

Design and Materials Selection of Autoclaves and Auxiliary Equipment

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ABSTRACT

Over the past 50 years, the use of autoclaves in the metallurgical industry has evolved from final metal recovery (hydrogen reduction) and purification (sulphide precipitate oxidation) to treating whole ores and concentrates. Recent process developments, such as the use of autoclaves for laterite leaching, have necessitated higher temperature and pressure operations, which in turn have seen marked advancements in technology and manufacturing capabilities to keep in step with process demands. This paper will discuss the advances in technology in terms of design, materials and construction of autoclaves and liners, typical dip tube design and geometric layout. The paper will also address specific auxiliary equipment, such as acid and steam injection systems, and components, such as agitator seal systems.

Vessel Design

General

The most common codes that have been used in the design of pressure vessels for pressure hydrometallurgy are ASTM Div 1 (North American), AS-1210 (Australian) and BS-5500 (British). The codes are similar, however, they have some variances in allowable stresses and design requirements, e.g., for masonry lining that will result in minor differences in shell thickness.

This section outlines the design considerations based on the ASME code.

In North America, unfired pressure vessels are designed to either ASME Section VIII, Division 1 or to Division 2. Division 1 uses approximate calculation methods adequate for most services. Division 2 provides an alternative to the minimum construction requirements of Division 1. Division 2 rules are based on more precise calculation methods and are more restrictive. For example, they do not permit the use of some materials that are allowed by Division 1, they prohibit some common design details and they specify which fabrication procedures may be used. The engineering and manufacturing costs will be more than for Division 1 criteria.

Where the stress intensity is controlled by ultimate or yield strength, Division 2 permits the use of higher design allowable stress values in the range of temperatures covered. Hence, for autoclave applications, Division 2 may be considered where savings in materials and labour justify the costs of the necessary engineering analysis

and more rigorous construction requirements. Where expensive materials or large vessels are being used, this may be the most cost effective approach.

In some cases, material savings accompanying the Division 2 criteria are not substantial until the shell thickness approaches 4" (100 mm) for carbon steel materials.

ASME SECTION VIII PRESSURE VESSEL CODE CHANGES

There are various criteria used in establishing pressure vessel code maximum allowable stress values. It is important to note that the Division 1 safety factor was recently changed from 4:1 (the figure used since 1944) to 3.5:1, bringing the stresses closer to those of most European pressure vessel codes.

This change was issued on July 1, 1999.

Also, the temperature at which the allowable stress values are affected has changed. For example, previously, a design temperature of 650°F (343°C) or less did not affect the allowable stress value of SA-516-70, a carbon steel plate material in the pressure vessel code and commonly used in vessel fabrication. That temperature value has been changed to 500°F (260°C). Yet, the allowable stress value for SA-106-B, carbon steel seamless piping material also used in vessel fabrication, does not change until 650°F. Each item needs to be reviewed to determine the allowable stress at the vessel design temperature. It should also be noted that the design temperature always affects external pressure calculations and flange ratings.

In addition to the change in the allowable stress values, the required minimum hydro-test pressure has changed from 1.5 times the maximum allowable working pressure (MAWP) to 1.3 times MAWP.

Vessels designed to the new Division 1 criteria, will be thinner, lighter and will cost less than vessels installed under the earlier code criteria. The material cost savings between Division 1 and 2 vessels will not be as significant under the new code rules.

An important impact to operators is that the increase in allowable stresses could lead to existing vessels being re-rated for higher design pressures and this could benefit some processes. A National Inspection Board Code interpretation indicates that re-rating due to code changes in allowable stresses is acceptable as long as certain criteria are fulfilled, however, some jurisdictions will still not allow it.

AUTOCLAVES

General

Currently accepted construction options for the vessels are titanium clad steel, a carbon steel shell protected by an impermeable membrane and acid resistant brick.

Brick-Lined Vessels

Historically, the most common design for pressure oxidation applications has been horizontal vessels with a carbon steel shell, lead and/or vinyl ester membrane, and two layers of acid resistant brick lining.

The ASME pressure vessel codes do not specifically consider brick linings other than to take into account the lining weight. However, the codes do state that determination of additional loads placed on the shell from the lining must be made. The British code, BC-5500, does contain design guidelines.

A primary objective of lining designs is a balance in stresses between the steel shell and the lining. Since the tensile strength of the acid brick and mortars are relatively very low, they must be maintained under compression or, at the worst, a small amount of tension. On the other hand, the compression must always be below the compressive strength of the brick to prevent spalling or crushing and to not impose severe additional tension on the steel vessel wall.

A number of variables contribute to lining stresses, such as:

- Selection of materials for acid resistance and erosion resistance (includes brick mortar and membrane). In selecting the materials for the bricks, consideration has to be given for the fact that bricks irreversibly swell under wet acidic conditions.
- Thermal properties of the brick and mortar (used to calculate thermal gradient across the lining system and hence temperature of each component and its corresponding expansion).
- Number of brick layers and thickness.
- Operating pressure and temperature.

Stresses are developed in the lining system before and during operation. Prior to a newly lined vessel going into operation, it is cured in an acid solution at a temperature around the boiling point. During this curing, a chemical reaction occurs in the brick that causes it to irreversibly swell, thereby, increasing the stresses and “tightness” of the lining system. Bricks have been known to swell during an initial period of a few weeks, stop and continue months later.

The lining system will be subject to various stresses during start-up, normal operation and shutdown and must be designed accordingly along with procedures for heat up and cool down to minimize stresses that could damage the lining. These procedures include rates for pressurization and depressurization and rates for temperature increase, time for soaking and rates for temperature decrease.

In addition to the normal lining stresses, there are the additional loads imposed on the lining system by the vessel, such as those due to out of roundness of the vessel or bending of the steel shell. Tolerance criteria for out of roundness and deflection have been developed for the vessel to minimize the additional loads that will be transferred to the brickwork.

There are three major companies that design and install pressure vessel linings in North America, namely Stebbins, Didier and Koch. Each company has recommendations regarding materials for membranes, bricks and mortars and also for installation details and techniques. For example, Koch prefer to supply a sulphur enhanced elastomer (Pyroflex™) membrane, which, due to its flexibility, will take up

some of the brick expansion. For pressure oxidation vessels, Stebbins use their vinyl ester (AR 500™) membrane and Hydromet™ (lead-based) mortar for difficult vapour zone applications in oxidation applications. Didier developed their potassium silicate (Stellakit A™) mortar for the vapour zones. The companies have bricks that differ in swell properties and resistance to the vapour zone conditions, where, under steam condensing conditions, the bricks and mortar tend to soften.

Lead has been the traditional membrane lining for pressure oxidation service. Development of materials to replace the lead membrane is a priority for the vendors as awareness of the health hazards of lead has become apparent. Installation of a lead liner requires substantial safety precautions, is expensive and time consuming to apply, and there has been corrosion in areas around the vapour space nozzles where operating temperatures are highest.

One of the brick lining companies is testing fluoropolymer membranes to replace the lead membrane and has successfully used it in flash tank service.

Experience has shown that the lining companies should provide the single point design and supply of the autoclave system, including the vessel and the lining. They are most familiar with the performance and limitations of their products, including the thermal expansion and swell properties. As a result, they are in the best position to determine vessel design loads, and roundness and deflection tolerances and to pass this information along to the vessel fabricator.

TITANIUM VESSELS

Clad Vessels

Vessels are fabricated from carbon steel with an explosively bonded titanium cladding. Titanium and iron are not metallurgically compatible at high temperatures, and under conditions normally used for weld overlay or hot roll bonding, titanium and steel instantly react to form brittle compounds. Consequently explosion cladding is the preferred process for the manufacture for titanium clad steel, which is a solid state metal joining process that uses explosive force to create an electron sharing metallurgical bond between two metal components. The titanium cladding is used as a corrosion barrier only; its strength is not taken into account when designing the shell wall thickness. Optimum bond mechanical properties and plate sizes are produced when the yield strength of the titanium and base metal are below 345 MPa. Consequently, the optimum bond strength and toughness of titanium cladding results from a combination of titanium clad and a moderate strength pressure vessel steel such as SA 516 Gr 70. Titanium grades 1, 11 and 17 exhibit similar yield strength and bond performance and have been used to clad autoclaves used for nickel processing.

Special considerations must be taken in design, fabrication, welding and testing to ensure a reliable product. Clad fabrication is typically accomplished using a batten strap technique as shown in Figure 1.

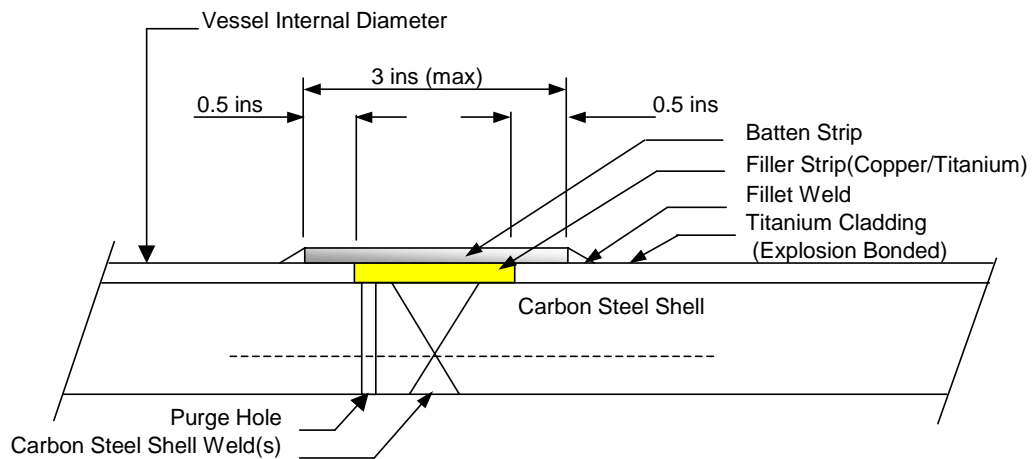


Figure 1a

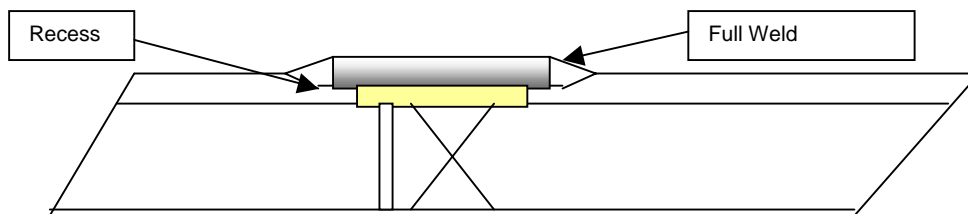


Figure 1b

Figure 1 - Batten Strap Techniques

The cladding is typically applied to a flat sheet of carbon steel of a thickness suitable for the pressure vessel design with the addition of a minor allowance for compression of the steel during cladding. The plate is formed into heads and rolled into section. To join two sections requires that, typically, 12 mm (0.5") titanium be removed from each side of the weld prep edge. The steel is welded using conventional fabrication procedures. The vessel is cleaned and prepared for titanium welding. In the batten strap technique, a filler, metal strip of copper or titanium, is used to fill the space where the titanium has been removed, and a titanium batten strip applied over the plate and the edges fillet welded to the clad titanium. The wider the joint the higher the stresses in the fillet weld during operation. Large diameter nozzles are frequently fabricated from clad plate using the same procedures. Small nozzles are typically lined with titanium sleeves and seal welded in the shell.

In some of the operating nickel autoclave plants there have been problems related to titanium material selection. All of the vessels have suffered cracking at the batten strips due to thermal expansion issues. Batten strip design and test procedures, such as hot cycle testing, have been developed to minimize failures during start-up and operation.

Figure 1a shows the conventional design of batten strip where only a fillet weld has been used to attach the batten strip. It is this design that has had problems. Figure 1b is a modified version whereby the vessel cladding has been recessed for the

batten strip, allowing a full weld and increasing the strength to accommodate the differences in thermal expansion.

Vessel Heads

There are some options in the selection of heads for the vessels in a pressure hydrometallurgical circuit. The selection is generally made on cost and delivery and, for the higher pressures, the head selection is between hemispherical and ASME 2:1 elliptical. While a 2:1 head may be preferable because of the agitator placement in the “volumetric” centre of a compartment, a hemispherical head does not change these volumetric centre criteria to the extent that it affects operations.

Both types of head can be brick lined or explosion clad.

Nozzles

Historically, the most troublesome issue in the maintenance of brick-lined autoclaves has related to the vessel nozzles, in particular, the nozzles located in the vapour space. There can be up to 30 nozzles on a vessel. To minimize penetration in the brick linings, nozzles are grouped together into a “multiple nozzle” with one lining penetration. Initial autoclave designs had a lead membrane that extended for the vessel shell through the nozzle and over the face of the flange. One of the principal problems was keeping the lead at a low enough temperature (<85°C) to prevent creep and corrosion because there was insufficient annular space provided in the nozzle to allow for the temperature to drop from the operating temperature to an acceptable membrane temperature. Insulating bricks, polyethylene blocks and insulating rope have been tried with limited success. Designs with fibreglass over the lead, or the lead replaced with a sulphur-enhanced elastomer have also been tried. The most successful design has been the use of an Inconel 625 overlay through the length of the nozzle and into a “bulls eye” in the vessel shell, with the overlay protected by brick and/or mortar and an insert. The temperature is not critical in this design and the Inconel has withstood the environment that exists behind the brick lining. The insert is used to protect the bricks in the nozzle from mechanical damage and will limit the exposure to condensate that would otherwise form and flow down the bricks and mortar causing premature failure.

At the face of the nozzle flange, the termination of the membrane(s), the nozzle insert and sealing surface all occur. This requires the fabrication of a custom designed Inconel, or other high alloy seal ring fitted onto the face of the flange. The rings are designed to prevent the autoclave environment from reaching the steel shell by sealing the ends of the membrane(s) and across the nozzle insert, and will include machined sections for the gasket(s). Spiral wound gaskets and non-metallic gaskets have both been used successfully. Spiral wound gaskets have very high seating stress requirements, hence the gasket width should be carefully selected so that the bolting and flange thickness do not have to be custom designed. Figure 2 shows typical sealing face arrangements for brick-lined and clad vessels.

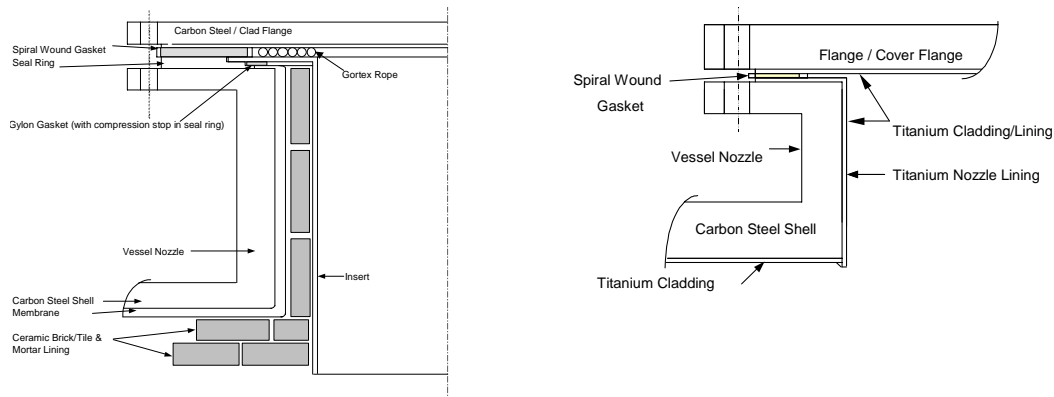


Figure 2 - Typical Sealing Face Arrangement

Nozzles on pressure vessels may be supplied as integrally reinforced components or fabricated from pipe and flanges with reinforcing pads. The pressure vessel code requires that the area of the shell removed by the opening must be replaced with reinforcement, within dimensional limits set by the code. With forged nozzles, this reinforcement can be eliminated and welding reduced.

Some installations have extremely large agitator nozzles to be able to withdraw the agitator in its entirety to reduce maintenance downtime. This results in very large nozzles that require significant reinforcement and custom design of the flange and bolting. This design also results in additional stresses within the lining system. When a large section of the lining is removed, additional loads are transferred to the remaining section further increasing the compressive loads on the bricks. Also, the nozzle insert has to be reinforced to handle the increased lining stresses. In addition, with such a large section of the arch removed the self-supporting nature of the lining system can be compromised. This design is costly, not only in the large nozzle and the associated blind flange, but also in increased crane capacity, building height and a structure to hold the agitator assemblies, hence the maintenance saving must be compared with the capital costs and lining issues.

AGITATOR MECHANICAL SEAL DESIGN

Typically, the mining industry autoclave designed agitator seal is a flexible rotor, boundary lubricated mechanical seal. It contains several features that have been developed specifically to solve past problems of this difficult application. With design operating temperatures of the autoclaves, now at 270°C and design pressures over 70 bar, there are several unique challenges to sealing these autoclaves. As can be surmised, the integrity of the operation of the autoclave is dependent on the agitator seal system.

Failures have been studied and classified into categories to help determine the features of the design that should be offered for this sealing application. The

following is an explanation of the major design features that are required to successfully seal mining autoclaves in the future.

Stable Operating Platform

One of the primary sources of seal failure on the mining autoclave is the instability of the platform on which the seal must operate. This instability is brought about by a variety of conditions, listed below with explanations, that are the primary concerns regarding seal design and stability of the equipment.

Squareness, Perpendicularity and/or Angularity

Both the seal manufacturer and the equipment manufacturer have largely ignored perpendicularity of the mounting flange. Tight tolerances for perpendicularity are typically not enforced on the mounting flange because it is thought that the seal design could adequately “compensate” for the squareness problems. However, in actual operation, it has been found that, for a long seal life, it is essential to have the mounting flange surface to which the seal attaches be perfectly perpendicular to the centreline of rotation of the shaft.

When the flange mount is not perpendicular to the centreline of shaft rotation, the seal hardware must accommodate a wobble or oscillation for every 360° of shaft rotation. When a point on the flexible rotor is in one position, it may be in the “most forward” position on the sleeve due to the cocked nature of the shaft or flange. Then when that point is moved 180° to the other side of the shaft as the shaft turns, it moves to the “most rearward” position. This wobbling of the rotor flexes all of the components in the seal leading to wear and ultimately premature failure.

As the hardware flexes up and down, the secondary seals must slide or roll to accommodate the movement. With autoclave high pressure differentials across the outer seal, the pressure flexes the secondary seal tight against the shaft so this rolling or sliding motion becomes difficult and very damaging resulting in chaffing the shaft sleeve and tearing, nibbling and extruding the o-ring. Also, due to the angularity that is developed between the flexible rotating face and the sleeve it is necessary to open up the clearance otherwise the parts may actually come in contact and bind against the sleeve.

The seal manufacturer can accommodate large amounts of out-of-squareness. However, as with all things, there is a limit and the simple fact is that if perpendicularity is not held to close tolerances, the expectant seal life is reduced. Often this issue does not manifest itself in an immediate failure of the seal, but will certainly reduce the life of the seal.

Shaft Deflection and/or Runout

In the past, this issue has received far more attention than perpendicularity, since shaft deflection and runout can cause more immediate and identifiable failures that are not as subjected by pressure as perpendicularity related issues. If the shaft is deflected and hits a stationary component of the seal, the cracked stator and galled sleeve can immediately be recognized and subsequently clearances opened to accommodate the radial movement of the shaft.

However, little thought is given to whether the failure was caused by a one-time shaft impact that jars the shaft to one side in the seal area, or by an ongoing deflection. When the centreline of rotation moves to one side, this contributes to an angularity problem. This problem is then added to the angularity problem generated by the previously described out of squareness that exists due to manufacturing tolerances and/or assembly stack-ups.

This kind of deflection can also exist if the centreline of the shaft transcribes a pattern outside of the assembled centreline position. This simply causes more flexing of the flexible components, which in turn increases wear, causing decreased seal life.

Concentricity

For any seal to realize the maximum benefit of clearances that have been designed into a seal, it is vitally important that the seal be centred properly around the shaft during installation. If the seal is installed in such a manner that sets the stationary components closer on one side than the other, then this smaller gap is more easily traversed and damage is more readily accomplished.

Design Features to Accommodate an Unstable Operating Platform

A “flexible” stator as designed by Flowserve is shown in Figure 1 as opposed to the flexible rotor design. The design developed by Flowserve accommodates a one-time perpendicularity problem with the equipment and operation by moving and locking in a “best” fit position. Therefore, if the equipment designer has designed an appropriately rigid shaft, this seal will be immune to perpendicularity problems that arise from manufacturing tolerances and assembly tolerances. In addition, if there are thermal expansions in the equipment that contribute to perpendicularity problems, the flexible stator seal will accommodate these changes.

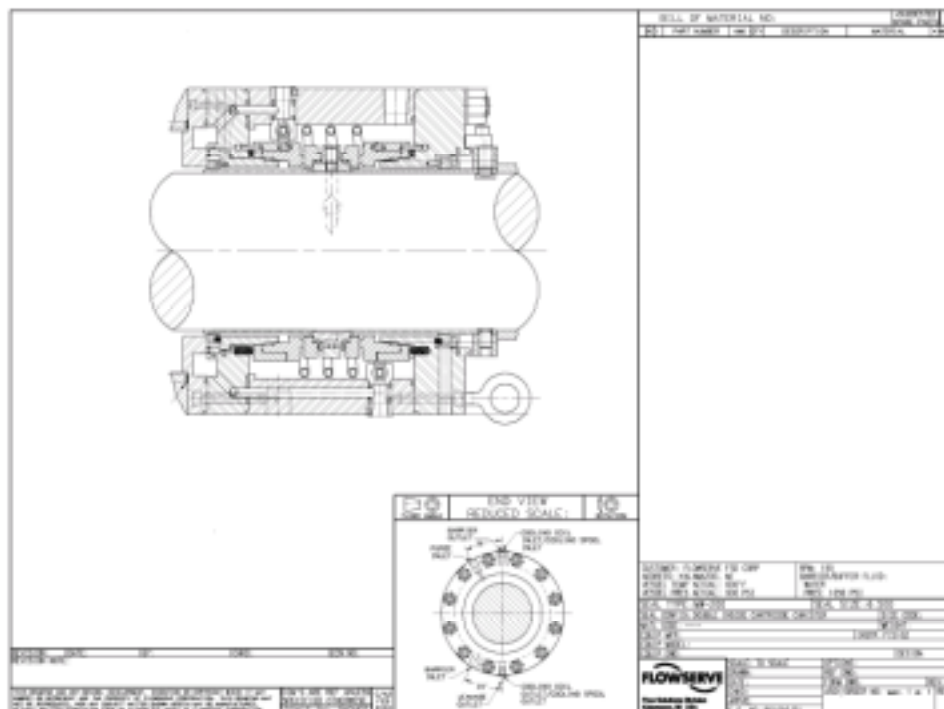


Figure 3- Flowserve Flexible Rotor Double Mechanical Seal

It must be pointed out that even though the flexible stator design eliminates the perpendicularity problem, it is still subject to runout and concentricity issues that may create perpendicularity problems just like a flexible rotor design. Therefore, it is vitally important that the shaft be made rigid enough to minimize radial deflections.

Although seal manufacturers can develop seals that will tolerate some angularity problems, it is vitally important to continually push for a better, more stable equipment platform. As the pressure and temperature designs of autoclave are being pushed higher, all the forces generated by imbalances will lead to even greater wear on the seal components and ultimately result in an even higher frequency of failure. Ultimately, there is no substitute for a square and rigid equipment design to increase seal life.

OPERATION AND RELIABILITY OF THE AUTOCLAVE AGITATOR SEAL BARRIER FLUID SYSTEM

A major cause of failures with agitator seal systems is the barrier fluid quality and the pressure and flow reliability. Most mines have an elaborate and expensive barrier fluid system, complete with at least one back up system.

The mining autoclave seals operate in a high pressure and temperature environment. The autoclave seals operate on top of a vessel that can contain acidic slurries close to 270°C and 60 bar operating pressure. If barrier fluid flow, or pressure, or flow and pressure are lost, the seal has little or no time to survive.

If pressure is lost, the lower seal then loses its lubrication due to gaseous phase vapours being forced between the inner faces at high pressure. Often the lower face can be blown open, unseating the secondary seals, which will then have little or no chance of reseating once pressure is restored.

Usually when pressure is lost, so is flow. With temperatures in the autoclave so high, it takes little time for the heat to soak up into the seal area. Firstly, the o-rings associated with the lower seal rings, typically Kalrez™ o-rings, are lost. They overheat, soften, try to extrude, and take a compression set then the seal never operates properly again as the inboard o-rings have been lost.

Design Features to Accommodate Poor Barrier System Reliability

One of the key features of the new mining seal standard design is that it almost eliminates the need for high pressure cooling. Typical utilization of API Plan 54 (ANSI Plan 7354) by mining autoclave operators requires that all cooling be provided by the barrier fluid: the fluid is sent to the seal cavity where it absorbs a tremendous amount of heat, and then flows through to a cooler where the heat is removed then circulated back through the seal. When flow stops, the seal is almost immediately damaged due to inadequate cooling of the o-rings or loss of pressure.

API Plan 54 Pressurized external barrier fluid reservoir or system supplying clean fluid to the seal chamber. Circulation is by an external pump or pressure system. Reservoir pressure is greater than system pressure being sealed. It is typically used with dual pressurized seals.

With the new mining seal, cooling is accomplished by a thermal dam called a “cooling spool” between the vessel and the seal cartridge. Water flows through this spool and carries away heat that is being conducted through the vessel walls into the seal housings. The water that flows through this spool is at a low pressure and operates independently of the high-pressure barrier fluid system. If barrier fluid flow is lost, this feature allows the lower o-rings to survive for a longer period until the barrier fluid flow is once again flowing.

Another feature of the new mining seal is the addition of a cooling coil inside the seal cavity. This coil assists Plan 54 by removing heat at the seal, so there is not as much thermal strain on the Plan 54 system. And as with the cooling spool, should the flow of barrier fluid be interrupted, the low pressure cooling coil will continue to do its job, saving the seal from being destroyed immediately by heat. The cooling coil will remove heat that is being conducted up the shaft and into the seal housing.

To assist in the removal of heat from the shaft, the seal sleeve has two opposing tangential holes that lead to an open cavity between the shaft and the sleeve. Motion of the shaft moves the barrier fluid in one hole and out of the other, circulating fluid over the shaft and under the seal. This also helps moderate temperatures on the o-rings to combat o-ring damage when barrier fluid flow is lost.

The new mining seal is designed with pressure reversal capabilities on the inboard faces. With a loss of barrier fluid pressure, the balance diameter shifts to keep the faces closed. Naturally it is not desirable for the faces to run in this configuration, but for a short time the seal is expected to survive due to the cooling spool, cooling coils and balance diameter shift.

An additional feature in this seal is the inboard bushing and flush port. Water can be injected into the vessel through this bushing. Even though the flow rate is low (and pressure is higher than vessel operating pressure), some cooling is accomplished with the flush. This flush also provides a barrier between the vessel and seal faces so there is less likelihood that the corrosive atmosphere of the vessel will affect the face materials. Also very important is the fact that this water held in the bushing area will help keep the faces wet during a barrier fluid pressure outage. Instead of blowing gas between the inboard faces, the water in the bushing area will move up between the faces and help keep them wet and survive.

It is important to understand that while a seal will tolerate operational instabilities, there is still no substitute for properly designed and smooth operating equipment. It is important to continue to carry the word forth that seals will simply last longer and cost less to operate and maintain if they are run on equipment with very good squareness and runout characteristics. The seals will tolerate a certain amount of abuse, but for the seals to eventually reliably run for at least one year at every installation will depend on the quality of the installed machine and the reliability of the barrier fluid system it operates in.

Design features have been developed due to necessity rather than want from seal manufacturers and should not be an excuse for sloppy operations, design, or manufacturing. It is important to always push for a better environment for the seals.

In summation, the features to look for in the seal design are:

- flexible stator design, which tolerates squareness issues better than flexible rotor designs;
- low pressure cooling in the spool and coils to help seal survive longer during normal operation, and also to make the seal much more survivable in barrier fluid upset conditions;
- the cooling under the sleeve to help the o-rings survive by eliminating hot spots on the shaft;
- a seal canister with a flat face that has complete metal surface contact out beyond the housing bolting to control rocking and squareness problems.

SEVERE SERVICE VALVES

In pressure acid leach and pressure oxidation, the term “severe service” has become synonymous with valves used in these plants. This is due to the high operating temperature, pressure, scaling slurry duty application of the valves. Commonly, the valves are a two-piece design floating ball valve. Materials of construction are selected based on the service conditions and both the ball and seat are typically coated with a plasma sprayed ceramic coating incorporating a bond layer if required to increase the bond strength of the coating.

Historically, there have been only three main suppliers of these valves to the mining industry: Dresser (Cooper TXT), Mogas and Velan (Securamax). In terms of population, the most used is Securamax (Goldstrike, Lihir, Cawse, Murrin Murrin), followed by Dresser (Bulong) and then Mogas. The Securamax valve has been the most popular in Australia.

The valves are similar in that they are all a floating ball design. Securamax and Mogas are a forged two-piece design while Cooper’s is cast two-piece construction. Though all three valves are bi-directional, however, Mogas and Securamax’s have a preferred flow sealing direction. In terms of strength, all three valves have strong drive train design and very good coating technology, which sets them apart from other manufacturers for this particular duty. Because of this and because of the reputation of these three valves, inroads by other manufacturers for supplying valves for this application are difficult.

Design Fundamentals

Since the start-up of the PAL plants in Australia, the basic design of these valves has not changed significantly; however, some key valve requirements have emerged.

Strength

The drive train, including the actuator, must be capable of three to five times the valve torque requirements on maximum differential pressure on water. Consequently, other drive components must each be considered individually and in relation to other components in terms of the valve torque requirement. The key design considerations are as follows:

- Ball drive slot: As strong as the stem.
- Stem: Strongest component of the drive assembly.
- Drive adaptor: Design to fail, if the double-D or square connection to the actuator is required.
- Mounting bolts and dowels: To exceed the maximum torque requirements by a safe margin.
- Keys and keyways: Designed as the failure point if all else fails, i.e., failure of the key prevents any other damage to the drive train.

Where valve manufacturers have not considered the above, early failures and damage to the drive slot and/or failure of the valve to operate have resulted.

A large number of design factors affect valve torques due to the following:

- Slurry between the ball and the seat due to improper seat to ball loading by the load ring or frequent flow reversal;
- Scale build up absorbing clearances either due to infrequent valve operation or stagnant flow conditions for extended periods of time;
- Stem leakage and hardening of the process due to improper packing installation, lack of live loading, or infrequent examination and in-situ correction of packing torque;
- Physical obstruction inside the valve (vessel lining material, such as brick, hard scale, etc.);
- Coating loss due to either mechanical factors or chemical attack, thereby increasing torque requirements.

Bi-Directional Sealing Versus Fixed Seats

The Cooper's valve is a true bi-directional sealing valve and therefore has some advantages over Mogas and Securamax, though both are considered bi-directional, however, they have a preferred sealing direction. In some critical cases with a primary sealing direction, there should be a fixed seat on the downstream side. Examples of this are the primary (inboard) block valve on the autoclave discharge line or the outboard block valve on the autoclave feed pump. Experience has shown that in these critical duties, the seats must be fixed or leakage can occur behind the seat

leading to body erosion. The seat can be fixed by bolting, welding, incorporating the seat as part of the body seal ring or it can be held in place by a spigot on the body or a load ring on the seat. Additionally, a gasket or seal around the outside of the seat is also required.

For some pump isolation applications, bi-directional sealing is desirable and these valves should be identified early in the design stage. If not picked up in the development stage of a project it may be picked up during a HAZOP. Conversely, taking the opposite approach and specifying all the valves to be the same can create interchangeability and/or serviceability issues on a population of valves where fixed seats are specified/applied.

Materials of Construction and Coatings

Although material test work by clients and engineers alike take place to select materials that are compatible with the process, often, too much reliance is then placed on coatings without considering other factors. Basically, the base material of the body, ball, seats and stem must be able to resist corrosion without coatings.

Coatings by themselves should not be relied upon as the corrosion barrier. The reason is that no coating or bonded coating is 100% fail/corrosion-proof since coatings are porous. Coating porosity of less than 3% on the ball and seat is considered good, however, it cannot be measure without destroying the component. To ensure that the coatings are within specifications, attaching a coupon during the spraying can be done to give a good indication of porosity.

If the coatings are not properly applied, crevice corrosion can occur under the coating leading to premature failure. Knowing this, it is questionable why the components are coated in the first place. However, the simple reason for this is that the coatings provide a low friction wear resistant surface, which can be lapped to produce close tolerance seal prior to installation. If the substrate is not resistant enough to the corrosive medium (process) then it makes no difference how dense or corrosion resistant the coating is because failure will occur at the interface layer leading to premature coating failure due to spalling.

Essentially, the better the coating (density and adhesion [bond strength]) is on a suitable substrate, the longer the coating will stay on improving the sealing and reducing the torque requirements of the valve. When the coating fails, then this can lead to substrate damage resulting in an increased lifetime cycle cost of the valve.

The bond coat is there to provide a layer between the substrate and top coat to increase bond strength, compensate for differential thermal expansion and reduce the likelihood of permeability leading to spalling. It is important to clarify that coatings fail due to a breakdown of the bond or attack by the medium (process) on the coating; they typically do not wear thinner.

Typical coatings that have been used with varying degrees of success are the following:

- TiO₂ and CrO₂ mixes;

- Nanostructured coatings (first developed by Mogas);
- Ta bond with CrO₂;
- Inconnel 625 bond with CrO₂;
- Other proprietary materials and bond coating combinations.

Each component of the valve should be specified in relation to, not only its compatibility to the process, but also to its operating environment. For example, the ball can be specified in a different grade of material than the stem due to coating and load requirements. Given the torque requirements of the valves in autoclave service, it is apparent that the stem needs to be in a stronger material than the ball itself while still being compatible with the process. As an example, Ti Gr 5 is one of the strongest grades of titanium that may be chosen for the stem material due to its strength and availability, while the body may be specified in Ti Gr 12 and the ball may be TiO₂ coated Ti Gr 3. Obviously, the available choices of base material and coatings are many and consultation with manufacturers and operations should be sought. Table I lists the common grades of titanium and their mechanical properties.

Table I - ASTM Titanium Grades, Composition and Mechanical Properties

Grade ASTM	Composition	Yield Strength (ksi)	Yield Stress (ksi)	Modulus of Elasticity (psi-106)
1	Pure Ti	35	25	14.9
2	Pure Ti	50	40	14.9
3	Pure Ti	65	55	14.9
4	Pure Ti	80	70	15
5	Ti, 6% Al, 4% V	130	120	16.4
7	Ti, 0.15% Pd	50	40	14.9
9	Ti, 3% Al, 2.5% V	90	70	13.1
11	Ti, 0.15% Pd	35	25	14.9
12	Ti, 0.3% Mo, 0.8% Ni	70	50	14.9
13	Ti, 0.5% Ni, 0.05% Ru	40	25	14.9
14	Ti, 0.5% Ni, 0.05% Ru	60	40	14.9
15	Ti, 0.5% Ni, 0.05% Ru	70	55	14.9
16	Ti, 0.05% Pd	50	40	14.9
17	Ti, 0.05% Pd	35	25	14.9
18	Ti, 3% Al, 2.5% V, 0.05% Pd	90	70	15.3
19	Ti, 3% Al, 8% V, 6% Cr, 4% Zr, 4% Mo	115	110	14.9
20	Ti, 3% Al, 8% V, 6% Cr, 4% Zr, 4% Mo, 0.05% Pd	115	110	14.9
21	Ti, 15% Mo, 2.7% Nb, 3% Al, 0.25% Si	115	110	14.9
23	Ti, 6% Al, 4% V	120	110	16.3
24	Ti, 6% Al, 4% V, 0.05% Pd	130	120	16.4
25	Ti, 6% Al, 4% V, 0.05% Ni, 0.05% Pd	130	120	16.4
26	Ti, 0.1% Ru	50	40	14.9
27	Ti, 0.1% Ru	35	25	14.9
28	Ti, 3% Al, 2.5% V, 0.1% R	90	70	13.1
29	Ti, 6% Al, 4% V, 0.1% Ru	120	110	16.3

Maintenance of Valves

There are a number of philosophies currently practiced in the industry regarding servicing these critical valves, including:

- Run to failure then remove and repair;
- Remove and repair on a fixed term with the aim to increase the time between fixed terms;
- Monitor by various methods to identify the valves relative “health” and repair at the next opportunity.

During the start-up of autoclave plants, “run to failure” is quite common as the plants tend to have a lot of downtime not necessarily attributed to the valves but that gives plenty of opportunity for maintenance to remove, replace and repair. Until long run times can be established by operations, this is the norm for maintenance. Ultimately the best practice would be a combination of “fixed term” and external monitoring system. This would reduce repair costs with the aim of increasing the fixed term frequency rate. This can be accomplished by improving coatings and overlays on key components by analyzing the valves when they are removed.

To extend the service life even further, specialized service providers can be called upon to instigate a valve monitoring program. The program will include the following:

- Acoustic monitoring, which can detect internal leakage and, using historical references and/or empirical data from the vendor, co-relate leakage rates to give an indication of the state/condition of the valve.
- Monitoring of valve movement.
- Establishing valve movement to flush and prevent crevice corrosion.
- Repairing procedures to minimize coating attack.
- Invasive maintenance on glands and actuators.

Monitoring of the valve is typically done while in the closed position where there is a known differential acting over the valves sealing surface. Readings are taken and over a period of time, and a history is recorded. Once the readings are processed, they are compared to the trends of the valve. This database, once developed, then uses these and other records (type of valve [size, class], frequency of operation, differential pressure) to assist maintenance personal to plan for valve changeouts aid in sparing levels. The main benefit of a valve monitoring program is the ability to ascertain the valve condition without having to remove it for visual inspection.

Other than acoustic monitoring another diagnostic tool being used is a digitally-controlled partial stroking device. The concept involves sending a DCS signal which partially opens the valve off its seat and then closes it. If done on a routine basis it not only breaks up any build up of product between the ball and the seat, but it also allows a series of readings to be taken by the stroking device. The readings are then analyzed by data acquisition software and a report is generated. The

readings cover torque, packing friction, actuator air supply and the general condition of the ancillaries.

SLURRY LETDOWN AND VENT ANGLE VALVES

Slurry letdown plugs and chokes have historically caused autoclave operators many problems with trim, plug and actuator failures. The designs have changed markedly over the years from the early days of the gold industry from fixed diameter chokes to the now more commonplace flared (diverging) designs. The application of higher temperature and pressure autoclaving brought on by the nickel laterite industry in Australia has necessitated the advancement in the design and technology of these valves.

Significant progress has been made in extending choke service life. The majority of the development and advancement in technology has been attributed to the effort of Caldera Engineering who, over recent years, have spent time and effort in research and development to address specific problems with these valves. As an example of the great gains that have been made in recent years, a comprehensive redesign of the choke has resulted in one mine consistently seeing a four-fold increase in the service life of the choke. The longer service life of the redesigned choke has been attributed to several features, including the segmented design, the external ceramic sleeve near the choke outlet, the ceramic status alarm and the greater length, all of which will be described below.

In 1998, the pressure acid leach plants in Australia (Murrin Murrin, Cawse and Bulong) came into production. These plants have more stages of letdown and higher pressure and temperature drops between flash stages than gold POX plants. From the beginning, the PAL plants in Australia faced catastrophic failures of ceramic seats and plugs. It was evident then that the new plants were showing limitations in the traditional valve designs. Since that time Caldera Engineering have been at the forefront of development of the “new” generation of letdown valves. These valves have been designed to solve specific problems that were exemplified by the PAL industry. The successes in the PAL industry are now reaping rewards in the POX industry as well.

Ceramic Selection

Ceramic plugs and chokes are required for slurry letdown and in some vent applications where the differential pressure results in critical flow. In the early days of development, a few mines attempted to operate without ceramic plugs and chokes when they consumed their normal spares inventory and resorted to running with metal plugs and/or chokes. The life of metal components in these situations has reportedly been measured in hours and days.

Hexoloy SA has been the standard material for ceramic angle valves because of its outstanding corrosion and erosion resistance. However, parts made using Hexoloy SA ceramic can fail due to fractures and cracking; in essence the higher the erosion/corrosion resistance the lower the fracture toughness. Timing of the failures can be unpredictable. For this reason, mines have experimented with other ceramic

materials. One Nevada mine and one PNG mine commissioned and ran their plants with Cercom PAD Type B SiC plug heads. The Cercom ceramic is very similar to Hexoloy SA in many areas. Due to what seemed to be a higher plug failure rate, along with price and delivery issues, both mines later standardized on Hexoloy SA. Other mines that experimented with materials generally returned to Hexoloy SA because the other ceramics either wore out too quickly or fractured more frequently than Hexoloy SA. In 1998, a grade of Hexoloy SA, called Enhanced Hexoloy SA, became commercially available. Enhanced Hexoloy SA has a higher Weibull modulus and greater average flexural strength than Hexoloy SA.

In an ongoing effort to improve plug and choke performance, various ceramics have been tested, including silicon carbides, silicon nitrides, zirconium oxides, aluminium oxides and variations of these types. The following is a list of ceramics that have been evaluated.

Silicon Carbides

Carborundum Enhanced Hexoloy SA (SiC)
 Carborundum Hexoloy SA (SiC)
 Carborundum Hexoloy ST (SiC-TiB₂)
 Cercom PAD SiC Type B
 Dow Corning SiC
 Alanx ceramic/metal composites (CMC)

Silicon Nitrides

Ceradyne Si₃N₄ (Ceralloy 147)
 Kyocera Si₃N₄
 Technologie GmbH SiN₄ (GPSN)

Zirconium Oxides

CoorsTek TTZ (MgO ZrO₂)
 Carpenter MgO ZrO₂ (MS grade)
 Zircoa Zycron L (MgO ZrO₂)

Aluminium Oxides

Alpha Group Al₂O₃
 Diamonite Al₂O₃
 DuPont Research Al₂O₃

Ceramic Mixtures

CoorsTek Super P (SiC whisker-reinforced Si₃N₄)
 Sialon
 CoorsTek ZTA (zirconia-toughened aluminium oxide)
 Alanx ceramic/metal composites (CMC)

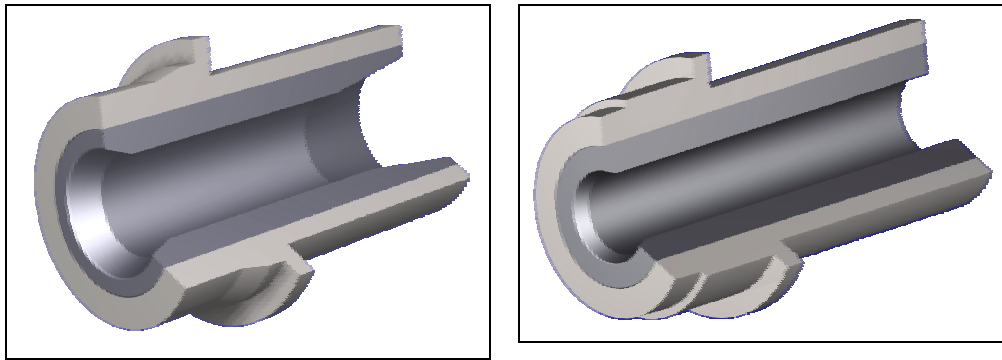
Subsequent evaluations have not identified a ceramic that would be considered an improvement compared to Hexoloy SA. Each of these ceramics was lacking in one or more of the following properties: erosion resistance, corrosion resistance, thermal shock tolerance, flexural strength, fracture toughness or commercial availability. Hence, Hexoloy SA remains the ceramic of choice for most slurry letdown and vent applications. Nearly every site uses this ceramic for slurry letdown valves and it is the most common ceramic in vent valves.

It goes without saying that even though the ceramics have outstanding properties in terms of corrosion and wear resistance, they are prone to catastrophic failure due to their inherent brittleness or lack of fracture “toughness.” Typically it only has one failure mode – catastrophic – which can be the result of residual tensile stress, impact with a hard dense object, or large thermal gradient. If long cycle times

are the goal with the ceramic components, it is evident that the onus is mutually shared between the end-user (the mine) and the manufacturer. Manufacturers will be responsible to lower stress concentrations in the seat and plug designs, thereby increasing the reliability of the valve and its components, and it is the responsibility of the process design engineers and plant operators to do everything at their disposal so as not to cause any undue stress on the components (i.e., thermal shock).

Choke Design

Traditionally the ceramic chokes were a one-piece design similar to that shown below. The design features a one-piece ceramic insert or liner and a shrink-fitted metal housing. Chokes with one-piece ceramic inserts and one-piece metal housings represented the best designs that were available from about 1994 until 1999. Typical diverging and straight choke designs are shown below



Pictures Courtesy of Caldera Engineering

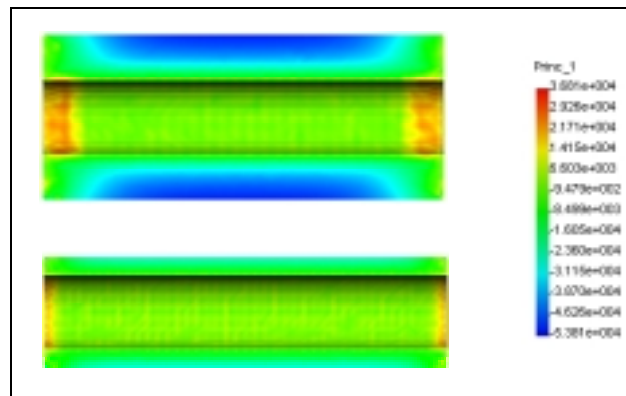
POX mine operators have struggled with slurry letdown choke failures for many years, but it had reached a stage where things became acceptable. In general, the advancement in the valve designs was somewhat complacent. When the PAL nickel mine autoclaves in Western Australia came into being they were plagued by choke failures. In these mines, ceramic inserts experienced serious fractures. Many of these failures occurred after the parts had been in service for only a few weeks. It was essential to determine what had suddenly changed, and why were the PAL nickel plants suffering so badly with valve failures compared to the gold POX plants.

Firstly, the nickel mines used chokes that were considerably longer than those previously used in autoclaves. Additionally, some chokes were designed with an ID that flared outward at the choke exit (diverging nozzle). This required a ceramic insert to have an ID small enough to control flow at the top of the choke and a ceramic OD large enough to provide adequate wall thickness as the choke flared toward its outlet. As a result of longer length and larger outer diameters, the nickel mine ceramic inserts had thicker walls and greater mass than those previously used in the autoclaving industry.

In 1999, researchers from Saint-Gobain Advanced Ceramics studied several choke failures from slurry letdown valves in nickel mines in Western Australia. After

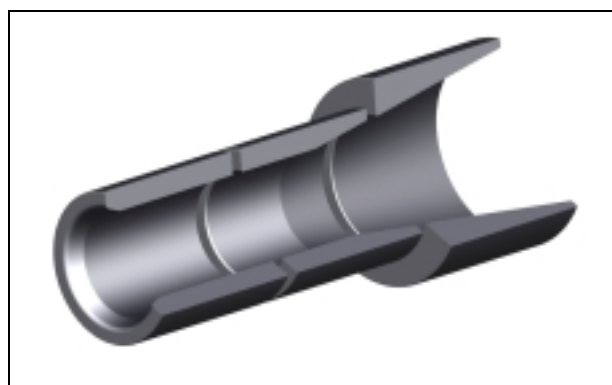
examining these choke failures, the researchers recommended designing chokes with thinner and lighter ceramic pieces.

Finite element analysis confirmed that in conditions of cold shock, the thicker ceramic cylinders develop higher tensile stress than those with thinner walls. In the example shown below, the tensile stress in the thin-walled cylinder at the bottom of the figure was reduced by one-third as compared with the thick-walled cylinder at the top of the figure.



Lower Tensile Stress in Thinner Ceramic Cylinder

Acting on the recommendation from Saint-Gobain, Caldera Engineering developed segmented choke designs for ceramic angle valves as shown below. The segmented choke divides the overall length of the ceramic insert into two or more shorter pieces. By using multiple pieces, it is possible to design larger ceramic ODs near the flared outlet of the choke and smaller ODs near the narrower top of the choke, resulting in a significant reduction in the ceramic mass. The service life of several representative chokes was lengthened by a factor of more than four.



Slurry Letdown Choke Ceramic Segments

With segmented choke designs, ceramics are of a simple shape and tensile stresses in the ceramics are low. Segmented chokes can be repaired by selectively replacing the failed components.

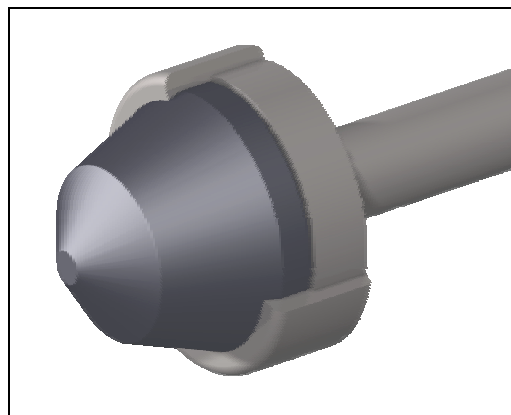
The longer service life of the redesigned chokes has been attributed to the segmented design. The main attribute of the segmented design is that stress is

significantly reduced on the ceramics. Chokes are now removed due to metal wear on the holder, ceramic cracks or size changes. The new segmented choke designs by Caldera Engineering have yet to cause a shutdown in an HPAL plant due to catastrophic failure.

Plug Design

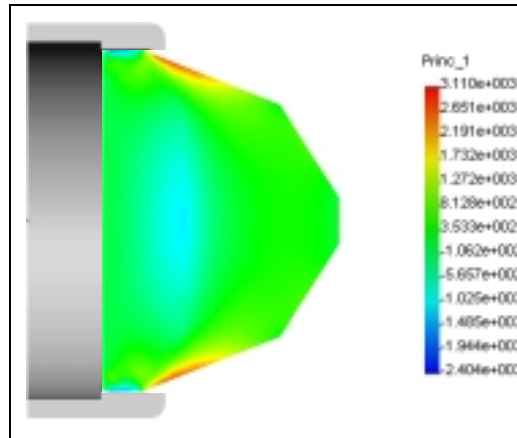
Recently, Caldera Engineering have also advanced the design of the valve plug. Similar to the choke assemblies, failures have occurred due to stress concentrations in the plugs.

The figure below depicts the conventional ceramic angle valve plug design. This design consists of a ceramic plug head and a metal retainer, base and stem. The metal retainer is placed around the ceramic head and is fixed into position using a shrink-fit method where the metal retainer is heated and then placed around the ceramic head. When the metal retainer cools, an interference fit secures the head. The retainer is then welded to the base, which was previously welded to the stem. This design could be called a “one-piece” design. This design was considered to be the best available from about 1995 until 1999.



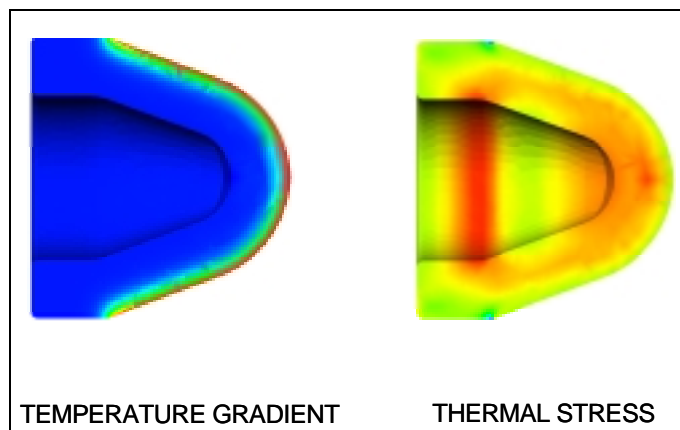
Typical Angle Valve Plug Design Courtesy of Caldera Engineering

Until the HPAL plants in Australia came on-stream, not much work had been required in developing new plug designs. However, similar to the chokes, catastrophic failures started occurring more often than in POX applications. Again, this was primarily due to the larger temperature and pressure drops required in HPAL, particularly in autoclave vents. In 1997, Caldera Engineering started performing finite element analyses (FEA) on existing ceramic angle valve plug designs. It was determined that the shrink-fit retainer creates a residual tensile stress on the surface of the head, extending to the inner portion of the ceramic. As ceramics are weaker in tension than compression, it became apparent that residual tensile stresses in the ceramic head should be avoided. Residual stresses for a representative plug design are shown in the figure below.



Residual Tensile Stress in Plug Head Courtesy of Caldera Engineering

Temperature swings that are part of typical autoclave start-up and shutdown procedures can induce extensive stress in the ceramic head resulting in failure. This tended to be the case in the early days of HPAL operations. Rapid downward temperature swings, referred to as “cold shock,” add tensile stress to that stress already in the head. As the HPAL operations have higher pressure and temperature drops, it is easy to see why the operations were experiencing premature failures compared to the established run times of plug heads in the POX operations. A qualitative view of the stress caused by cold shock is shown below. The red colour at the plug head surface illustrates this tensile stress.



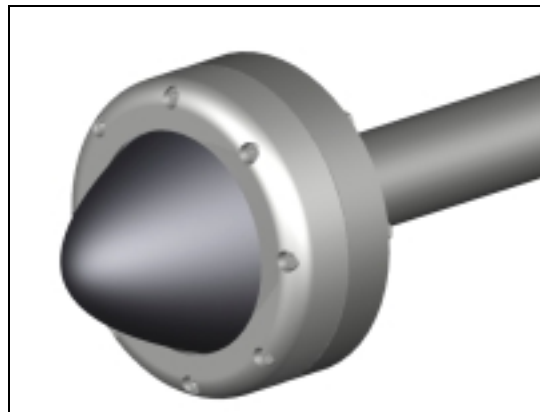
Thermal Gradient and Resulting Stress due to Cold Shock
Courtesy of Caldera Engineering

Understanding these residual tensile stresses was considered important in light of the fact that the autoclave industry has struggled with many plug failures. A typical failure is shown below. Once a failure occurs, the ceramic plug fractures and part of the head gets separated from the valve plug. When this occurs it is usually not possible for the autoclave operators to maintain adequate flow control, resulting in a shutdown.



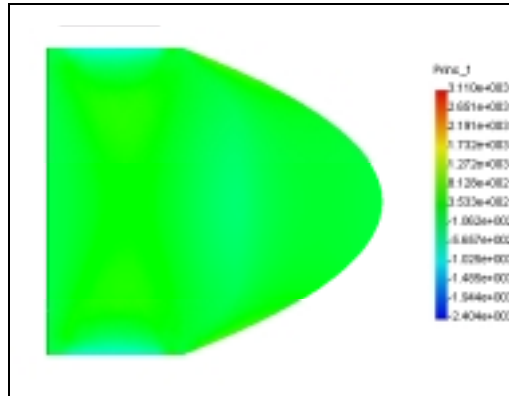
Typical Failure of Flash Letdown Plug Photo Courtesy of Caldera Engineering

In 1999, Caldera engineers designed a plug with a lower level of residual tensile stress in the ceramic plug head. This was achieved by shrink-fitting a thin metal band around the ceramic head. Bolts were used to attach the head and band assembly to the base. In addition to achieving lower residual stress, the new plugs also allow replacement of the head without completely disassembling the valve and installing a new plug. At the same time, Caldera also designed plugs with a parabolic shape (without abrupt transitions between sections of differing angles), further reducing stress. The combination of their design is shown below.



Banded Plug Design with Parabolic Ceramic Head Courtesy of Caldera Engineering

The result of the new designed plug is that the maximum compression is in the middle of the plug's shrink-fit surface, compared to the previous one-piece designs, which apply large compressive loads near the transition between the major OD and the tapered portions of the head. Finite element analysis suggests that a reduction in residual tensile stress of about 75% can be achieved by using a banded, parabolic head instead of a one-piece plug design with an angled head. A representative finite element analysis is shown below.



Reduced Residual Tensile Stress in Plug Head using Banded Design: Caldera Engineering

Status Alarm

As stated previously, when a ceramic component fails, it fails catastrophically, usually with little or no warning. With chokes, failure is sometimes not detectable during normal operations. This can lead to vessel and vent header damage if not detected early. Broken plugs can alter the flow path resulting in abnormal wear patterns, which result in damage. To solve this problem, Caldera Engineering have developed ceramic status alarms that can transmit signals to plant operators when failures occur in the ceramic heads or chokes.

The status alarm is relatively simple in concept. It consists of a pressurized titanium tube in the housing that wraps around the lower portion of the choke. The tube is placed in the choke such that a failure of the choke would sever the tube, resulting in a pressure loss in the tube. A pressure switch mounted on the tube outside the choke detects the pressure loss and signals the event to the control room or other location.



Choke with Ceramic Status Alarm Courtesy Caldera Engineering

Tight Shutoff Plugs and Chokes for Vent Applications

Before the PAL nickel industry in Australia got started, there was no demand for a tight shutoff ceramic valve. However, this is one area where there is a difference between POX and PAL. POX autoclaves need to continuously vent, not only due to the build up of non-condensable gases, but also to release heat energy due to the exothermic reactions. In PAL autoclaves for the nickel laterite industry, the reactions are not highly exothermic and the only appreciable heat that is generated is that due to the heat of dilution of acid and, therefore, significant amounts of live steam are used to attain the higher operating temperatures. Consequently, these autoclaves need to “conserve” as much energy as possible and literally cannot afford to vent.

Most laterites contain some carbonates and so, eventually, there will be a build up of non-condensable gases that has to be released, but most of the time venting is not required or desired. Initially the laterite mines in Australia used Hexoloy choke and plugs similar in design to the slurry letdown valves, as it was the only material and valve type that can possibly do the duty (large pressure drop - critical flow). However, because the Hexoloy chokes and plugs would crush each other if closed tightly, the valves could never be “fully” closed. Given the high operating pressure (4800 kPa to 5600 kPa), even the smallest fraction of an opening resulted in significant steam losses. Achieving the tight shut necessitated the use of materials other than Hexoloy or a redesigned valve.

Various materials were investigated in an attempt to get a tight shutoff. The material had to still be corrosion and erosion resistant and be able to handle the mechanical stresses derived from tight shutoff. Various materials were tested: stainless steel plug head with a Stellite overlay, tungsten carbide, titanium with a variety of overlays, and different types of ceramics, such as PSZ, without success.

In mid-2000, Caldera redesigned the plug and choke for vent applications with a tight shutoff. Essentially the ceramic cone on the plug and the liner on the choke are designed to withstand the wear associated with throttling control. Additional ceramic surfaces on the plug and choke holder seal against each other and achieve tight shutoff when the valve closes. The plug base can flex to some degree, helping the seal surfaces to achieve a uniform loading during contact.

The new design has been in use since November 2001 in four autoclaves in Australia and has also been in use since May 2002 in a POX plant in Nevada. All are operating successfully.

PAL Acid Injection System

One major technical hurdle that was necessitated by the PAL has been the acid injection system. The nickel laterite plants in Western Australia (Bulong, Murrin Murrin and Cawse) were designed by different engineering companies, and hence the acid injection system designs were quite different. Due to unforeseen design errors (whether it be due to the process or mechanical failures), Cawse and Murrin Murrin both had to make significant modifications soon after start-up. The acid injection system that has become the standard that others have copied or relied upon is the acid

injection system designed by SNC-Lavalin for the Bulong Nickel Mine. A simplified schematic of the injection system is shown in Figure 4.

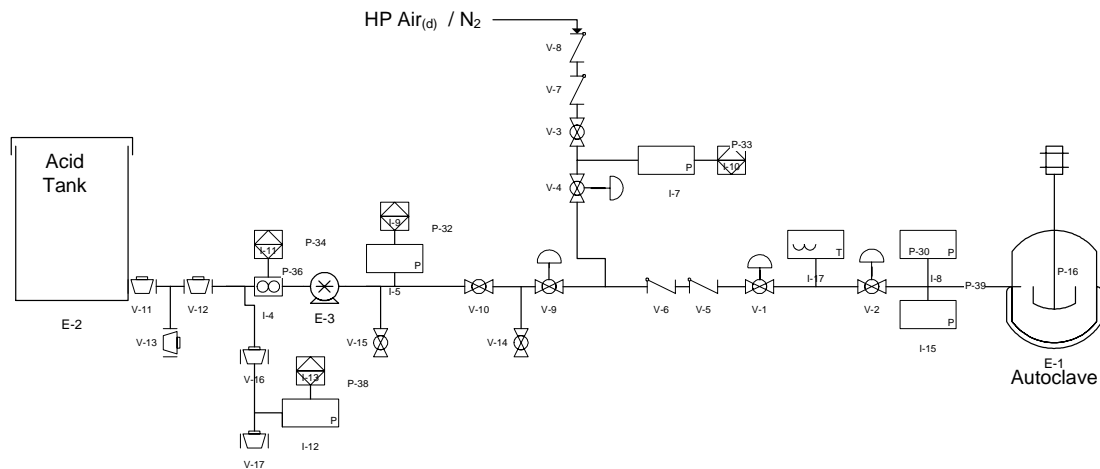


Figure 4 - Acid Injection System

The features include double isolation at the autoclave with double non-return valves, high pressure gas acid purge system, and pressure and temperature alarms in case of valve or lance failure. The spool piece between the two isolation block valves is tantalum lined Alloy 20[®] and the acid lance is tantalum lined titanium.

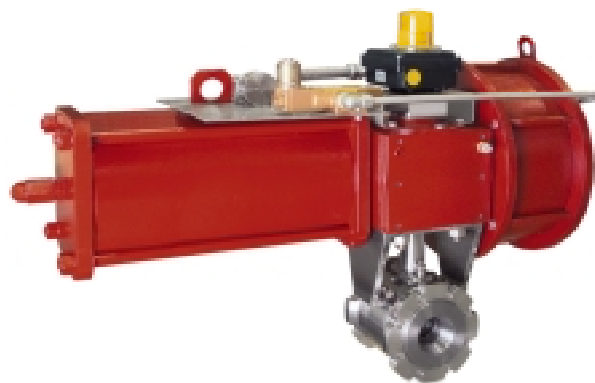
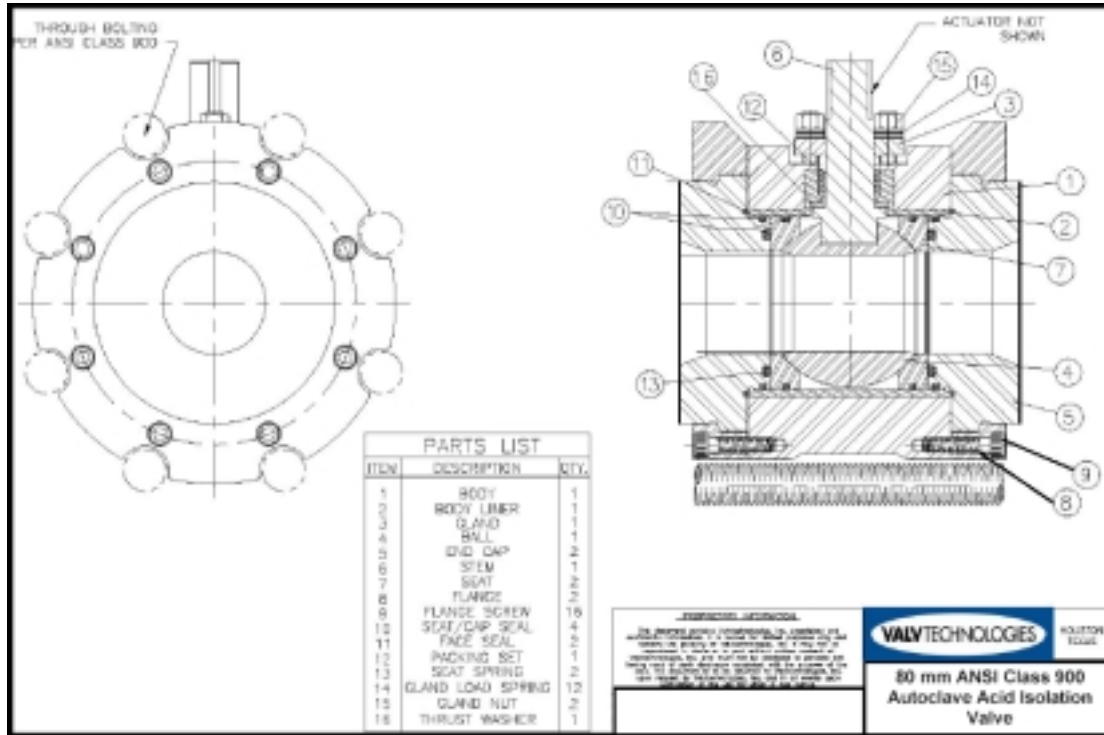
The acid injection system posed many technical design challenges regarding the acid lance, purge system and acid isolation valves, that had not been previously designed or applied in any operation. Corrosion and operating safety of the acid isolation valves were paramount. One of the main technical hurdles to first overcome was to find a material that could withstand the arduous process conditions. Under normal operating conditions, relatively “cold” 98% acid flows through the injection system into the autoclave. However, the material has to cater for when the autoclave is boxed in at temperature and pressure (270°C and 60 gpl acid).

The autoclaves are titanium lined (typically Ti Gr 11, 17, 1), which is sufficient for the process at temperature with residual free acid levels. However, the conditions that may be present for the acid isolation valves and the acid lance are more extreme. The material has to be corrosion resistant if the acid purge system fails and the remaining acid in the dip tube reacts with the process in the acid lance. This condition can cause the temperature inside the lance and the closed face of the isolation valve to exceed 300°C and vary from 98% acid to 60 gpl.

Autoclave Acid Isolation Valves

Many materials were considered for this duty, including gold- and platinum lined/coated components, however, they were too expensive and, more importantly, too weak to handle the valve seat loadings. The coating would fail during valve stroking and cause the valve to fail. The material that was chosen for the acid isolation valves was a tantalum alloy: tantalum with 2.5% tungsten (UNS R05252). Tantalum alloys exhibit excellent corrosion resistance for concentrated acid and dilute acid at a varied range of temperatures.

The original acid valve was designed and developed by Valvtechnologies, Inc. of Houston, Texas with input from SNC-Lavalin while detailing the PAL circuit for Bulong Nickel. The valve is depicted below. The valves have an Alloy 20[®] body that is fully lined with tantalum. Due to the limited strength of tantalum, the body is a wafer design and the valve seals bi-directionally. Today it remains as the only valve that has performed extremely well in this application.



In operation, the valve has showed some marked differences in performance while in service. Bulong's acid injection system is side entry while that of one of the other operations is top entry. In operation, the top entry valves suffered from ball surface corrosion, while similar valves at Bulong had not. Superheated fuming sulphuric acid can corrode the ball surface and seat if the purge system fails in the top entry design; this is because the shutoff valve is in the vapour space rather than in the slurry space, as with Bulong.

Valvetechnologies, together with engineers at the other operation, have since improved the Bulong designed valve to address corrosion and other issues, as follows:

- The stem shaft no longer has plasma coating for galling prevention. Instead, a Teflon[®] thrust washer with an integral stem sleeve is used to avoid tantalum-to-tantalum contact. This has prevented surface corrosion and the resulting packing leaks.
- Sizes on the o-ring grooves were adjusted based on DuPont's recommendation in order to prevent permanent o-ring deformation due to excessive loading. This also resulted in lowered torque due to lesser load of large face o-rings, which create the constant “spring” load on the seats.
- The packing was changed from Chevron type to braided rope for improved radial wear and decreased packing leaks.
- End cap retainers now have bolting guides to aid the field installation of a wafer type valve.
- Ball coatings have been changed from Cr₃O₄ to TiO₂ for increased corrosion and wear resistance.
- Packing gland followers were changed from one piece to a two-piece design in order to make the gland less susceptible to misalignment. The material was changed from 316SS to Inconel X750 to allow for a higher load without deflection.
- The mounting hardware material was changed to stainless steel to prevent corrosion if a packing leak does occur.
- After redesigning the packing, set loading and stem coating the high loads ceased to be a problem.

Acid Injection Lance

The original acid injection lance was designed by SNC-Lavalin in collaboration with AstroCosmos Metallurgical. The design is quite novel as it was the first of its kind that had been built.

It not only had to be corrosion resistant for the arduous duty but also strong enough to withstand the forces of agitation. At PAL operating temperatures (in excess of 250°C), tantalum loses a lot of its mechanical strength and, therefore, the lance could not be simply made of solid tantalum. As a result, a double walled titanium, fully tantalum lined acid lance was constructed. The titanium was chosen not only for its strength but also for its corrosion resistance should there be a failure of the tantalum lining. It was constructed as a double walled pipe to, not only give the lance strength, but also allow for detection if the tantalum lining failed, essentially breach detection. As tantalum cannot be welded to titanium (other than silver brazing), the lance had to be constructed in such a manner as not to expose the titanium to concentrated acid should there be a breach and, if there was a breach, that the breach could be detected and the acid injection system be shut down. A simplified drawing of the acid lance is shown in Figure 5.

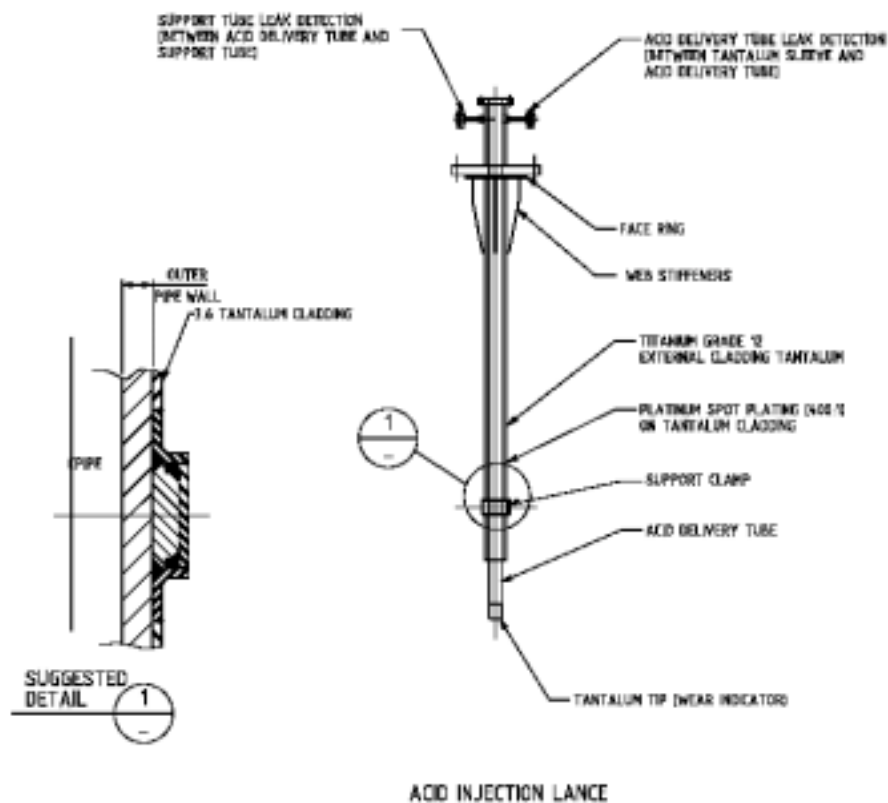


Figure 5

Since the first acid lances that were designed and built for Bulong Nickel others have been built and installed at two other pressure leach operations. Since the original Bulong design, the lance has been improved by AstroCosmos as manufacturing and welding techniques included the development of explosion clad tantalum-to-titanium techniques.

CONCLUSION

Various pressure hydrometallurgical processes are now widely used on a range of ores and concentrates. Operating conditions range up to 260°C and 5000 kPa, and higher designs are in progress for higher temperatures.

Equipment has been developed and has commercially been proven to handle these conditions. Autoclaves are either titanium clad or lead and brick lined. Various alloys, titanium or super duplex stainless steels are used for the internal structures, dip pipes and other connecting pipes and valves. Suitable agitator impellers and seal systems have been designed for a wide range of conditions and duties and new installations are now generally trouble-free in this respect.

Although there is room for further improvement, multiple stage slurry pressure letdown and heat recovery systems have been developed to the point that they can be installed with confidence. This is also true of the autoclave feed pumps, which are currently operating at temperatures 200°C.

As more complex ores continue to be exploited and environmental constraints make pyrometallurgical treatment less attractive, high temperature hydrometallurgical processing is bound to become more prevalent.

ACKNOWLEDGMENTS

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