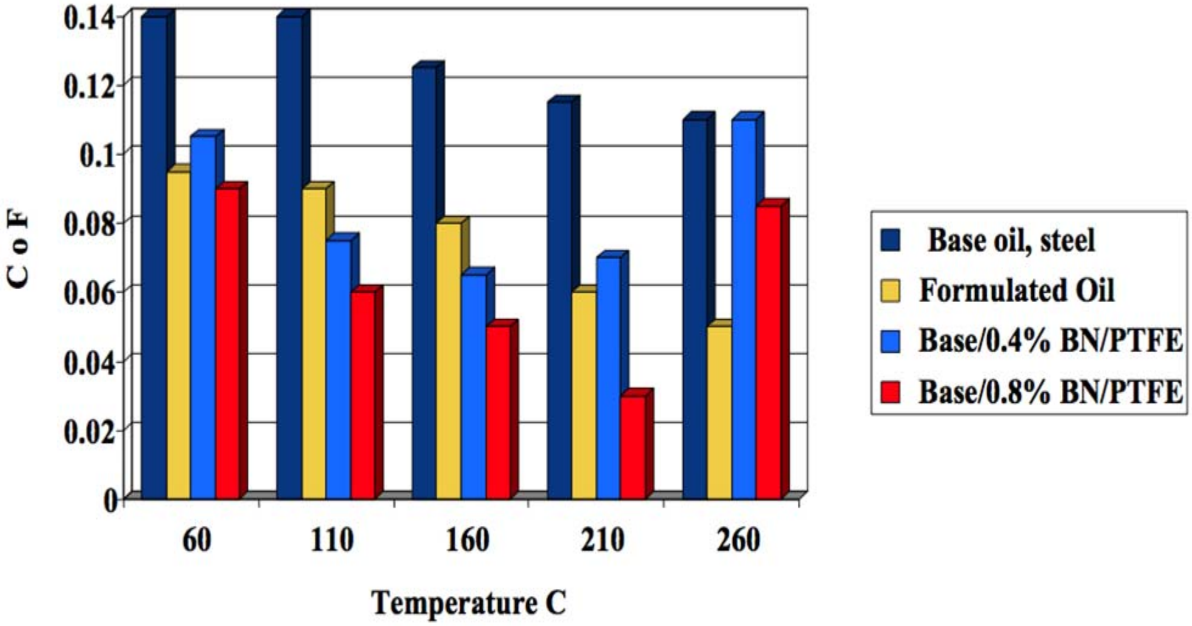
FluoroCeramic Cerflon® Data – HFRR Cerflon® on DLC

- The following HFRR (High Frequency Reciprocation Rig) tests were conducted in 2004 and show "Coefficient of Friction" measurements with BN/PTFE in oil alone at varying temperatures. They were conducted on both steel and ceramic materials.

- HFRR is a micro processor-controlled reciprocating friction and wear test system that provides a fast, repeatable assessment of the performance of fuels and lubricants. It is particularly suitable for boundary friction measurements of engine oils, greases and other compounds. It is also used for boundary friction coefficient measurements for automobile fuel economy modeling.

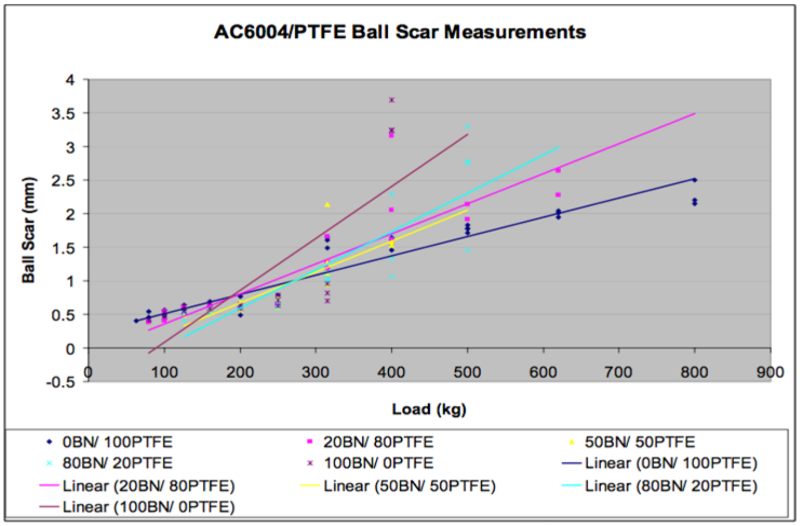
- HFRR was developed in consultation with a major fuel additive manufacturer and the Tribology Laboratory of Imperial College, London, there are currently over 400 systems in use in more than 40 countries worldwide.

In the table below, the Coefficient of Friction of the base oil with Cerflon® at 0.4% and 0.8% solids outperformed the fully formulated oil at several temperatures.



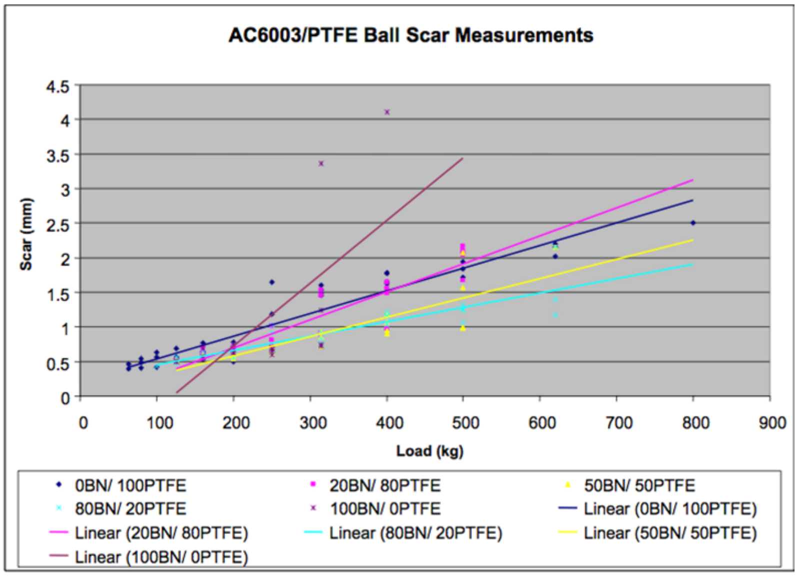
Momentive Performance Materials Data Wear Scar

AC6004 shows improvement over PTFE alone at loads below 300 kg.



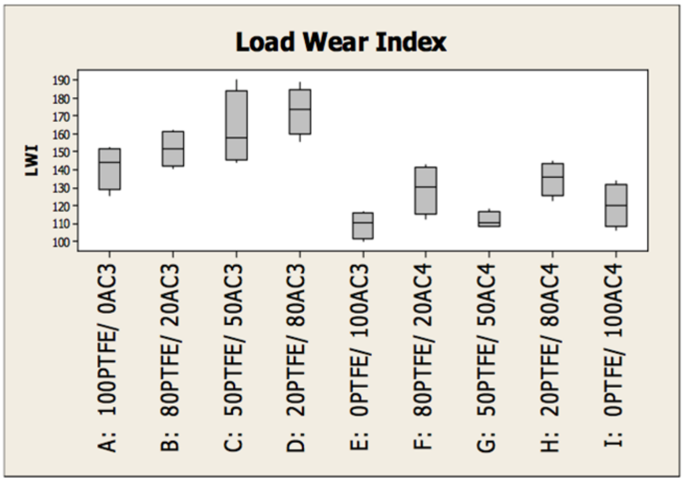
Momentive Performance Materials Data Wear Scar

50% and 80% AC6003 show improvement in wear scar at all loads compared to PTFE alone or with just 20% BN



Momentive Performance Materials Data
Load Wear Index

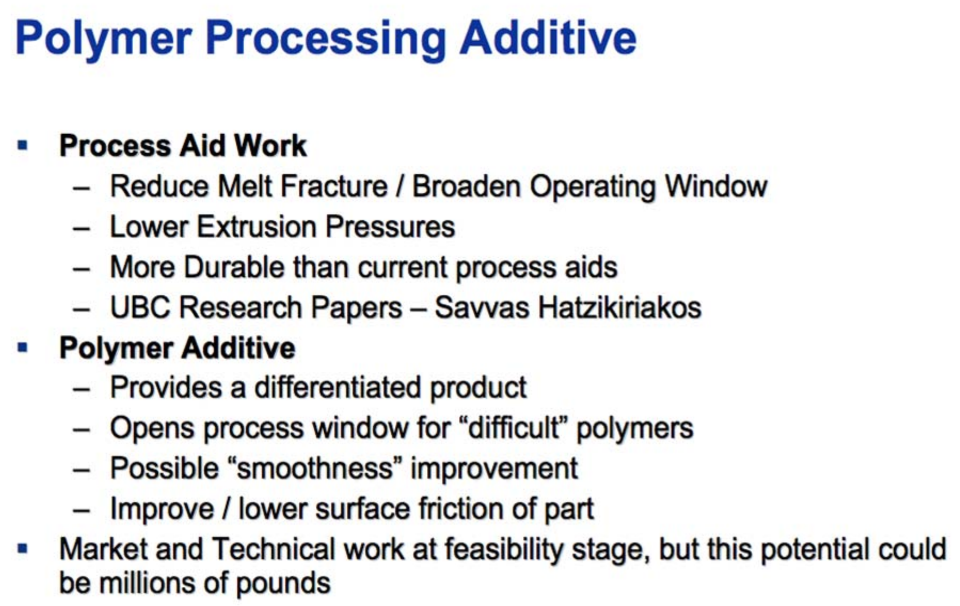
- AC6003: Synergy between BN and PTFE
- AC6004: No improvement over PTFE alone
- Strongly correlates with Acheson ladder study



Summary of Data

- PTFE / BN blend show improved lubrication versus single lubricants additives – PTFE, MoS_2, graphite, BN
- Synergistic effect between PTFE and BN
  - A reinforced fluoropolymer occurs when Boron Nitride, which is stronger and tougher, is introduced into the matrix of a fluoropolymer thereby “reinforcing” the polymer
  - Boron Nitride has similar surface energy as fluoropolymers enabling it to fully wet fluoropolymer
- White color compared to graphite and moly
- Economic advantage over use of BN only
- Memory effect for possible additional protection

FluoroCeramic Cerflon® as a Polymer Processing Additive



Polymer Processing Additive

- **Process Aid Work**
  - Reduce Melt Fracture / Broaden Operating Window
  - Lower Extrusion Pressures
  - More Durable than current process aids
  - UBC Research Papers – Savvas Hatzikiriakos
- **Polymer Additive**
  - Provides a differentiated product
  - Opens process window for “difficult” polymers
  - Possible “smoothness” improvement
  - Improve / lower surface friction of part
- Market and Technical work at feasibility stage, but this potential could be millions of pounds

FluoroCeramic Cerflon® versus Molybdenum Disulfide (MoS_2)

1. **Cerflon® outperforms MoS_2 based on all of our current data.**
2. **It is not a commodity and is patent protected.**
3. **It has a trademarked name instead of Molybdenum Disulfide, Moly or MoS_2.**
4. **It is not black.**
5. **MoS_2 has more than twice the density of BN (5 g/cc vs. 2.2 g/cc). This means that if the particle morphology (size and shape) is similar, twice as much MoS_2 will be needed to provide similar coverage to a surface.**
6. **BN is also more resistant to oxidation and stabile in air above 600C whereas MoS_2 begins to oxidize near 400C.**
7. **It is more economical.**
8. **It is readily available.**

Solid Lubricant Comparison - Graphite - MoS_2 - PTFE – Boron Nitride

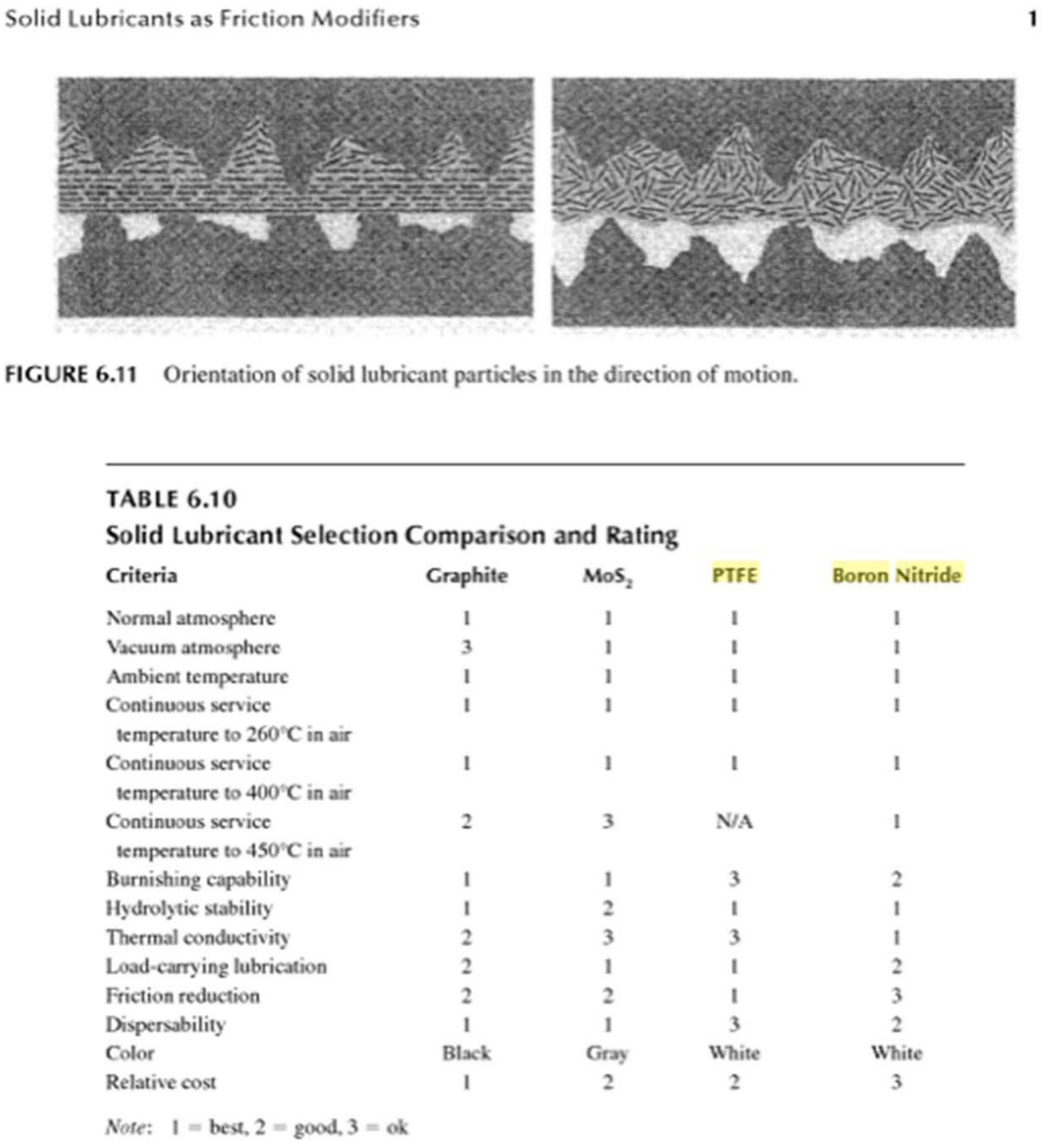


FIGURE 6.11 Orientation of solid lubricant particles in the direction of motion.

TABLE 6.10

Solid Lubricant Selection Comparison and Rating

Criteria	Graphite	MoS_2	PTFE	Boron Nitride
Normal atmosphere	1	1	1	1
Vacuum atmosphere	3	1	1	1
Ambient temperature	1	1	1	1
Continuous service temperature to 260°C in air	1	1	1	1
Continuous service temperature to 400°C in air	1	1	1	1
Continuous service temperature to 450°C in air	2	3	N/A	1
Burnishing capability	1	1	3	2
Hydrolytic stability	1	2	1	1
Thermal conductivity	2	3	3	1
Load-carrying lubrication	2	1	1	2
Friction reduction	2	2	1	3
Dispersability	1	1	3	2
Color	Black	Gray	White	White
Relative cost	1	2	2	3

Note: 1 = best, 2 = good, 3 = ok

FluoroCeramic Cerflon® versus Emralon® 334

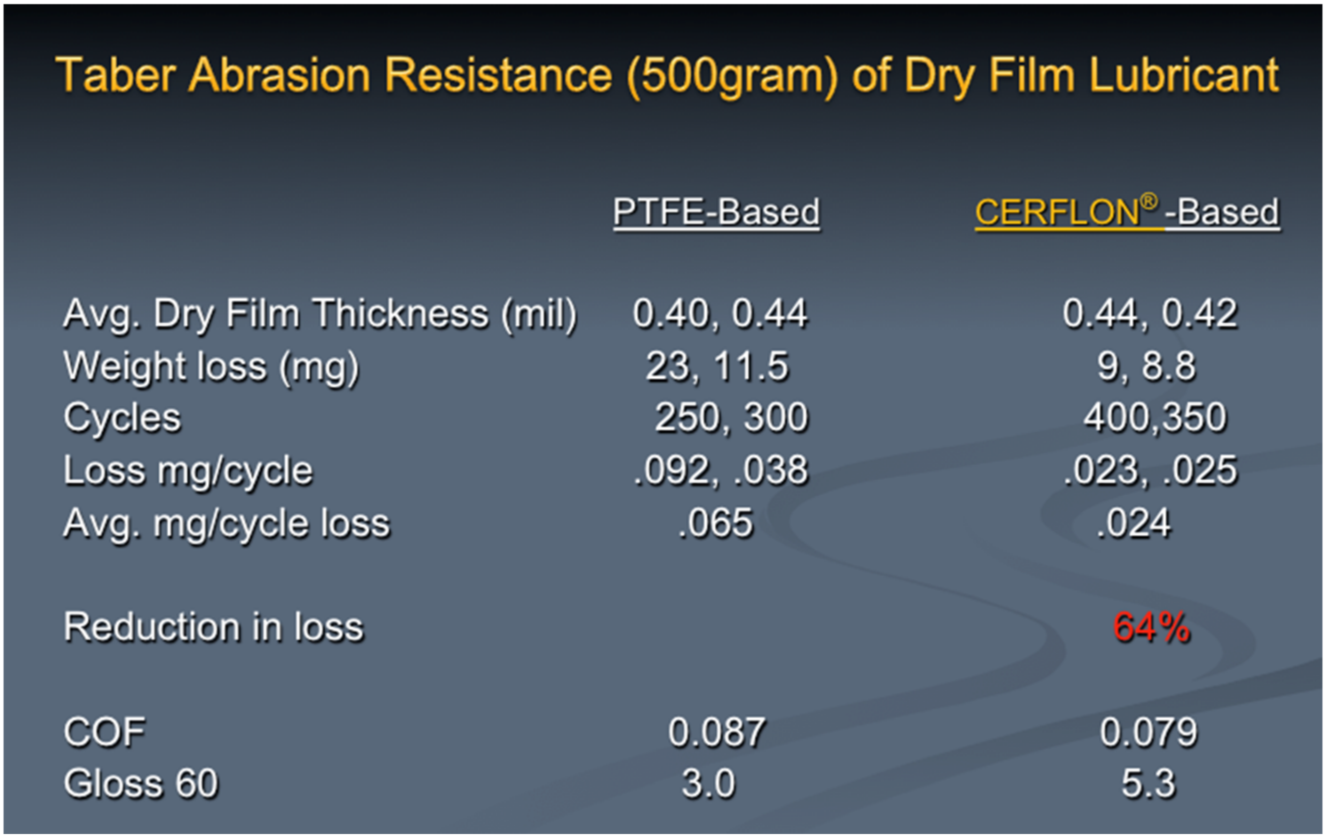
Emralon® 334 is one of a series of Henkel resin bonded lubricant coatings designed to provide dry film lubrication and release properties in a variety of industrial and consumer applications.

Emralon® 334 consists of a fluoropolymer lubricant dispersed in an organic resin and solvent system. Its superior physical properties, 500°F (260°C) service temperature capabilities, and excellent salt spray resistance make it particularly suitable for the lubricant coating of automotive components. This coating's low coefficients of static and dynamic friction offer significant advantages for carburetor parts; where elimination of stick-slip motion is a major factor in achieving good fuel economy.

The COF of Emralon® 334 was 0.087. The Emralon® 334 had a "Gloss 60" of 3.0 on a 10 scale.

T-013 Cerflon®

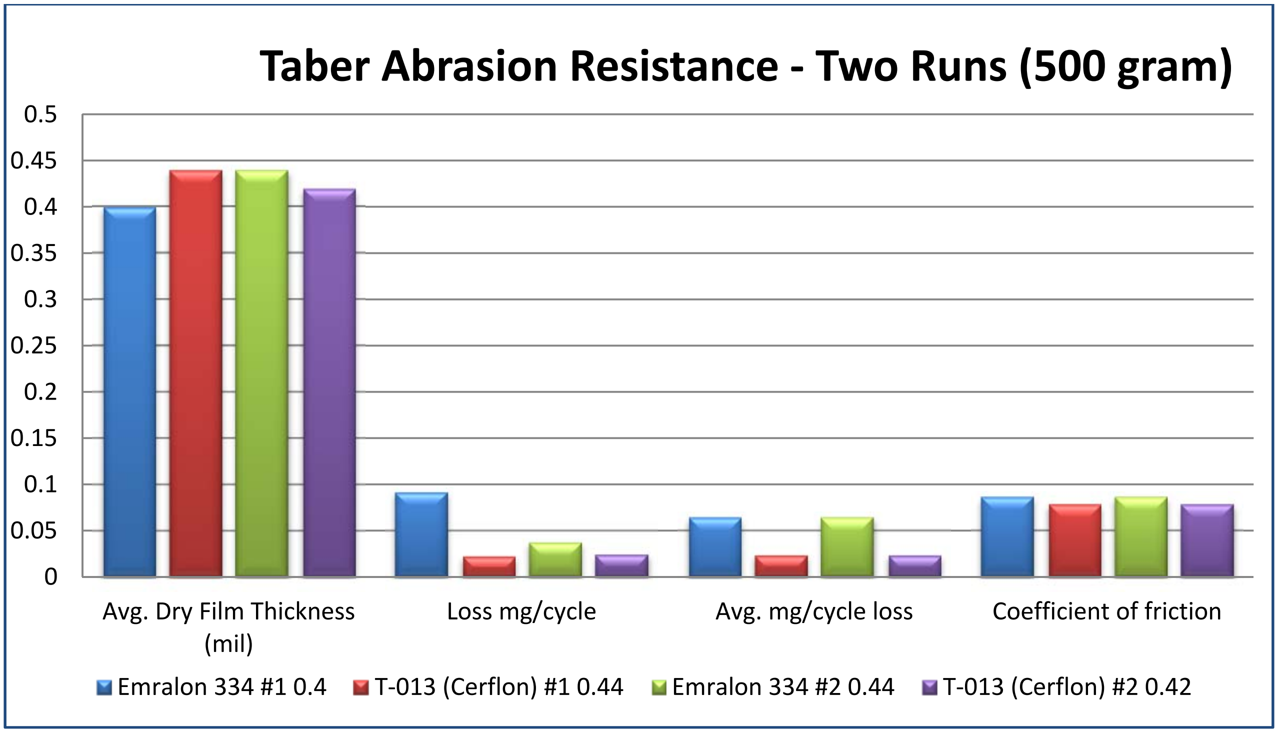
T-013 is a laboratory sample of Emralon® 334 with the addition of Boron Nitride as the only difference. COF of Cerflon is 0.079. The improved COF is probably due to the gloss being higher. Cerflon® had a "Gloss 60" of 5.3 on a 10 scale.



Taber Abrasion Resistance (500gram) of Dry Film Lubricant

	PTFE-Based	CERFLON®-Based
Avg. Dry Film Thickness (mil)	0.40, 0.44	0.44, 0.42
Weight loss (mg)	23, 11.5	9, 8.8
Cycles	250, 300	400,350
Loss mg/cycle	.092, .038	.023, .025
Avg. mg/cycle loss	.065	.024
Reduction in loss		64%
COF	0.087	0.079
Gloss 60	3.0	5.3

FluoroCeramic Cerflon® versus Emralon® 334 Chart



FluoroCeramic Cerflon® in Dry Film coating

The following set of data achieved improved abrasion resistance by increasing Boron Nitride levels and lowering Cerflon® levels. When 100% PTFE was tested, the abrasion resistance was well below 4000 cycles. It also measured dynamic coefficient of friction and found that with no Cerflon® the COF was approximately 1.0 but with the Cerflon® the COF dropped to 0.8 and with just Boron Nitride it was about 0.85. At that time, (2002), the biggest concern was the cost of the Boron Nitride (>$40) and its impact on the coating cost.

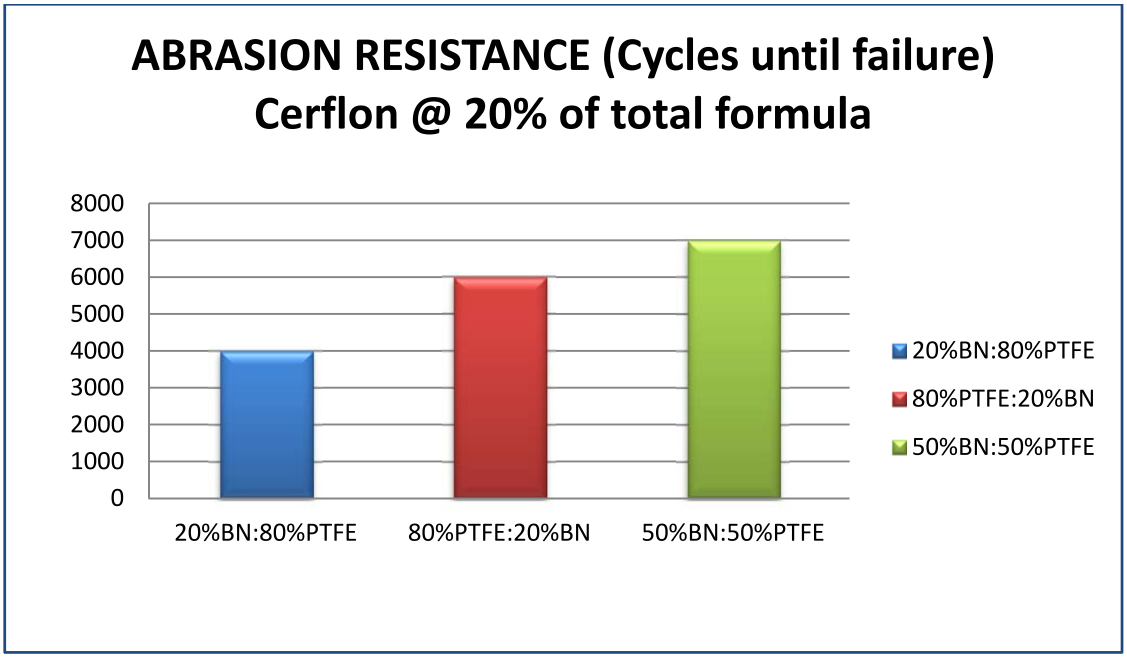
PTFE ABRASION RESISTANCE (cycles until failure)

100% PTFE	**<4000**

Cerflon® ABRASION RESISTANCE (cycles until failure)

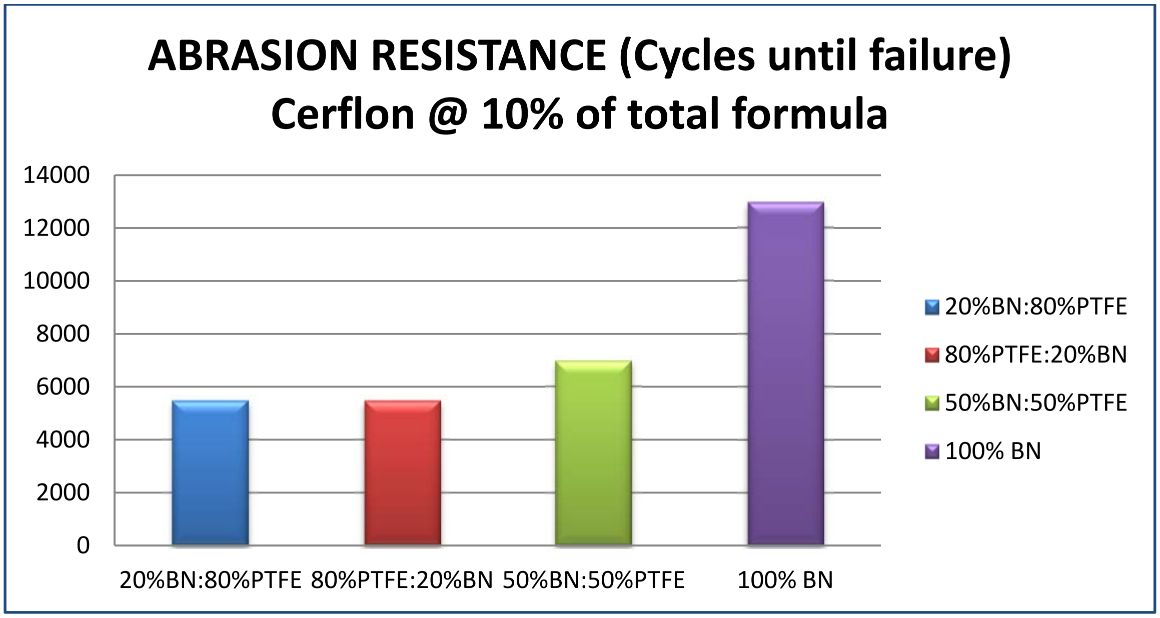
Cerflon® evaluated at 20% of the total formula

20% BN: 80% PTFE	**4,000**
80% BN: 20% PTFE	**6,000**
50% BN: 50% PTFE	**7,000**



Cerflon® was then evaluated at 10% of the total formula

20% BN: 80% PTFE	**5,500**
80% BN: 20% PTFE	**5,500**
50% BN: 50% PTFE	**7,000**
100% BN	**13,000**



FluoroCeramic Cerflon® - St-Gobain 2001 Falex Testing

SAINT-GOBAIN
ADVANCED CERAMICS

Mr. Howard Leendertsen
Chairman CEO
Ceramic Reinforced Technology
The Highlands
Seattle, WA 98177

December 10, 2001

Dear Mr. Leendertsen:

Attached is the data obtained by Saint-Gobain, Advanced Ceramics Corp. from the Falex Corporation regarding three lubricant samples containing boron nitride. The sample identification is as follows:

AS1353 = Acheson SLA-1710 (BN in mineral oil
AS1354 = Acheson SLA-2010 (BN/PTFE in mineral oil
AS1355 = Acheson SLA-1720 (BN in isopropanol

If you have any questions regarding the data please do not hesitate to contact me.

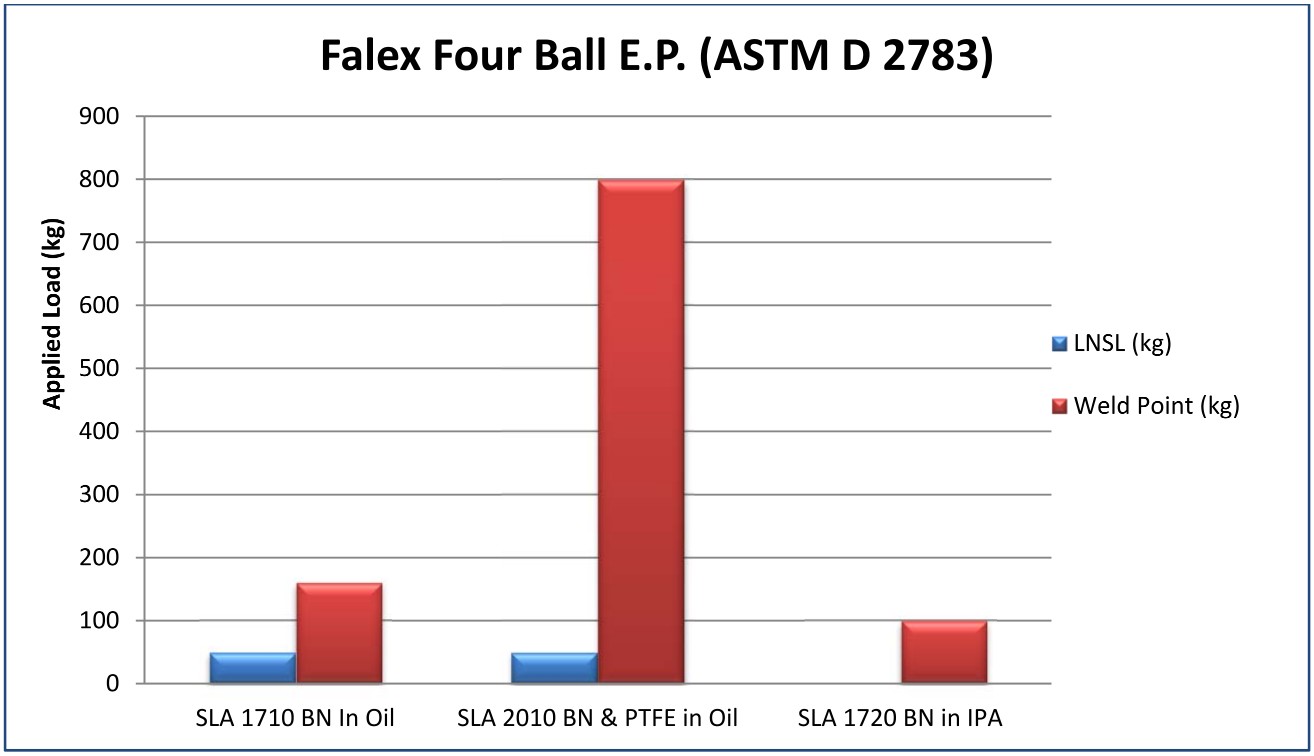
Sincerely,

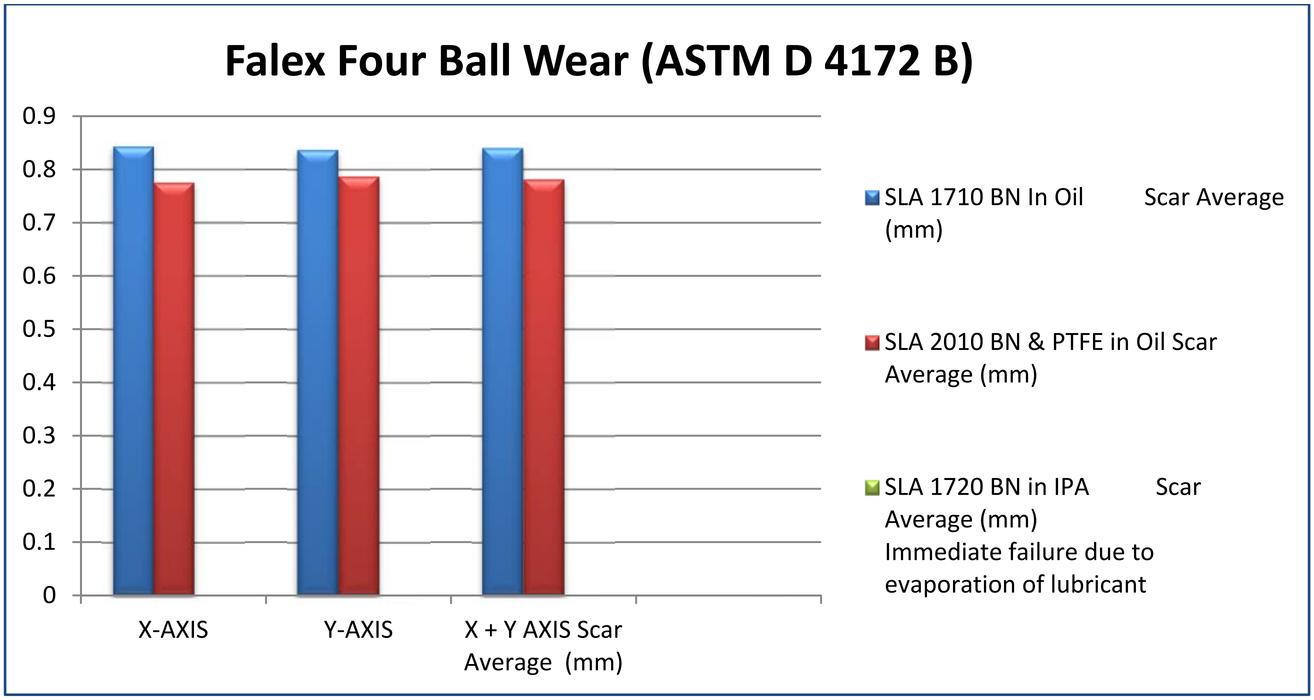
Gene Pruss

Eugene A. Pruss, Ph.D.
Senior R & D Engineer
Saint-Gobain, Advanced Ceramics Boron Nitride
168 Creekside Drive
Amherst, NY 14228
Telephone: 716-691-2049 Facsimile:
716-691-2080 eugene.a.pruss@saint-gobain.com

Saint Cobctin Advtt11ccd Cl-r.1m1cs Corporation Boron Nitride

Summary charts are below for the Falex data that follows:





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Website: www.falex.com

Falex Tribology N.V.
Heverlee, Belgium

COMPANY: ***Saint Gobain***
DATE: 10/09/01
TECHNICIAN: NJP/DJH

PAGE: 1
TEST#: 1804394
TEST DATE: 10/08/01

MACHINE: FALEX FOUR-BALL E.P.
METHOD: ASTM D 2783, Measurement of Extreme-Pressure Properties of liquid
MACHINE SERIAL#: 801800001357

TEST RESULTS:

FALEX AA ID#: AA-5017

SAMPLE ID: **AS1353**

LNSL (kg): 50

WELDPT (kg): 160

LWI: 23.65

TEST PARAMETERS:

SPEED (rpm): 1760 (± 60)
TEMPERATURE (˚C): Ambient
LOAD (kg): Per 10 seconds

SPECIMENS:

BALL MATERIAL: AISI-E52100
HARDNESS (HRc): 64-66
GRADE: 5

INTERPRETATION:

LAST NON-SEIZURE LOAD, kg (LNSL)
The last load at which the measured scar diameter is not more than 5% above the compensation line at the load. Beyond this point, incipient seizure occurs, indicating momentary breakdown of the lubri-cating film.

WELD POINT, kg (WELDPT)
The lowest applied load in kilograms at which the rotating ball welds to the three stationary balls, indi-cating the extreme-pressure level to the lubricants-force (or newtons) has been exceeded.

LOAD WEAR INDEX (LWI)
The load-carrying property of a lubricant. It is an index of the ability of a lubricant to minimize wear at applied loads.

FALEX AA ID#: AA-5017
SAMPLE ID: AS1353

TEST#: 1804394

TEST DATA:

APPLIED LOAD (kg)	SCAR MEASUREMENTS 1 (mm)	2 (mm)	3 (mm)	Avg (mm)	Cmpn (mm)	LDh FACTOR	CORRECTED LOAD FACTOR (kg)
50	0.346	0.348	0.362	0.344	0.360	16.10	46.8
	0.339	0.326	0.341				
63	1.734	1.886	1.757	1.791	0.390	21.86	12.2
	1.907	1.672	1.791				
80	2.019	1.898	2.065	2.101	0.420	30.08	14.3
	2.184	2.180	2.259				
100	2.192	2.483	2.188	2.459	0.460	40.50	16.5
	2.765	2.617	2.511				
126	2.858	2.714	2.760	2.880	0.500	55.20	19.2
	3.075	2.914	2.957				
160	**WELD**			0.000	0.540	75.80	

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Heverlee, Belgium

COMPANY:	***Saint Gobain***	PAGE:	3
DATE:	10/09/01	TEST#:	1804395
TECHNICIAN:	NJP/DJH		

MACHINE:	FALEX FOUR-BALL E.P.
METHOD:	ASTM D 2783, Measurement of Extreme-Pressure Properties of liquid
MACHINE SERIAL#:	801800001357

TEST RESULTS:

FALEXM ID#:	M# 5019
SAMPLE ID:	**AS1355**
LNSL (kg):	xxx
WELDPT (kg):	100
LWI:	7.9699

TEST PARAMETERS:

SPEED (rpm):	1760 (± 60)
TEMPERATURE (°C):	Ambient
LOAD (kg):	per 10 seconds

SPECIMENS:

BALL MATERIAL:	AISI-E52100
HARDNESS (HRc):	64-66
GRADE:	5

INTERPRETATION:

LAST NON-SEIZURE LOAD, kg (LNSL)
The last load at which the measured scar diameter is not more than 5% above the compensation line at the load. Beyond this point, incipient seizure occurs, indicating momentary breakdown of the lubricating film.

WELD POINT, kg (WELDPT)
The lowest applied load in kilograms at which the rotating ball welds to the three stationary balls, indicating the extreme-pressure level to the lubricants-force (or newtons) has been exceeded.

LOAD WEAR INDEX (LWI)
The load-carrying property of a lubricant. It is an index of the ability of a lubricant to minimize wear at applied loads.

FALEX AA ID#: AA# 5019
SAMPLE ID: **AS1355**

TEST#: 1804395

TEST DATA:

APPLIED LOAD (kg)	SCAR MEASUREMENTS 1 (mm)	2 (mm)	3 (mm)	Avg (mm)	Cmpn (mm)	LDh FACTOR	CORRECTED LOAD FACTOR (kg)
10	0.410	0.444	0.435	0.415	0.210	1.88	4.5
	0.397	0.410	0.396				
13	0.499	0.513	0.492	0.482	0.230	2.67	5.5
	0.463	0.467	0.456				
16	0.576	0.541	0.565	0.533	0.250	3.52	6.6
	0.522	0.484	0.511				
20	0.618	0.588	0.613	0.601	0.270	4.74	7.9
	0.598	0.584	0.605				
24	0.627	0.636	0.653	0.631	0.280	6.05	9.6
	0.624	0.608	0.640				
32	0.620	0.641	0.640	0.624	0.310	8.87	14.2
	0.604	0.631	0.609				
40	0.901	0.906	0.937	0.876	0.330	11.96	13.7
	0.817	0.835	0.857				
50	2.040	2.034	2.110	2.017	0.360	16.10	8.0
	1.963	2.007	1.948				
63	2.454	2.338	2.073	2.253	0.390	21.86	9.7
	2.238	2.343	2.070				
80	2.703	2.577	2.671	2.613	0.420	30.08	11.5
	2.697	2.503	2.527				
100	**WELD**			0.000	0.460	40.50	

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Heverlee, Belgium

COMPANY: ***Saint Gobain***
DATE: 10/10/01
TECHNICIAN: NJP/DJH

PAGE: 5
TEST#: 1804396
TEST DATE: 10/08/01

MACHINE: FALEX FOUR-BALL E.P.
METHOD: ASTM D 2783, Measurement of Extreme-Pressure Properties of liquid
MACHINE SERIAL#: 801800001357

TEST RESULTS:

FALEX AA ID#:	AA-5018
SAMPLE ID:	**AS1354**
LNSL (kg):	50
WELDPT (kg):	800
LWI:	113.7

TEST PARAMETERS:

SPEED (rpm):	1760 (± 60)
TEMPERATURE (°C):	Ambient
LOAD (kg):	per 1Oseconds

SPECIMENS:

BALL MATERIAL:	AISI-E52100
HARDNESS (HRc):	64-66
GRADE:	5

INTERPRETATION:

LAST NON-SEIZURE LOAD, kg (LNSL)
The last load at which the measured scar diameter is not more than 5% above the compensation line at the load. Beyond this point, incipient seizure occurs, indicating momentary breakdown of the lubricating film.

WELD POINT, kg (WELDPT)
The lowest applied load in kilograms at which the rotating ball welds to the three stationary balls, indicating the extreme-pressure level to the lubricants-force (or newtons) has been exceeded.

LOAD WEAR INDEX (LWI)
The load-carrying property of a lubricant. It is an index of the ability of a lubricant to minimize wear at applied loads.

FALEX AA ID#:	AA-5018	PAGE:	6
SAMPLE ID:	**AS1354**	TEST#:	1804396

TEST DATA:

APPLIED LOAD (kg)	SCAR MEASUREMENTS 1 (mm)	2 (mm)	3 (mm)	Avg (mm)	Cmpn (mm)	LDh FACTOR	CORRECTED LOAD FACTOR (kg)
50	0.358 0.339	0.332 0.359	0.342 0.359	0.348	0.360	16.10	46.2
63	0.687 0.632	0.643 0.589	0.666 0.679	0.649	0.390	21.86	33.7
80	0.808 0.736	0.738 0.670	0.832 0.728	0.752	0.420	30.08	40.0
100	0.939 0.742	0.901 0.848	0.868 0.809	0.851	0.460	40.50	47.6
126	1.152 1.013	1.180 1.070	1.162 1.059	1.106	0.500	55.20	49.9
160	1.149 1.228	1.267 1.195	1.271 1.151	1.210	0.540	75.80	62.6
200	1.358 1.326	1.315 1.257	1.320 1.275	1.309	0.590	102.20	78.1
250	1.436 1.330	1.396 1.334	1.411 1.331	1.373		137.50	100.1
315	1.437 1.390	1.513 1.449	1.589 1.447	1.471		187.10	127.2
400	1.432 1.459	1.542 1.540	1.446 1.451	1.478		258.00	174.5
500	1.668 1.443	1.641 1.656	1.801 1.576	1.631		347.00	212.8
620	1.921 1.925	1.840 1.847	1.913 1.932	1.896		462.00	243.6
800	**WELD**			0.000		649.00	

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COMPANY:	**Saint Gobain**	DATE:	10/10/01
SAMPLE ID:	**AS1353**	PAGE:	7
FALEXAA#:	AA-5017	TEST#:	1905635
TECHNICIAN:	DJH	TEST DATE:	10/5/01

MACHINE: FALEX FOUR BALL WEAR
METHOD: ASTM D 4172 B
MACHINE SERIAL#: 801900002616

TEST PARAMETERS:

SPEED (rpm):	1200 (± 60)	BALL MATERIAL:	AISI-E52100
TEMPERATURE (°C):	75 (± 1.7)	HARDNESS (HRc):	64-66
LOAD (kg):	40 (± 0.2)	GRADE:	5
DURATION (min):	60 (± 1.0)	ANSI SPEC B:	3.12

WEAR DATA:

SCAR MEASUREMENTS (mm):

	BALL 1	BALL2	BALL 3
X-AXIS:	0.831	0.857	0.843
Y-AXIS:	0.826	0.850	0.838
AVG SCAR		**0.841**	

INTERPRETATION:

AVERAGE SCAR DIAMETER, mm (AVE SCAR)
The average size of the scar diameters worn on the three lower clamped balls, used for comparison of lubricants.

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COMPANY:	**Saint Gobain**	DATE:	10/10/01
SAMPLE ID:	**AS1354**	PAGE:	8
FALEXAA#:	AA-5018	TEST#:	1905636
TECHNICIAN:	DJH	TEST DATE:	10/5/01

MACHINE: FALEX FOUR BALL WEAR
METHOD: ASTM D 4172 B
MACHINE SERIAL#: 801900002616

TEST PARAMETERS:

SPEED (rpm):	1200 (± 60)	BALL MATERIAL:	AISI-E52100
TEMPERATURE (°C):	75 (± 1.7)	HARDNESS (HRc):	64-66
LOAD (kg):	40 (± 0.2)	GRADE:	5
DURATION (min):	60 (± 1.0)	ANSI SPEC B:	3.12

WEAR DATA: SCAR MEASUREMENTS (mm):

	BALL 1	BALL2	BALL 3
X-AXIS:	0.763	0.782	0.782
Y-AXIS:	0.787	0.794	0.782
AVG SCAR		**0.782**	

INTERPRETATION:

AVERAGE SCAR DIAMETER, mm (AVE SCAR)
The average size of the scar diameters worn on the three lower clamped balls, used for comparison of lubricants.

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Heverlee, Belgium

COMPANY:	**Saint Gobain**	DATE:	10/10/01
SAMPLE ID:	**AS1355**	PAGE:	9
FALEXAA#:	AA-5019	TEST#:	1905637
TECHNICIAN:	DJH	TEST DATE:	10/5/01

MACHINE: FALEX FOUR BALL WEAR
METHOD: ASTM D 4172 8
MACHINE SERIAL#: 801900002616

TEST PARAMETERS:

SPEED (rpm):	1200 (± 60)	BALL MATERIAL:	AISI-E52100
TEMPERATURE (°C):	75 (± 1.7)	HARDNESS (HRc):	64-66
LOAD (kg):	40 (± 0.2)	GRADE:	5
DURATION (min):	60 (± 1.0)	ANSI SPEC 8:	3.12

WEAR DATA: SCAR MEASUREMENTS (mm):

	BALL 1	BALL 2	BALL 3
X-AXIS:			
Y-AXIS:			

AVG SCAR

COMMENTS:
This test was an immediate failure due to the lubricant nearly completely evaporating prior to the test start up.

INTERPRETATION:

AVERAGE SCAR DIAMETER, mm (AVE SCAR)
The average size of the scar diameters worn on the three lower clamped balls, used for comparison of lubricants.

FluoroCeramic Cerflon® Data

Testing conducted at Southwest Research Institute (SWRI)

Fuels and Lubricants Research (Tribology)

The resistance to abrasion of four fluids was investigated using the Ball on Cylinder Lubricity Evaluator (BOCLE) (ASTM D 5001). The ball on cylinder lubricity evaluator assesses the wear aspects of the boundary lubrication properties of a fluid. The fluid under test is placed in a test reservoir in which atmospheric air is maintained at 10% relative humidity. A non-rotating steel ball is held in a vertically mounted chuck and forced against an axially mounted steel ring with an applied load. The test cylinder is rotated at a fixed speed while being partially immersed in the fluid reservoir. This maintains the cylinder in a wet condition and continuously transports the test fluid to the ball/cylinder interface. The wear scar generated on the test ball is a measure of the fluid lubricating quality.

The first fluid tested was 100-wt% of a 150 solvent refined paraffinic base oil. The second fluid tested was a combination of 98.0-wt% of a 150 solvent refined paraffinic base oil and 2.0 wt% of 10 wt% Boron Nitride/150 solvent refined paraffinic base oil dispersion. The Boron Nitride dispersion used was JB33018 (Acheson Colloids). The resulting second fluid included 0.2 wt% Boron Nitride and 98.8-wt% 150 solvent refined paraffinic base oil. The third fluid was a stabilized dispersion of PTFE (SLA1612, Acheson Colloids). The stabilized PTFE dispersion included 95.0-wt% of 150 solvent refined paraffinic base oil and 5.0-wt% of a 20-wt% of PTFE in 150 solvent refined paraffinic base oil. The resulting third fluid included 1.0 wt% PTFE and 99-wt% 150 solvent refined paraffinic base oil. The fourth fluid consisted of a mixture including 93.0 wt% of a 150 solvent refined paraffinic base oil, 5.0 wt% of the PTFE dispersion and 2.0 wt% Boron Nitride dispersion to give a fourth fluid including 1.0 wt% of PTFE, 0.2% wt% Boron Nitride and 98.8% wt% 150 solvent refined paraffinic base oil carrier.

The results of the BOCLE tests follow:

	Composition Number:	**Scuff Depth (mm)**	
1.	150 solvent refined paraffinic base oil	**100%**	**0.7**
2.	150 solvent refined paraffinic base oil	**98%**	**0.635**
	JB33018 Boron Nitride	**2%**	
	150 solvent refined paraffinic base oil	**95%**	**0.585**
	PTFE (SLA1612)	**5%**	
3.	150 solvent refined paraffinic base oil	**93%**	**0.53**
	PTFE (SLA1612)	**5%**	
	JB33018 Boron Nitride	**2%**	

DuPont Dow Elastomers Cerflon® Tests

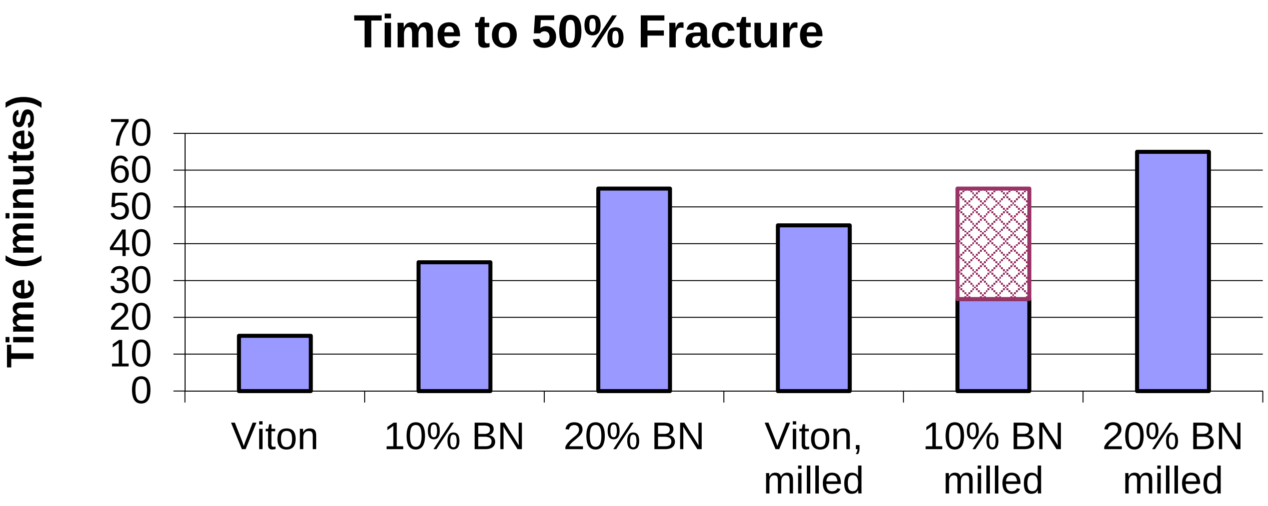
The following tests were conducted through the cooperation of DuPont Dow Elastomers and CRT. Our objective was to evaluate Viton® process additives with Cerflon® versus just PTFE. We also wanted to demonstrate improvement in abrasion resistance as well as try to discover uniqueness in processing or properties when Cerflon® was added to the Viton® additive.

Given the results, possible market segments and market niches were discussed with DDE as to whether an opportunity exists to make subtle product improvements and be rewarded. The segments we focused on were the TiO2 and anti-block white masterbatch segments and the possibility that Cerflon® additives would allow higher PTFE/Viton® loadings, which would improve extrusion performance. As a group, we felt if one or both of these segments showed improved performance or properties, the Cerflon® technology demonstrated commercial value.

Day one narrative:

Today we finished the first series of testing. The results prove one thing if nothing else. Too much data can ruin a perfectly good theory. I have plotted a summary of the results to date below. The time to achieve a melt fracture level of 50% is plotted by the solid blocks.

The first day's testing showed that adding more BN improved performance. The second day's testing showed the same thing until we ran the 10% BN and VITON milled sample. For the data to be perfect the missing segment shown in the hatched section should be there and for whatever reason it is not.

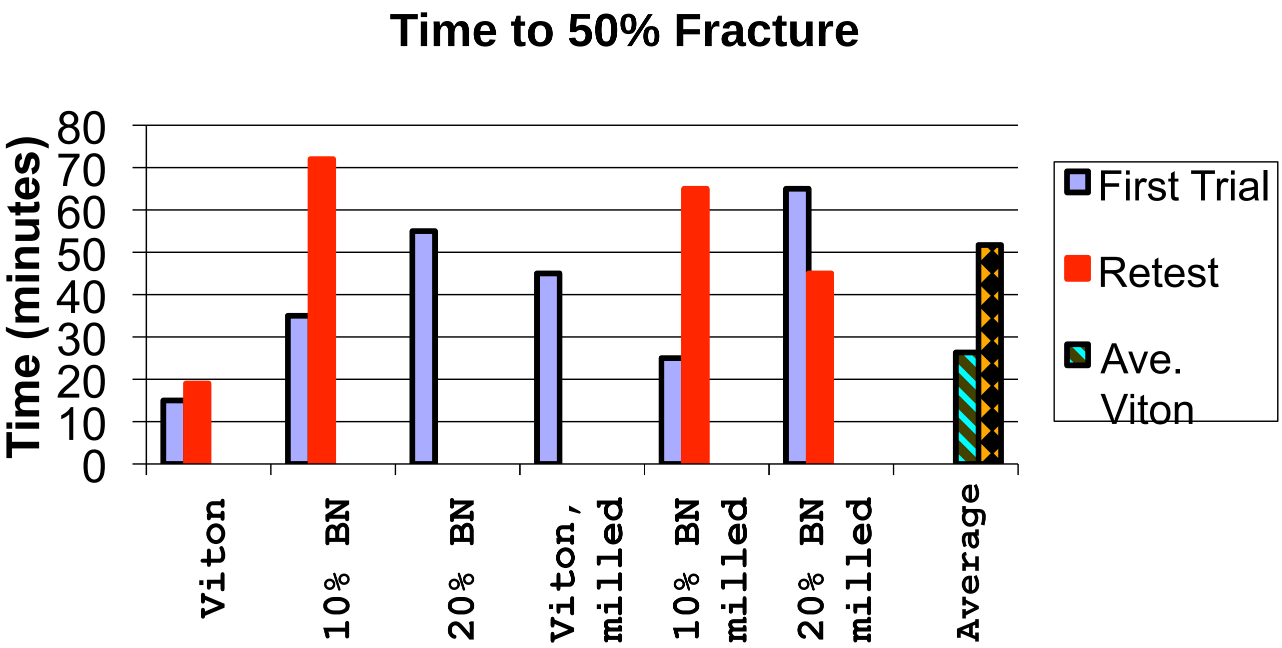


*It is also interesting to note that the milled VITON sample gave better results than the sample that was not milled.

We know from experience that heat history degrades fluoroelastomer performance in other types of tests. We will repeat some of these numbers to determine how good a test we are running. We will also try to understand what, if anything, this means. I am still encouraged. There is something happening here that is worth understanding.

Day two narrative:

We retested several of the solutions. The results to date are summarized in the attached chart. These results raise some obvious questions about the reproducibility of this particular test. However, it is worthwhile to note that the average time to fracture for coatings without BN is about half that of the coatings that do contain BN. There is a difference.



THE EFFECT OF COMBINING BORON NITRIDE AND FLUOROELASTOMER ON MELT FRACTURE IN EXTRUSION BLOW MOULDING*

Abstract

Boron Nitride (BN) is a new polymer processing aid, which not only eliminates surface melt fracture in the extrusion of molten polymers but it also postpones the critical shear rate for the onset of gross melt fracture to significantly higher values depending on resin type and additive concentration. In this work, the influence of BN powder as a polymer processing additive is first examined in the extrusion of blow molding of high-density polyethylene (HDPE) resins in order to evaluate its usefulness and performance in operations other than simple extrusion. The equipment used includes both a Battenfeld/Fisher 50mm-extrusion blow moulding machine and parallel-plate rheometer. Two types of HDPE, which are blended with boron nitride at various concentration levels, are tested accordingly. It is found that the degree of BN dispersion, characteristics of the HDPE resins, extrusion temperature and induction time play an important role in eliminating melt fracture. Finally, the influence of combining BN with fluoroelastomer as potentially better processing aids on the melt fracture is examined. It is found that this combination is a superior processing aid allowing extrusion blow molding at very high shear rates.

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[*] This paper will be submitted for the next ANTEC conference (ANTEC-2001), and to the Journal of Vinyl and Additive technology.

THE EFFECT OF COMBINING BORON NITRIDE AND FLUROELASTOMER ON MELT FRACTURE IN EXTRUSION BLOW MOULDING (DATA)

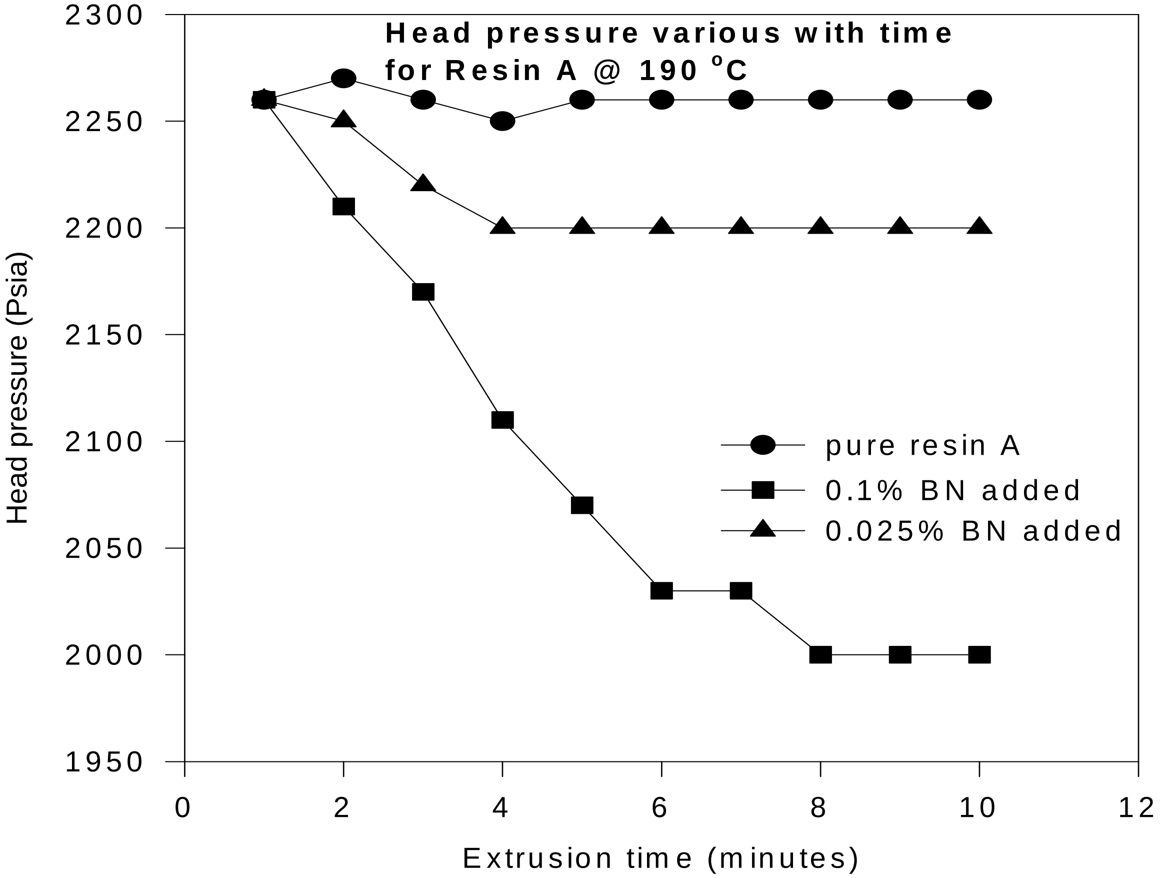

Figure 4: Transient extrusion runs showing the head pressure versus extrusion time for Resin A with and without BN at 190°C. Note that the final equilibrium pressure and the time to reach steady-state vary with BN levels.

Boron Nitride as a Processing Aid for the Extrusion of Polyolefins and Fluoropolymers

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The influence of a new processing additive (fine particles of boron nitride) on the processability of polyolefins and fluoropolymers in extrusion is studied. The equipment used includes an Instron capillary rheometer with two types of dies, namely capillary dies and special annular dies (Nokia Maillefer wire coating crosshead) attached to the rheometer, and an extruder. Two metallocene polyethylenes and several Teflon® fluoropolymers were tested using these two pieces of equipment. The additive had a significant effect on the extrudate appearance of polyethylene and fluoropolymer particularly in the crosshead dies. It was found to eliminate surface melt fracture and to postpone the critical shear rate for the onset of gross melt fracture to significantly higher values depending on resin type, temperature, and additive concentration (typically 0.005% to 0.5%). To explain the possible mechanism for the effect of the additive on the processability of the resins, rheological measurements using both parallel-plate and sliding-plate rheometers were carried out. The rheology of the resins did not seem to change significantly with the addition of boron nitride except for the low-shear-rate (low-frequency) range, where the behavior of the filled resin was found to be similar to that of a crosslinked polymer or a phase-separated entangled blend. Practical wire coating and tubing extrusion studies for these resins were also carried out.

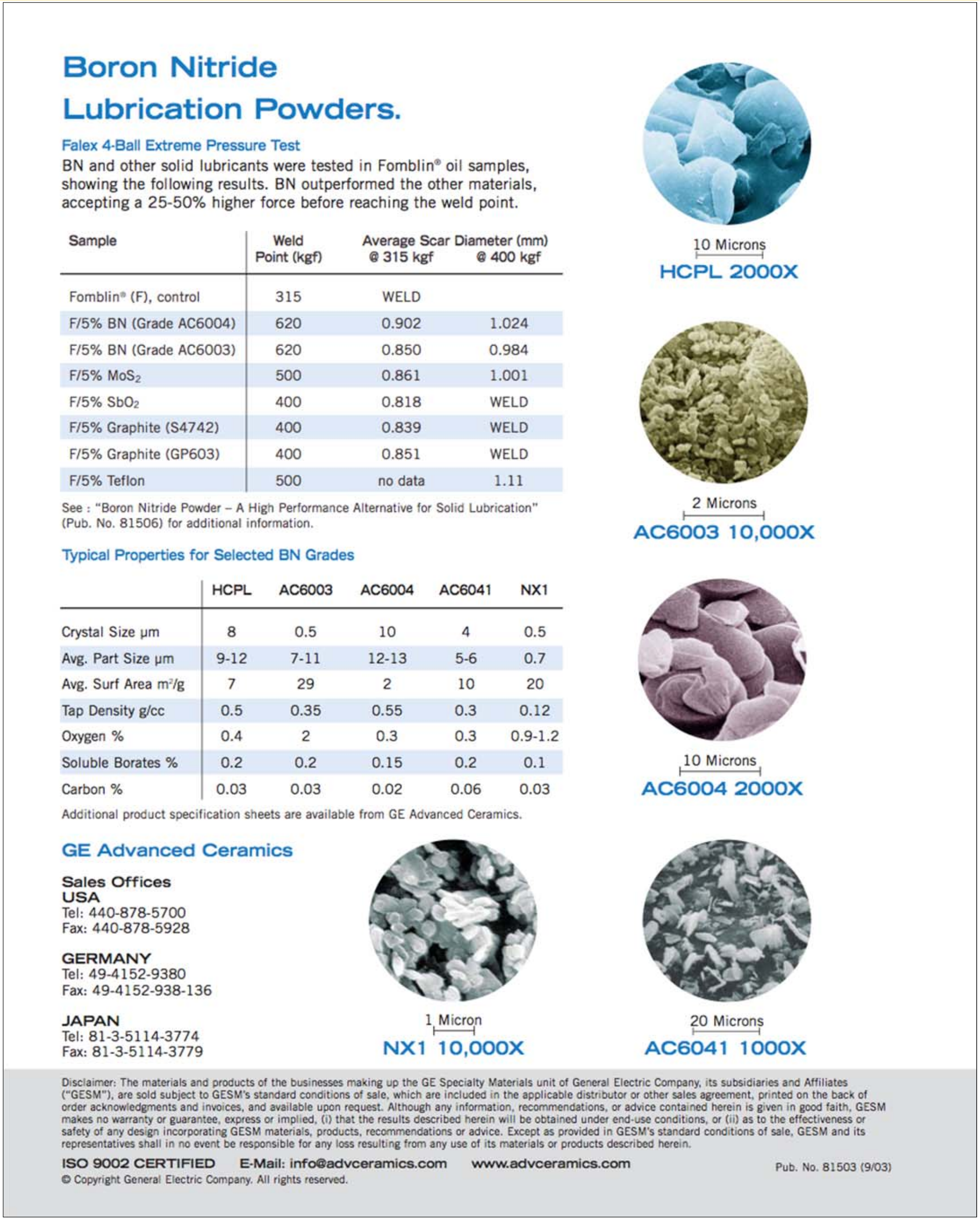
INTRODUCTION

The rate of production of many polymer processing operations including fiber spinning, film blowing, extrusion, and various coating flows, is limited by the onset of flow instabilities (1–3). In particular, in extrusion processes, when the throughput exceeds a critical value, small amplitude periodic distortions appear on the surface of extrudates (surface melt fracture or sharkskin). At higher throughput rates these take a more severe form of larger irregular distortions (gross melt fracture) (4). The surface melt fracture is believed to originate in the land of the die next to the die exit (5–8), while gross melt fracture to be initiated at the die entry (4, 9).

To increase the rate of production by eliminating or postponing the melt fracture phenomena to higher shear rates, processing additives/aids must be used. These are mainly fluoropolymers that are widely used mainly in the processing of polyolefins (HDPE, LLDPE). They are added to the base polymer at low concentrations (approximately 0.1%), and they essentially act as die lubricants, modifying the properties of the polymer-wall interface (increasing the slip of molten polymers). As a result of this lubrication effect, the onset of instabilities is postponed to much higher output rates, while the power requirement for extrusion is significantly reduced. It should be emphasized that these additives can eliminate only surface (sharkskin) and stick-slip (oscillating or cyclic) melt fracture. To the best of the authors' knowledge, they do not appear to have an effect on the extrudate appearance in the gross melt fracture region.

FluoroCeramic CERFLON® Data - GE Advanced Ceramics

5% BN treat rate accepted 25-50% higher force before reaching the weld point



Boron Nitride Lubrication Powders.

Falex 4-Ball Extreme Pressure Test

BN and other solid lubricants were tested in Fomblin® oil samples, showing the following results. BN outperformed the other materials, accepting a 25-50% higher force before reaching the weld point.

Sample	Weld Point (kgf)	Average Scar Diameter (mm) @ 315 kgf	Average Scar Diameter (mm) @ 400 kgf
Fomblin® (F), control	315	WELD	
F/5% BN (Grade AC6004)	620	0.902	1.024
F/5% BN (Grade AC6003)	620	0.850	0.984
F/5% MoS_2	500	0.861	1.001
F/5% SbO_2	400	0.818	WELD
F/5% Graphite (S4742)	400	0.839	WELD
F/5% Graphite (GP603)	400	0.851	WELD
F/5% Teflon	500	no data	1.11

See : "Boron Nitride Powder – A High Performance Alternative for Solid Lubrication" (Pub. No. 81506) for additional information.

Typical Properties for Selected BN Grades

	HCPL	AC6003	AC6004	AC6041	NX1
Crystal Size µm	8	0.5	10	4	0.5
Avg. Part Size µm	9-12	7-11	12-13	5-6	0.7
Avg. Surf Area m^2/g	7	29	2	10	20
Tap Density g/cc	0.5	0.35	0.55	0.3	0.12
Oxygen %	0.4	2	0.3	0.3	0.9-1.2
Soluble Borates %	0.2	0.2	0.15	0.2	0.1
Carbon %	0.03	0.03	0.02	0.06	0.03

Additional product specification sheets are available from GE Advanced Ceramics.

10 Microns
HCPL 2000X

2 Microns
AC6003 10,000X

10 Microns
AC6004 2000X

1 Micron
NX1 10,000X

20 Microns
AC6041 1000X

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Boron Nitride powder additives for high performance lubrication

Boron Nitride (BN) ceramic powders are exceptionally lubricious additives for high performance solid and viscous lubricants. Applications for BN as a lubricant include:

- Fillers for solid polymer shapes used as sliding components, etc.
- Dispersions in oils, greases, and aqueous solutions
- Water and aerosol-based coatings for metalworking, glassforming, and other processing applications
- Metal matrix and ceramic-metal composites for abradable seal and other demanding uses
- Electrodeposited, plasma, and thermal spray coatings

BN brings the following performance benefits to lubricating systems:

- Low coefficient of friction – measured as low as 0.12 in graphite-like BN grades*
- High temperature and chemical stability lets BN perform where other lubricants fail
- High thermal conductivity improves heat dissipation and reduces localized overheating; this is combined with excellent dielectric properties
- A high load-carrying capacity makes BN useful under extreme pressure or vacuum
- Low thermal expansion minimizes internal force generation in high temperature applications
- BN's white color gives lubricating systems a cleaner appearance vs. graphite or molybdenum disulfide

Engineered to your specifications

GE Advanced Ceramics (formerly Advanced Ceramics Corp. and now part of GE Quartz) offers over 75 grades to suit your application, with a broad range of densities, surface areas, and particle sizes. GE also has the expertise to tailor the properties of BN powder to meet a broad range of customer requirements.

GE Advanced Ceramics is a leading producer of BN and serves the global market with facilities in the U.S., Europe and Asia.

GE Advanced Ceramics

* Falex Corporation COF Test