



*By José Antonio Almeida José Carlos C. Veiga* Teadit Indústria e Comércio Ltda Rio de Janeiro, Brazil

> November 18-20, 2003 Jacob Javits Convention Center New York, NY, USA

Authors: José Antonio Almeida José Carlos C. Veiga

Teadit Indústria e Comércio Ltda. Rio de Janeiro, Brazil

Keywords: PTFE Gasket Sheets PTFE Gaskets Filled PTFE Sheet Restructured PTFE Structured PTFE

#### 1. Introduction

PTFE gaskets are used in applications where it is necessary to achieve a high chemical resistance. PTFE has distinct physical properties to meet the needs of each application. As with any fluid sealing material there is overlap. Several materials can be used successfully in the same application. In this paper, several PTFE gaskets types available in the marketplace are compared. Tests, which included temperature cycle, torque creep relaxation retention, and compressibility, were performed and the results analyzed.

# 2. Molded Sintered PTFE Sheet

Molded Sintered PTFE sheets were the first products introduced in the market. Manufactured from virgin or reprocessed PTFE resin, without fillers. which is first molded, then compressed and sintered. As with any plastic product the PTFE exhibits a creep characteristic when subjected to a compression force. This characteristic is very detrimental to the gasket performance since it requires frequent retightening to avoid and reduce leaks. Creep behavior is increased dramatically with an increase in temperature. The main advantages of molded PTFE are low cost, ample market availability and high chemical resistance.

#### 3. Skived PTFE Sheet

Skived PTFE (sPTFE) is manufactured from virgin or reprocessed PTFE resin without fillers, by skiving a sintered PTFE billet. This process was developed to overcome the manufacturing deficiencies of the Molded Process. However its products have the same creep behavior problems.

#### Abstract:

To maintain a good seal, gasket materials must exhibit dimensional stability and resistance to temperature, pressure and chemical media. Conventional PTFE gasket materials have an outstanding chemical resistance but lack dimensional stability. Under pressure and temperature, conventional PTFE materials creep and flow. After a short period in service, gaskets made conventionally, from pure PTFE, are unable to maintain the high loads and as a result, lose thickness due to creep relaxation. Re-tightening is often necessary to keep the joint leak free. Several alternatives are used to reduce this problem. In this paper, several PTFE gaskets sheets available in the marketplace are compared. Tests like temperature cycle, torque retention, creep relaxation and compressibility have been performed and the results analyzed. From the results, it is shown that "Structured PTFE Filled Sheets" exhibit the best balance of the desired properties.

Shown in Figure 1 (with magnification of 100 times).

#### 4.Molded or Skived Filled PTFE Sheet

To reduce the creep behavior of Molded or Skived PTFE sheet materials mineral fillers or fibers are added. However, due to the manufacturing process (molding or skiving) this reduction is not enough to produce a long-term effective seal. Figure 2 shows the microstructure of a Filled Skived PTFE sheet with a magnification of 100 times. Visible are the silica filler particles in the PTFE matrix.

#### 5. Expanded PTFE

Before sintering, hot expansion of the PTFE gives it the ability to overcome creep. Gasket products expanded in one direction (cords or tapes) or bi-axially (sheets) can be produced. Figure 3 shows a one direction PTFE structure and Figure 4 shows a bi-axially oriented structure. Expanded PTFE (ePTFE) has a high chemical resistance and exhibits a very high compressibility ideal for use with fragile or glass lined flanges. Most Expanded PTFE products in the market do not have fillers. Its main drawback is the handling and installation of large gaskets or installation when it is not possible to separate the flanges. It is often used as a replacement for the Hollow Glass Micro-Spheres (Figure 5) sheet with the advantage of a higher chemical resistance; Glass Micro-Spheres are attached by Caustic Soda. However, in the case where gaskets are installed in a long line with several flanges in series, installation problems can occur due to the reduced gasket thickness after seating. The total length of the piping may not be enough to compensate.

# 6.Restructured Filled PTFE Sheet

То reduce the creep a new manufacturing process was developed to produce filled PTFE sheets. The material is subjected to a lamination process before sintering, creating a highly fibrillated structure. Creep at both room and high temperature is substantially reduced. To meet the chemical service needs several mineral or artificial fillers are used, such as Barite, Mineral and Synthetic Silica, Barium Sulphate or Hollow Glass Micro-Spheres. Each filler has a specific service application but there may be overlap in many of these applications. This process is referred as rPTFE. Figure 5 shows rPTFE filled with hollow glass micro-spheres with magnification of 100 times. The microspheres can clearly be seen inserted in the fibrillated PTFE matrix. Figure 6 shows a rPTFE filled with Barite with magnification of 100 times and Figure 7 the same product enlarged 500 times. Figure 8 shows a grain of Silica inserted in the fibrillated PTFE structure with magnification of 100 times.

#### 7. Tests Performed

Several tests were performed to evaluate the properties of each of material. The materials tested are identified throughout this paper as follows:

- First digit: <u>P</u> denotes a PTFE sheet
- Second digit: type of sheet
  - R restructured PTFE with filler
  - F skived filled
  - V skived virgin
- Third digit: type of filler.
  - B: barite
  - S: silica
  - G: hollow glass micro-spheres
  - F: fiber glass
- Fourth and fifth digit: manufacturer

#### Examples:

PRB11 – restructured sheet with barite, manufacturer 11. PFG19 – reinforced sheet with glass micro-spheres, manufacturer 19.

#### 7.1. Temperature Cycling

All temperature cycling test were performed using standard ASME B16.5 flanges. After installation of the gasket, heat is applied. When the temperature reaches 250°C (482°F) the system is pressurized with Nitrogen. After stabilizing the temperature, the heat is turned off until it reaches 28°C (82.4°F) when it is turned on again. This cycle is repeated 3 times. The pressure decay is monitored and reported. Figures 9 and 10 show the comparison between hollow-glass spheres filled rPTFE and sPTFE. Due to the higher creep relaxation; the sPTFE gasket lost about 63% of the initial N2 pressure. On the other hand, the rPTFE lost less than 1%. Figure 10 shows the reduction of the gasket stress of the skived material (PGV) compared with the rPTFE (PRG1). This reduction is the cause of the higher leak rate for the PVG product.

#### 7.2. Deformation under stress

Gasket specimens are compressed between two pre-heated smooth platens for one minute. Figure 11 shows pictures of the gaskets after the test. The temperature is 260°C (500°F) and the gasket stress is 10 MPa (1500 psi). The skived PTFE gasket deforms loosing its shape. This very simple test shows clearly the higher creep resistance of the rPTFE as compared with the sPTFE

#### 7.3. Creep at High Temperature

This test is performed following the DIN 28090-2 procedure. The test apparatus is shown in Figure 43. For PTFE products, compression modulus at room temperature (eksw) and at elevated temperature (ewsw/t) should not exceed 20% and 50% respectively. And elevated temperature test is performed at 150°C (302°F) for 16 hours. Figures 12 through 15 show the behavior at room temperature and Figures 16 through 19 at high temperature. Figures 20 show the average values at room temperature and Figure 21 at elevated temperature. The products filled with hollow glass microspheres show higher compressibility

than others. This material is designed for fragile flanges where this characteristic is required to assure the proper gasket seating. The less dense structure of this product can also be seen in Figure 5. Comparing, the sPTFE with rPTFE the former has higher compressibility. At 150°C (302°F) the creep for the sPTFE is about 50%, this value is very high than rPTFE results. This test confirms the findings of the Temperature Cycling described earlier in this paper. It can also be noticed that the Silica filler gives the best resistance to creep at high temperature.

#### 7.4. Stress relaxation

This test follows the DIN 52913 procedure. The test apparatus is shown in Figure 44. Sample material is installed with a seating stress of 50 MPa (7251 psi) and the temperature is raised to 150°C (302°F). After 16 hours the remaining gasket stress is measured. Figures 22 through 25 show rPTFE results. Figure 26 shows the comparison of the average values for sPTFE and rPTFE. It can be noticed that the diverse rPTFE products have a similar behavior for each kind of filler, and they exhibit lower stress relaxation than the sPTFE products.

#### 7.5. Sealability ASTM

Using ASTM F37 B procedure the test sample is seated with 7 MPa (1000 psi) and tested with isooctane at 0.7 bar (9.8 psi). The testing apparatus is shown in Figure 46. Results are show in Figures 27 through 31. Regardless of the filler, rPTFE products have a higher sealability than the sPTFE. In addition the Barite filled rPTFE showed the best results.

#### 7.6. Compressibility and Recovery

This test follows the ASTM F36 procedure. The compressibility is the change in thickness when a seating stress of 14.5 MPa (5000 psi) is applied on the material. The recovery is how much it recovers when this load is removed. Both are expressed as a percentage of the initial thickness. Figures 32 through 37 show the test results. The charts show that the materials can be separated as either being of high or low compressibility. If the filler is hollow glass micro-spheres, no mater if it is skived or restructured, the compressibility and recovery are high. For other fillers, the

compressibility is lower and the recovery is high, if taken as a percentage. However, if analyzed as an absolute value it is very low. Considering the average values both sPTFE and rPTFE have a similar behavior.

#### 7.7. Sealability DIN

This test follows the DIN 3535 part 4 procedure. This standard is used to qualify Gaskets for use with gas valves, appliances and pipe work. At 32 MPa (4641 psi) gasket stress is applied and the leak rate measured with Nitrogen at 40 bar (580 psi). To qualify for this application the leak rate must be less than 0.1 mg/sec.m. The test apparatus is shown in Figure 45. All rPTFE products show a leak rate that qualifies for this On service. the other hand. skived/molded products may not qualify, as shown in Figures 38 to 42.

#### 8.Scanning Electron Microscope (SEM)

To examine the morphology by the SEM analysis, specimens were fractured and gold coated. Figures 47 through 51 show two different morphologies; Virgin or Filled Skived PTFE exhibit fine fibrils (Figures 47 and 48) on the other hand Restructured PTFE (Figures 49 through 51) show larger fibrils in addition to the fine ones. This difference in morphology explains the better mechanical properties of rPTFE sheets.

#### 9.Conclusions

Fibrillations of the PTFE matrix during the manufacturing processes along with the fillers increase the mechanical properties especially at high temperatures, overcoming the most undesirable property of PTFE based gaskets which is the high temperature creep relaxation.

It was shown different fillers meet the demands of the application such as high compressibility for fragile flanges or high mechanical strength for higher pressures.

The test results described throughout this paper clearly show better performance of the restructured filled PTFE gasket sheets for maintaining a good seal when compared with both filled and unfilled skived products.





Figure 2







































































Figure 45

Figure 46













