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### Introduction

Flange Adhesion associated with many gasket compositions has been a problem for gasket users for years. Apart from the separation of flanges, the problem becomes the task of adequately removing the adhering gasket material in a safe and timely manner and without damaging the costly flanges. Solvent-based gasket removers may be effective but can present health and safety Issues. In many plants the use of wire brushing or wire wheels is a common practice, but if not done properly can lead to damaged process equipment or system contamination.

In 2007, after listening to the "Voice of the Customer", Garlock Sealing Technologies embarked on an extensive effort to improve on both the formulation and the application method of its anti-stick coating. The goal was to introduce the improved Anti-Stick as part of Garlock's investment into innovative technologies that allowed for a transition away from Toluene and towards a Patent Pending Process utilizing a safer, more environmentally friendly solvent. This process has since been recognized nationally with a Clean Air Excellence Award by the US EPA.

The intent of this paper is to:

- Review the fundamental causes of gasket adhesion to flanges.
- Discuss the characteristics of anti-stick coatings and other alternatives.
- Provide data which illustrates why the Garlock Flange Free<sup>™</sup> coating differentiates itself from other alternatives in the marketplace.

#### Section 2 Garlock Flange Free<sup>™</sup>

**Coating** : A technical explanation of gasket to flange adhesion

### What Causes Flange Adhesion

The mechanism of adhesion has been investigated for years; several theories have been proposed in an attempt to provide an explanation for adhesion phenomena. However, no single theory explains adhesion in a general, comprehensive way. The bonding of an adhesive to an object or a surface is the result of various mechanical, physical, and chemical forces that overlap and influence one another. [1,2]

The prevailing theories on adhesion include:

### Adsorption

The adsorption theory is based on the assumption that the adhesive "wets" the surface of the contact or sealing surfaces, meaning that the adhesive when applied to the adherent spreads spontaneously. For this to occur the surface tension of the adhesive must be lower than the surface free energy of the adherent. Adhesive strength arises as a result of intimate contact between the adhesive and adherent through secondary intermolecular forces at the interface, collectively known as Van der Waals forces. These secondary forces include dipole-dipole forces, dispersion forces and hydrogen bonding.

#### Mechanical Interlocking

The mechanical interlocking theory is based on the fact that at the microscopic level all surfaces are very irregular, consisting of crevices, cracks and pores. A bond arises when the adhesive penetrates or surrounds these features and hardens.

#### Chemisorption

The chemisorption theory is also based on the adhesive "wetting" the adherent. Adhesive strength arises as a result of the formation of ionic, covalent or metallic chemical bonds. Such bonding produces much stronger bonds than those created by Van der Waals forces.

#### Electrostatic

The electrostatic theory is based on the formation of an electrical double layer at the adhesive – adherent interface. Adhesive strength is attributed to the transfer of electrons across the interface, creating positive and negative charges that attract one another.

### Diffusion

The diffusion theory is based on the interpenetration of polymer chains at the interface between polymers. Adhesive strength is attributed to molecular interlocking.

Since most flanges are composed of metal alloys, we can ignore diffusion as being a factor.

# **GRAPHITE GASKETS**

Flexible graphite gaskets consist of interlocked "worms" of exfoliated flake graphite and contain no organic binders, thus the interface between gasket and flange consists of two solids. The mechanisms of adsorption or electrostatics are usually involved for adhesion to occur. To achieve high adhesion forces through adsorption mechanisms, extremely close distances between two solids are required - this condition is not often met with typical flange surface finishes and stresses, therefore lower adhesion factors are present. Although empirically we find that minimal flange adhesion occurs with flexible graphite gaskets, also known as

GRAPH-LOCK<sup>™</sup> (See Figure 1.), the low cohesive strength of the graphite flake allows them to separate at these low levels. For this reason, small amounts of graphite will consistently remain on the pipe flange after gasket removal as the graphite flakes separate from one another. Prior to the installation of a new gasket it is imperative that all the graphite material is removed. While the graphite is fairly easy to remove, this extra cleaning does add labor costs, and is of significant concern to some customers relative to FME (Foreign Material Exclusion) issues.



Figure 1: GRAPH-LOCK® Gasketing

## PTFE GASKETS

Minimal flange adhesion occurs with PTFE based gasketing compositions such as GY-LON<sup>®</sup>. (See Figure 2.) The well known nonstick properties of Teflon, the DuPont trade name for PTFE, are due to its low surface energy [3]. PTFE is also often used as an anti-stick material to coat the surface of other gasketing compositions.



### COMPRESSED FIBER GASKETS

By and large, the problem of flange adhesion is associated with compressed fiber gasketing. While there are numerous compositions in the marketplace, they all contain an organic rubber binder. By design, the extent of cure or degree of cross linking of these binders is typically lower than that of a homogeneous rubber gasket. The softer, less cross-linked rubber allows the gasket to conform to the flange and thus improves sealability.

The problem here in terms of flange adhesion is, under heat and pressure, the binder flows out and wets the flange allowing adhesion mechanisms such as adsorption, chemisorption and mechanical interlocking to come into play. These forces can be very high and result in the problems associated with flange adhesion. Figure 3 shows typical sticking with uncoated or poorly coated compressed fiber gaskets.



Figure 3: Competitive carbon fiber gasket with rubber binder after 450°F adhesion testing.

#### Preventing gasket/flange adhesion

The basic strategy is to coat the gasket with a low surface tension semi solid or liquid (e.g. PTFE, silicone, a platy solid, or an antiseize compound) to prevent the binder from wetting out on the flange. The objective in the development of Garlock's Flange Free<sup>™</sup> coating was to create a new platy coating system with improved anti-stick characteristics, but without the unwanted "side effects" relative to sealability, crushing, chemical resistance, process contamination, corrosion, and handling found in the alternatives.

Anti-seize compounds vary in composition but typically consist of metal particles in petroleum-based oil with other additives. The general consensus among gasket manufacturers is that they are not recommended for three reasons [4,5]: 1) Under heat and pressure, the metals in the compound can adhere to the flange surface causing distortion of the flange facing and/or filling of the serrations. When this condition has been allowed to progress, there is no amount of additional torque that will allow the gasket to seal. (See Figure 4.)



Figure 4: Flange serrations filled with anti-seize compound.

2) Coating gaskets with anti-seize compounds can cause various problems as the gasket is compressed. A lubricated gasket not only has a tendency to extrude and split, but also can be forced out of the flange by internal pressures and lack of friction. Here the friction created by the flange serrations plays a role. (More on this later in this paper)

3) The petroleum oil in anti-seize compounds can soften some gasketing compositions. This event describes the lack of chemical compatibility between the gasket composition and the anti-seize compound.

Although the use of silicone anti-stick agents can be effective, there are separate issues to be concerned with. One such issue would be that silicone can contaminate the fluid in the pipeline. When the pipe or vessel contains paint or chemicals for making photographic film, the silicone can cause a lack of adhesion of the paint or film surface. Figure 5 depicts a fisheye or pinhole defect where the painted surface has craters due to the presence of silicone. For that reason, the use of silicone is banned from many gasketing applications where this could be an issue. [18]



Figure 5: Fish eye defect in paint caused by silicone. [18]

PTFE based anti-stick agents can also be effective, however the issue is the lack of thermal stability. PTFE begins to decompose above 500°F, well below the maximum service temperature of most compressed fiber gasketing compositions. Upon decomposition, halogenated decomposition products can be formed. These byproducts can be hazardous and corrosive to the flange and piping system. Utilizing a solution without these hazards is paramount.

The most desirable strategy is the use of a platy inorganic material as a blocking agent to prevent the binder from wetting out on the sealing surface. Particles such as talc, mica, vermiculite and graphite are effective as anti-stick agents due to the cleavage of their layered crystal structure. These materials cleave to form thin sheets, which when milled, result in flake-like structures. Figure 6, Figure 7, and Figure 8 show SEM (Scanning Electron Microscope) images of graphite, talc, and mica. The flat plate-like structure allows the particles to form a laminar barrier structure on the surface of the gasket.



Figure 6: SEM Image of Flake Graphite Image provided courtesy of the McCrone Atlas of Microscopic Particles



Figure 7: SEM Image of Talc



Figure 8: SEM Image of Muscovite Mica Image provided courtesy of the McCrone Atlas of Microscopic Particles

Talc, mica, vermiculite and flake graphite are all natural materials, but not pure substances. Grades vary on their morphology, degree of purity and levels of undesirables [6,7,8,9].

Graphite of course has a fundamental issue relative to its color and its propensity to be "messy".

Graphite is also an electrical conductor and behaves like a noble metal in terms of galvanic corrosion [10]. In wet or humid environments, contact of graphite with aluminum can result in severe galvanic corrosion [11]. Graphite gaskets wetted by seawater can also cause rapid localized attack of most stainless steel alloys [12,13]. At elevated temperatures graphite can also carburize some stainless and nickel alloys causing them to be more susceptible to intergranular corrosion [14, 15, 16].

What we were looking for in an alternative anti-stick agent was a material with the desirable flake morphology without some of the undesirable characteristics.



Figure 9: SEM of Particles used in Garlock Flange Free<sup>™</sup> Coating

Unlike materials such as talc, mica, vermiculite and flake graphite which are mined, the platy particles used in the Garlock Flange Free<sup>™</sup> Coating are synthesized from refined materials under very highly controlled conditions, which results in a uniform high purity product. Figure 9 shows a SEM image of these particles.

These white platy particles are very compliant, such that they stack well, which aids in producing a continuous barrier. This also helps in terms of sealability of the gaskets.

Unlike graphite, the particles do not conduct electricity, therefore they do not contribute to galvanic corrosion.

As shown in Table 1, the particles contain extremely low levels of halogens and sulfur compounds. That is significant since those materials have the potential to create corrosion in some conditions.

Analyte	Concentration (ppm)
Chloride	< 5
Fluoride	< 0.5
Bromide	< 0.5
lodide	< 1
Sulfur	<2

Table 1: Garlock Flange Free<sup>™</sup> Coating, typical water leachable halogens & sulfur.

The particles are thermally stable, having an oxidation threshold of approximately 800°C, and have unusually high chemical stability [17]. The thermal and chemical stability of the particles contribute to the non-toxic and environmentally friendly characteristics of the material.

The Flange Free<sup>™</sup> coating components are not listed as carcinogens by the American Conference of Governmental and Industrial Hygienists (ACGIH), International Agency for Research on Cancer (IARC), Occupational Safety and Health Administration (OSHA), National Institute for Occupational Safety and Health (NIOSH) or National Toxicology Program (NTP). Relative to exposure limits the particles are classified as a Nuisance Dust.

The Flange Free<sup>™</sup> coating components are also not considered hazardous chemicals under Environmental Protection Agency (EPA) or Superfund Amendments and Reauthorization Act (SARA) guidelines and no regulations exist regarding their use, transport or disposal.

# Section 3 Garlock Flange Free<sup>™</sup>

Coating: The importance of even and adequate anti-stick distribution

# **Coating Quality**

We have discussed the merits of various strategies to prevent flange adhesion and the desirable properties of platy particles for this application. While this is a good starting point for an anti-stick coating, this is only part of what makes the Garlock Flange Free<sup>™</sup> Coating so successful.

Ultimately what is needed is a stable dispersion of these particles, without the aid of any organic binders, that uniformly wets out the surface of the gasket. Upon drying, it is imperative that the coating left behind does not readily wipe off. Garlock Engineering discovered through internal trials and evaluation of competitive anti-stick coatings, that the use of binders in the releasing agents leads to compromised adhesion values.



Figure 10: Garlock Style 9850 with previous anti-stick coating (4Q06)



Figure 11: Competitive compressed carbon fiber gasket with non-uniform anti-stick coating



Figure 12: Garlock Style 9900 with Flange Free™ Coating (Branded Side)



Figure 13: Garlock Style 9900 with Flange Free™ Coating (Non Branded Side)

Once a coating formulation has been optimized, it is obviously necessary to be able to consistently apply a uniform coating that is not too thin or too thick. Too thin of a coating can lead to inadequate blocking and subsequently an increase in flange adhesion. Too thick of a coating can lead to a reduction in sealability or crush resistance.

Prior to the introduction of the Flange Free<sup>™</sup> Coating, Garlock used a conventional naturally platy material (talc) for in its anti-stick coating. As illustrated in Figure 10, the coating was not entirely uniform. In areas that were coated, the platelets were deposited in "ridges" rather than being uniformly distributed. We found that to be similar to other anti-stick coatings on the market as seen in Figure 11.

Figure 12 and Figure 13 give a macroscopic view of Style 9900 with the new Garlock Flange Free<sup>™</sup> Coating to demonstrate the difference in appearance vs the original talc coating.

To prove that the dark areas on the competitive sample in Figure 11 were not coated with a thin layer of anti-stick platelets, a closer look was taken using SEM. SEM micrographs were taken in the locations as shown in a grid layout, to allow a representative picture to be taken over the surface of the coated gasket. (See Figure 14.)





The first series of SEM micrographs, Figure 15, show, on a microscopic level, the same competitive compressed carbon fiber gasket as Figure 11. The extremely rough surface is caused by non-uniform application of the platy anti-stick particles. The effect that these raised areas of platy particles have on sealability and adhesion will be discussed in greater detail in Section 5.

The next series of micrographs, Figure 16, illustrate Garlock's Flange Free<sup>™</sup> Coating. What is very apparent from the photos is the absence of the ridges of platy particles. The benefit of this more consistent coating on sealability and adhesion properties will also be discussed in Section 5.

# Section 4 Garlock Flange Free<sup>™</sup>

Coating: Adhesion testing protocol

ASTM Test Method F607 provides a means of determining the degree to which gasket materials (under compressive load) adhere to metal surfaces. The adhesion is expressed in terms of adhesive force per unit area of gasket surface. While adhesive force is important as an index in terms of ease of gasket removal, what also needs to be taken into consideration is the amount of residual gasket material remaining on the flange.

The test is typically conducted at 212°F (100°C) for 22 hours. The maximum recommended test temperature is 400°F (204°C). Since the adhesion force with Compressed Fiber Gasketing is due chiefly to the binder flowing under heat and pressure, and subsequently wetting out the flange surface, it is not surprising that the adhesion force at 400°F (204°C) is significantly higher than at 212°F (100°C). For this reason, the following testing was performed at 400°F for 22 hours.

In the course of the development of the Garlock Flange Free<sup>™</sup> Coating, Garlock was interested in examining the flange surface at high magnification after performing the F607 test. The test platens however were found to be too bulky to place into the SEM. To circumvent this issue Garlock fabricated



Figure 15: SEM micrographs of competitive carbon fiber gasket with non-uniform anti-stick coating



Figure 16: Garlock Style 9900 with Flange Free<sup>™</sup> Coating (Non Branded Side)

1 inch diameter disks out of 1018 Steel to serve as the flange surface. Samples of Garlock Style 3300, with and without Flange Free<sup>™</sup> coating were tested. Style 3300 was chosen because it is easier to track the chlorine compounds left on the surface of the flange (from the chloroprene binder) than to track natural and other synthetic rubber compounds with more common components.

When Flange Free<sup>™</sup> coating is not present on the surface of the gasket, the rubber binder can adhere to the flange surface. (See Figure 18.)

When a Style 3300 gasket is coated with Garlock Flange Free<sup>™</sup> coating, there were only traces of the Flange Free<sup>™</sup> coating and ink from the printed side. After repeated sealability testing without cleaning the

flange surfaces, neither the residual Flange Free<sup>™</sup> coating nor the ink adversely affected sealing performance. A macroscopic view of the 1 inch steel disk is shown in Figure 19, while a SEM micrograph of the same disk surface is shown in Figure 20.

#### Section 5 Garlock Flange Free<sup>™</sup> Coat-

**ing:** Insuring the coating does not impact the performance characteristics and physical properties of the sheet

Obviously, creating a coating that keeps the Garlock fiber gaskets from sticking to flanges is the main objective; however it is also imperative that the coating not have a detrimental effect on the gasket's performance. Garlock therefore tested gaskets to ensure that the materials maintained functionality and high performance characteristics when installed in flanges. Crush resistance, blowout resistance, sealability, and of course adhesion were all tested. The Flange Free™ coated products were then compared to materials with no coating, or in some cases, competitive products advertised as having an anti-stick coating.

### **Crush Resistance**

Crush resistance is possibly the property most affected when the wrong coating is used. A crushed gasket will spread sideways, towards the ID and/or OD, when the gasket can no longer handle more compression. The friction between the gasket and flange has a major impact on the amount of stress that can be applied before the gasket starts to split. As such, it's logical that a coating might affect that friction, and



Figure 17: Simulated flange surface after ASTM F607 testing with Garlock Style 3300 without Flange Free<sup>™</sup> coating.



Figure 19: Simulated flange surface after ASTM F607 testing with Garlock Style 3300 with Flange Free<sup>™</sup> coating.



Figure 18: 1000X SEM micrograph of simulated flange surface after ASTM F607 Testing with Garlock Style 3300 without Flange Free<sup>™</sup> coating.



Figure 20: 1000X SEM micrograph of simulated flange surface after ASTM F607 Testing with Garlock Style 3300 with Flange Free™ coating.

therefore lower the crush resistance of the gasket. Bolt lubricants and anti-seize compounds are, unfortunately, commonly applied to gaskets during installation. Garlock receives many gaskets every year that were coated with these materials, and have either blown out or were crushed and split apart. While lubricants certainly make the gasket easier to remove, they have a very negative effect on crush resistance, (See Figure 21.)

In this test, Style 9900 had a sudden change in thickness at a stress of approximately 17,000 psi, while the same material with Flange Free<sup>™</sup> coating survived 30,000 psi with no crushing. (Crushed gaskets show sudden thickness changes in a compression test when the gasket suddenly splits and flows sideways. These gaskets will no longer seal due to the substantial physical damage.)

### **Blow-out resistance**

Another property that can be affected by surface coatings is blow-out resistance, or the maximum internal pressure the joint can hold before gross leak and/or gasket rupture. As with crush resistance, the friction between the gasket and the flange surface is the largest factor in determining the pressure capability of a flange assembly. It can be shown mathematically that the outward forces on a gasket, created by the internal pressure pushing on the gasket's inside edge, will often exceed the tensile strength of a non-metallic gasket. It is friction that enables the joint to hold the system pressure. Coatings will affect the friction factor of a gasket in a flange assembly.

Blow-out tests were run in 2" 2500# raised face flanges, heated to 1000°F. The units were then pressurized until the joint leaked or the gasket ruptured. The results, Figure 22, show that Garlock Flange Free<sup>™</sup> coating did not adversely affect the gasket's pressure resistance.



Figure 21: Crush test results, Flange Free<sup>™</sup> vs. copper anti-seize



Figure 22: Flange Free<sup>™</sup> Coating Blowout Results, P x T Rating

## Sealability

The next property studied was sealability. Using the uncoated values as the baseline for comparison, sealability was measured (in ml/hr) using the ASTM F 37 method, for Fuel A and Nitrogen. Fuel A tests were done at a compressive load of 500 psi and an internal pressure of 9.8 psig, while Nitrogen tests were done at 3000 psi stress and 30 psig. In addition to comparisons between uncoated material and Flange Free<sup>™</sup> coated material, competitive gaskets were also tested with and without their "Non-stick" coating, Figure 23. Data for Figure 23 is provided in Table 2.

The comparison showed that Flange Free<sup>™</sup> coating had little or no effect on how well the gaskets sealed, while the competitive coating had a very detrimental effect on the leak rates. (Note: bolt lubricants used as coatings typically do not adversely affect sealability)

## Adhesion

Obviously, none of the above testing would be meaningful without an evaluation of the adhesion properties of the gasket. Again, uncoated and coated material as well as material from a competitor were compared. The adhesion properties were evaluated using ASTM F 607 methods. The platens were assembled with a 2 square inch gasket at a stress of 3000 psi, and heated in an oven at 212°F or 400°F. (Remember, it is the heat that creates the wetting out and adhesion of the rubber binder to the flange surface.) In these tests, the force to separate the platens was quantitively measured defining how much the gasket sticks. See Figure 24 for a graph of the required flange separation stress.

The stress required to separate two flanges is only part of the story when evaluating an anti-stick coating. An equally important aspect of gasket removal is the residue that the gasket leaves behind. In some cases, entire gaskets can be adhered to the flange. In others, a thin film is left in the serra-



Figure 23: ASTM F37 sealability relative comparison, Garlock Style 9900 vs. carbon fiber competitor

	Garlock 9900 Uncoated	Garlock 9900 With Flange Free <sup>™</sup> Coating	Carbon Fiber Competitor Uncoated	"Non-Stick" Coated Carbon Fiber Competitor
ASTM F37: Fuel A	1.00	1.12	1.49	4.48
ASTM F37: Nitrogen	1.00	1.13	12.56	31.79

Table 2: ASTM F37 sealability relative comparison, Garlock 9900 vs. carbon fiber competitor



Figure 24: ASTM 607 adhesion comparison, Garlock Style 9900 vs. carbon fiber competitor

tions of the flange. Garlock's Flange Free<sup>™</sup> coating minimizes both the required flange separation stress and residue left on the flange. Garlock Style 9800, Figure 25, was compared to a competitor carbon fiber gasket, Figure 26, and a competitor vermiculite gasket, Figure 27. While both the competitive carbon fiber and vermiculite gaskets adhere entirely to the flange, Garlock Style 9800 with Flange Free<sup>™</sup> coating was easily popped off the flange by hand.

Note: All extreme grade Garlock compressed fiber gaskets are shipped with Flange Free<sup>™</sup> coating as a standard. All performance and utility grade compressed fiber gaskets will be shipped with Flange Free<sup>™</sup> coating in the near future. Testing parameters were modified from the ASTM F607 specification to accommodate a larger gasket size, typically found in industry. Test parameters for Figures 25 - 27:

- Heated to 400°F for 24 hours.
- 25 ft-lbs. torque, creating 3600 psi compressive stress.
- 2-3/8" x 3-5/8" x 1/16" gaskets.
- 2", 600 lb flanges with 250 micro-inch serrations.



Figure 25: Garlock Style 9800 with Flange Free™ Coating



Figure 26: Competitor Carbon Fiber Gasket



Figure 27: Competitor Vermiculite Gasket

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