WELDED METAL PRESSURE SEALS FOR PROCESS SPECTROSCOPY

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INTRODUCTION

Continuous chemical analysis by means of in-situ molecular spectroscopy offers enormous potential benefits for the chemical process industries. In particular, it provides molecular structure information in real-time under actual reaction conditions while eliminating the costs and difficulties associated with sample extraction and conditioning. And, in principle, it can be applied under a wide range of conditions – such as extreme temperatures and pressures, highly reactive chemistries, and rapid chemical change – that would be quite problematic if not impossible for an extractive system. (See Technical Note: AN-918.) Despite these substantial benefits, in-situ process analysis still accounts for a very small fraction of the overall process control market. One major reason is that process engineers and plant managers are loath to introduce any unfamiliar or unproven equipment that could potentially disrupt production. Historically, these concerns have often been well founded. The available process interfacing equipment – i.e. optical probes and flow cells – simply did not possess the degree of robustness needed to provide long-term reliability under real-world process conditions. In particular, the methods used to seal the required optical window into the body of the probe or cell were not adequate to handle the wide range of conditions occurring in many processes.

There are three basic "realities" of in-situ process sample interfacing:

1. The sample interface is the most vulnerable and hence most critical part of the analysis system.

2. The cost of failure is far greater than the cost of the sample interface.

3. It is often the unexpected condition that poses the greatest threat.

In view of these realities, it is clear that virtually armor-clad sample interfacing equipment is needed in order for in-situ spectroscopic process analysis is to become widely accepted.

THE LIMITATIONS OF PREVIOUS WINDOW SEAL APPROACHES

In the past, windows have been sealed into sampling devices by four different means: O-rings, spring-energized elastomeric C-rings, brazing, and sweated-in press fit. Each of these is discussed briefly in this section.

O-rings are poorly suited to applications which involve temperature cycling over wide ranges since those elastomeric materials which have good chemical resistance tend to deform and take a set at high temperature. This results in a compromised seal when the temperature is reduced.

Spring-energized elastomeric seals are a definite improvement over O-rings for most process applications. However even these can leak when subjected to rapid temperature changes. And the small cross-section metal springs used to provide sealing pressure are gradually degraded by processes involving strong acids.

Brazing is plagued by a fundamental conflict between two basic requirements – the need for good chemical resistance and the need to function over a wide temperature range. The thermal requirement is driven by the disparity in expansion coefficients between the window material (typically sapphire or fused silica) and the metal body (stainless steel, Hastelloy C-276, or other alloys). In non-chemical applications, the thermal expansion issue is handled by brazing the window to a low expansion metal such as Kovar or Invar. However, all of the suitable low expansion metals have a high iron content and thus are subject to attack by a wide variety of chemicals.

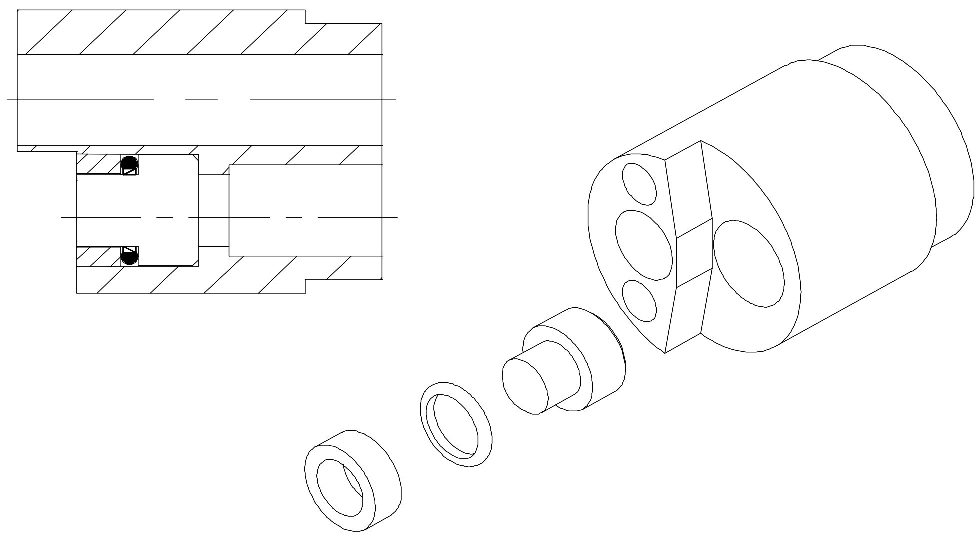

Figure 1: Cross section and exploded view of Axiom's welded metal seal assembly as used in the FPT-850 transmission probe.

In the past, attempts have been made to avoid the chemical attack problem by brazing sapphire windows directly to stainless steel or Hastelloy by means of either silver or gold alloy braze. However, these brazes tend to be brittle and, when subjected to the stresses caused by differential expansion, develop cracks over time. The question isn't so much if a braze will fail, but when.

Pressed-in windows have been successfully used for a few selected applications – such as polymer melt monitoring – where the sample medium is viscous. This sealing approach – which works much like a stopper in a flask – relies on the friction between the metal and a tapered sapphire widow to maintain integrity. It has been found to fail unpredictably at high temperatures or when subjected to side loading.

THE AXIOM WELDED METAL PRESSURE SEAL APPROACH

In order to provide a much more robust window seal than those previously employed, we have turned to the metal sealing technology commonly used in the aerospace and nuclear industries, diesel engines, and extreme chemical processing applications. Extreme duty metal seals are typically in the form of C-rings or O-rings fabricated from a variety of metals. They are designed for high performance in demanding situations and are used under such conditions as: vacuum to 10^{-11} torr, pressures to 6000 bar, and temperatures from - 250 ^{o}C to over 1000 ^{o}C. In their conventional applications, extreme duty metal seals serve as either face seals or axial seals and are clamped between mating metal parts by means of a bolt circle or other threaded structure. This approach is not particularly practical for use in most process monitoring applications. For example, the inclusion of a sufficiently robust threaded structure in a spectroscopic immersion probe would significantly increase both the size and cost of the probe. Our solution to this problem is to capture the seal along with a specially shaped sapphire window within a permanently-welded metal structure (Patent Pending).

The welded metal seal approach involves six elements: a custom shaped sapphire window, a Hastelloy C-276 body, a Hastelloy C-ring seal, a coating of a more compliant material such as gold, PTFE, or nickel, a Hastelloy press-fit sleeve, and an electron-beam weld. Figure 1 illustrates one of the two window assemblies used in an FPT-850 near-IR transmission probe. The seal is mounted on a shoulder on the sapphire window. This, in turn, is mounted against a shoulder in the window housing.

The assembly is then pressed together by the tightly fitting Hastelloy sleeve. This compresses the C-ring the specified amount to provide proper loading. At this point, the assembly is tested under vacuum using a Helium leak tester. After the assembly passes this test, the sleeve is permanently electron-beam welded in place and a second Helium leak test is performed.

The final assembly of the probe head involves electron-beam welding two window assemblies, a retro-reflector assembly, and a probe body together. The resulting structure is shown schematically in Figure 2. Figure 3 is a photograph of an FPT-850 probe, complete with a mounting flange as used in a typical application.

PERFORMANCE

With the welded seal approach, the only materials in contact with the process are sapphire, Hastelloy C-276, and a thin flash of gold, PTFE, or nickel, depending on the application. The capability for withstanding extreme temperature cycling is provided by thermally balancing the components of the structure and by the Hastelloy seal, which is compressed at high pressure prior to welding. This approach eliminates the sources of fatigue and stress failures common with brazed seals. And the elimination of elastomeric seals provides for reliable long-term operation at extreme temperatures. The resultant seal is highly resistant to corrosion due to its Hastelloy construction. Loading and retention of both the window and the metal seal are made permanent by the electron-beam weld in the minimum possible space with the fewest possible mechanical parts.

Metal C rings are designed to undergo both plastic and elastic deformation when properly installed. Plastic deformation enlarges the contact area to bridge across surface imperfections in the mating surfaces. High integrity sealing is further insured by the ductile outer plating layer of the seal (gold, silver, PTFE, or nickel as appropriate for the chemical environment) which, being inelastic and of relatively low compressive yield strength, flows into and fills the mating surface irregularities. Elastic deformation provides “spring-back” to maintain good sealing under thermal expansion.

Axiom’s welded metal seal design is currently used in various process probes rated for continuous operation at pressures to 200 bar and temperatures to 400 ^{o}C. These ratings are far below the limits of the various elements in the seal assembly. Thus, when operated within these limits, the seals should provide virtually unlimited service.

Compared with conventional elastomeric seals and the various types of brazes, the welded metal seal design provides:

- Greatly increased seal life
- Greatly increased temperature range
- Greatly increased capacity for temperature cycling
- The ability to handle high temperatures and pressures simultaneously
- The ability to withstand a wide range of aggressive chemical conditions

IMPLEMENTATION

The welded metal seal design was originally developed for use in Axiom’s FPT-850 near-IR transmission probes. Since these probes were first introduced during 1999, Axiom’s sales of near-IR transmission probes have been increasing at a compound rate of nearly 50% per year, and the FPT-850 has been adopted as the standard in-situ sampling product by the majority of the significant manufacturers of near-IR spectroscopic analyzers. With scores of FPT-850’s currently in service – including several multiplexed multiple probe installations – the welded metal seal design is already

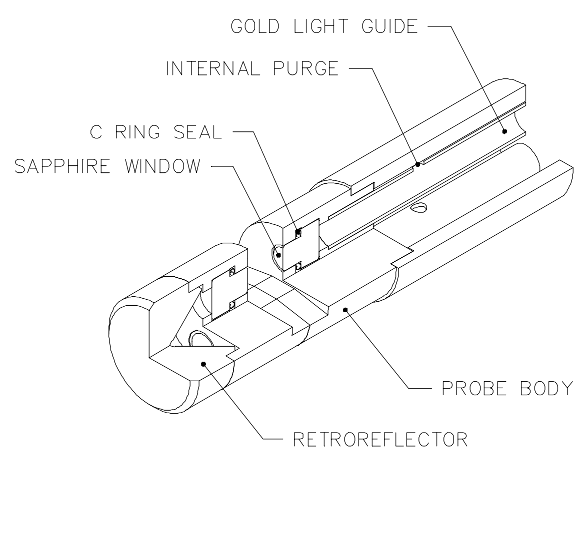

Figure 2: Breakaway view of the FPT-850 probe tip.

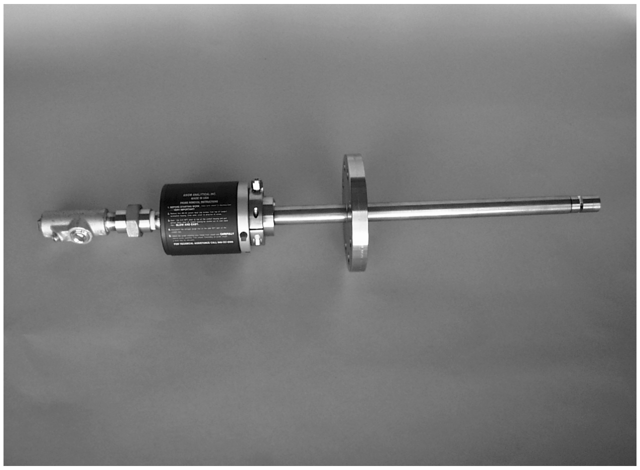

Figure 3: Photograph of the complete FPT-850, including conduit termination housing and mounting flange.

proving to be a major factor in the newly invigorated growth of in-situ near-IR process analysis. As a result of the success of the FPT-850, Axiom has now introduced four additional products utilizing the welded seal design. These are the FDR-780 Process Diffuse Reflectance Probe, the FCT-880 Extreme Duty Transmission Cell, the FPT-1850 Large Scale Transmission Probe, and the RFP-480 Process Raman Probe. The FPT-1850 and RFP-480 are particularly significant. The FPT-1850 provides the safety and convenience of top entry while extending the benefits of in-situ near-IR analysis to batch reaction vessels of virtually any size. The RFP-480, on the other hand, is the first Raman probe designed to meet the needs of the great majority of process applications. It thus promises to perform the same role for the very promising field of Raman analysis that the FPT-850 is now doing for near infrared.