



## Bulletin No. 206

### Temperature Limits of High Temperature Systems

Factors that determine the maximum allowable working temperature in reactors

### Temperature Limits

In this Tech Note we would like to discuss temperature limits, material of construction, gaskets, seals, high temperature operations and some alternative designs for very high temperature operations. There are a number of factors that determine the MAWT (maximum allowed working temperature) limit for a pressure vessel. For most applications it is the gasket or seal material. Other factors include materials of construction, gaskets, design, heater capabilities and accessories to name a few.

Vessels with O-ring seals are limited to 225°C unless exotic materials are used to extend this temperature to 275 to 300°C. PARR's design for contained PTFE gaskets extends the operating temperature range to 350°C. Flexible Graphite (FG) material essentially removes the gasket as the limiting factor. Here we are discussing the temperature limit itself, not the pressure limit which is also dependent upon operating temperatures. A separate tech note goes into detail on the various pressure and temperature limits of a PARR reactor.

### Materials of Construction

Generally the first thing we think of is the material of construction required for the application desired. We then must consider the maximum allowed working temperature of a vessel required. We assume everyone knows that the maximum operating pressure of a vessel is reduced as the strength of the construction material falls off at elevated temperatures. There is a maximum temperature for each material we use. The allowable strength for these metals falls off rapidly as they reach maximum operating temperature. Finally, the difficulties encountered with screw threads and other closure components operating at high temperatures



establish a practical temperature limit for externally heated vessels. We have found 600°C to be a reasonable limit.

Current maximums are:	
Material	Maximum Temp. ° C
T316SS	816
Monel Alloy 400	472
Inconel Alloy 600	650
Hastelloy Alloy B2/ B3	427
Hastelloy Alloy C-276	677
Nickel Alloy 200	316
Carpenter 20Cb3 Stainless	427
Titanium (All Grades)	316
Zirconium	371
4140 Steel (Split Rings)	538

You may have to drop well below the maximum allowable working temperatures to get really usable design strength for some of these alloys. It is important to note that these limits are for each component material of construction of the reactor. For example, the split rings are normally several hundred degrees cooler than the internal temperature of the cylinder and head as our standard heaters do not heat the split rings. However, this can become the controlling factor when small vessels are heated by placing the entire vessel in a furnace.

We will not design or rate vessels for a temperature above this maximum. We believe the code committee that established these limits has done so for good reasons and we think it is in our customer's best interests to abide by them. Again, a separate tech note goes into detail on the various pressure and temperature limits of a PARR reactor.

## Gaskets and Seals

The second thing we think of as we consider the maximum allowable working temperature of a vessel is the material of construction of the main head gasket. Gaskets fall into ranges rather than sharp cut-offs as temperature increases. Gaskets used on PARR reactors come in a variety of styles and materials.

There are four different types of gasketing material for the main head seal in PARR reactors and pressure vessels, each with its own advantages and limitations. Some of these are recent additions which have significantly



expanded the choices a user can consider when selecting a closure and gasket material for the intended operating conditions. Typical styles include flat PTFE gasket, o-ring, flat Flexible Graphite gasket, and metal gaskets.

## **Confined and Contained Flat PTFE Gaskets for Temperatures to 350°C**

The traditional and most popular main head gasket for PARR vessels is a flat gasket made of a PTFE fluoropolymer. In PARR flat gasket closures the gasket is held in a recess in the vessel cover. A matching pilot on the cylinder closes the recess, leaving the gasket completely confined with only a small inside edge exposed to the reactants within the vessel. This combination of complete gasket containment and the exceptional properties of PTFE materials produces a reliable closure for working temperatures up to 350°C.

Flat contained gaskets require an initial loading pressure in order to develop and to maintain a tight seal. In PARR designs this is produced by tightening a ring of cap screws in a split-ring cover clamp. Fortunately PTFE is slightly “plastic” and will flow under pressure, producing a seal that improves with each use as the gasket is forced into the faces on the head and cylinder. It also is a very forgiving seal which does not require the special care needed to achieve a uniform loading, which is essential when working with a metal or other non-plastic gasket material. An equally important advantage of the PTFE gaskets is their essentially universal chemical resistance.

## **Self-Sealing O-Rings**

PARR has greatly expanded its offerings of reactors and vessels which feature self-sealing O-ring closures. In these designs the sealing force on the gasket is developed from pressure within the vessel itself, eliminating the need for cap screws in the split ring to pre-load the seal. In these self-sealing closures the split ring sections simply lock the head and cylinder together.

Users who select the self sealing o-ring design must consider two important characteristics of elastomeric materials. First, they will not withstand operating temperatures as high as the PTFE gaskets. Secondly, none of these materials offers the universal chemical resistance of PTFE polymers. The chemical resistance is especially important since the o-ring is directly exposed to the contents of the vessel.

Although there are a number of available O-ring materials, the real choice comes down to two: Fluoroelastomer (FKM) O-rings, such as Viton, are a first choice for Parr self-sealing closures. They have good chemical resistance and a working temperature up to 225°C Perfluoroelastomer (FFKM) O-rings, such as Kalrez, have extremely broad chemical resistance and can be used at working temperatures up to 275 to 300°C. Unfortunately, this material should probably be

considered an “exotic” because it costs approximately 80 times as much as an FKM O-ring. And while it will raise the allowable working temperature to 275°C, as a practical matter, most users intending to work at this temperature level would be well advised to choose a closure with a flat PTFE gasket and a 350°C temperature limit. Other exotic O-ring materials are available, and there are economically priced materials such as ethylene-propylene that will resist some materials that cause FKM to fail, with only slight sacrifices in operating temperatures.

### **Contained Flat Flexible Graphite Gaskets for Temperatures to 600°C**

For operating temperatures above 350°C, PARR uses a recently developed flexible form of graphite, called Grafoil, which has proven to be an excellent high temperature sealing material. It consists of flexible layers of graphite bonded together to produce a gasket that is almost as easy to seal as a flat, PTFE gasket, but with an almost unlimited temperature range and excellent chemical resistance.

PARR has converted all of its standard designs to accept a flat, Grafoil gasket whenever operating temperatures above 350°C are required, replacing the metal gaskets formerly used for high temperatures. These flexible graphite gaskets are held in grooves identical to the ones used for PTFE gaskets and sealed with the same split-ring closures. This makes it possible to substitute a PTFE gasket whenever the vessel is to be used at temperatures below 350°C. Grafoil gaskets are reusable, but their service life is shorter than can be obtained with a PTFE gasket.

### **Metal Gaskets**

Metal gaskets have traditionally been the only gaskets available for use at temperatures above 350°C. PARR has designs for diamond cross-section metal gaskets which can be furnished for special applications, but we would recommend the flexible graphite gaskets described above for most applications.



## Trademarks of Sealing Materials

***A number of gasketing materials have so dominated their product categories that their Trade Names have become more common than the actual material designation itself. In an attempt to respect the value of these Trade Names and their proper usage and to minimize the disruptions in our descriptions, we have adopted the following generic material descriptions and designations for use in this catalog. Where available we have selected the ASTM material designation.***

<b>Common or Trade Name</b>	<b>Material</b>	<b>Designation</b>
Viton®	fluoroelastomer	FKM
Kalrez®	perfluoroelastomer	FFKM
Teflon®	tetrafluoroethylene polymer	PTFE
Grafoil®	flexible graphite	FG

***Viton®, Kalrez®, Teflon® are Registered Trademarks of DuPont. Grafoil® is a Registered Trademark of Graphtec, Inc.***

## Design

There are a number of design techniques that can be used to increase the temperature rating of a vessel. One of the problems that will occur with very high operating temperatures is that cap screws will oxidize, rust, seize and/or deform unless they are very carefully protected. Some designs use screw caps and big wrenches instead of fine threaded cap screws, and some seal the head directly to the vessel without a gasket. Other users simply weld the heads on and cut them off after the test. Basically there are only two design techniques we use. One is to make the cylinder of the vessel long like our tubular flow vessels and heat only the center section while allowing the closures to air cool and remain well below the hot zone temperature. The other is the internally heated vessel discussed below. While we will be the first to admit that it is an arbitrary limit, we have basically chosen 600°C as the maximum temperature for which we will rate an externally heated vessel.

## High Temperature Systems

### Internal Heaters

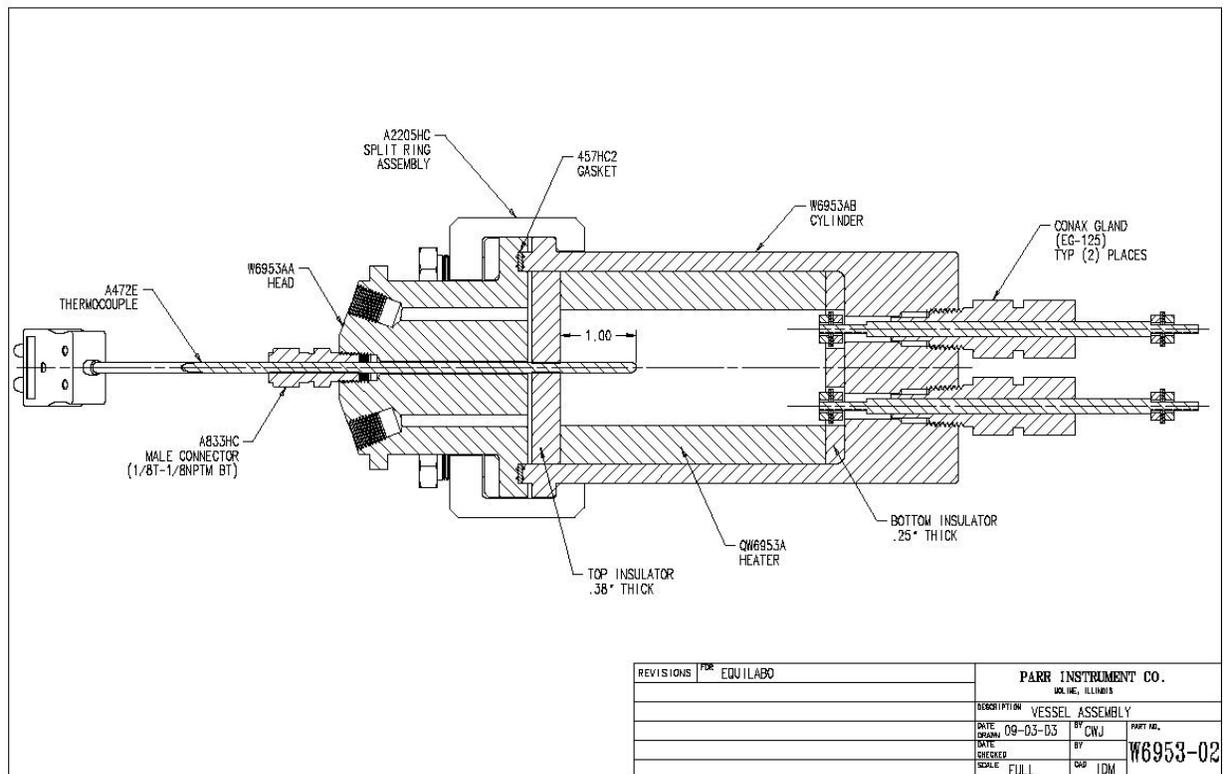
Another approach has proven useful in extending the maximum temperature limit. In this design the heater or furnace is placed inside the pressure vessel. This heater is surrounded by a layer of insulation. This creates a hot zone in the



center of the vessel and prevents the walls from exceeding their allowable limit. Properly designed, temperatures as high as 1200°C can be achieved in the core of the vessel while the walls remain below 250°C. This system is very energy efficient. Internal heaters can be less powerful than external heaters. Internally heated vessels are equipped with insulated electrical feed throughs to power the heater and multiple thermocouples to monitor and control the temperatures in the hot zone and the vessel inner wall.

The reactions or studies carried out in internally heated vessels must be limited to those which will not destroy the exposed internal heaters and insulation. These are normally gas-solid reactions or controlled atmosphere heat treatment studies. The heating elements are normally ceramic. Some users have developed induction style heaters and insulators and have extended their investigations to above 2500°C.

Although internal heaters can be installed in almost any non-stirred Parr pressure vessel, the 450 ml, Model 4762 pressure vessel is an excellent starting point. It can accommodate a cylindrical, insulated heater with an internal diameter of 1-1/2 inch that is 4 inches deep. This vessel is capable of producing and sustaining internal temperatures to 1200°C at an internal pressure of 3000 psi.



Model 4762 with Internal Heaters



## Protected Heaters

Internally heated vessels have also been produced using cartridge type heating elements inserted in specially designed "thermowells". These wells protect the heater from the reactants and expand the applications that can be studied. Cartridge type heaters have a maximum temperature of 850°C.

## Attached Accessories

Stirrer drives, needle valves, ball valves, pressure transducers, electrical glands and sampling systems are a few of the accessories which may not have a temperature rating as high as the vessel to which they are attached. Stirrer drives and pressure transducers are routinely protected with cooling sleeves, valves and gages are commonly installed where they will not be heated and will be air cooled. At any rate, a full analysis of each system must be made to determine that under the planned operating conditions, all of the components of the system will be well within their normal operating temperature and pressure limits.

## Special Custom Applications

We continue to believe that our willingness and capabilities to produce customized vessels for unique operating requirements are two of the valuable things we have to offer to you and to your customers. While we can not solve all of the application problems that come our way, we do invite your inquiries for systems with these higher operating temperatures.

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