For example, MBE (molecular beam epitaxy) processes are carried out at ultrahigh vacuum levels (10^{-9} torr) in order to allow us to put on a pure coating. At 10^{-9} torr, it takes about 10^3 sec to lay down a monolayer! Now, we can work more slowly and still make sure that the layers that we want to put on our surface are indeed placed there.

We can look at these facts another way: Let's compare the purity of our vacuum to the purity of something that you perhaps know a little about. Let's use ultrapure helium, sometimes called "five nines" helium (meaning 99.999% pure), as our example.

Most people think of this as being very pure. To get an equivalent level of purity in our vacuum chamber would require that we remove all but ten parts of every one million parts that were originally present. Looking at our table, we see that one millionth of atmospheric pressure is about $760/10^6$, or about 7.6×10^{-4} torr – barely into the high vacuum range! It's about 1 mtorr!

Even at 1 mtorr, our vacuum chamber has too many molecules (too much "dirt") to carry out some of the processes that we perform in a chamber. In fact, at a typical high vacuum working level (10^{-6} torr), we are in the parts-per-billion purity level. Ultrahigh vacuum gets us into the equivalent of parts-per-trillion purity level (one part per 10^{12}).

You can see that we can indeed get a very clean working environment by using a vacuum system. This may also help you to see some of the problems that go along with using a vacuum system. Let's go on now to look more in detail at some of those problems.

How Gases Are Pumped

One obvious problem is to get rid of all those gas molecules in our vacuum chamber. Let's look at what happens as we pump down.

Our roughing pump removes more than 99.99% of the air or gaseous contaminants from the chamber. We know that this is adequate to perform some vacuum processes but not those requiring high purity.

As we begin pumping at atmospheric pressure, we are pumping out the air. This air is sometimes called the volume gas. As we continue to pump, we begin pumping the molecules which have been fastened loosely to the walls. After these are gone, the remaining gas load is mostly water vapor. In fact, 75% to 95% of it is water vapor.

When the chamber is vented, the water vapor clings to all surfaces and to its own layers. Water can build up layers about 100 molecules thick on surfaces! After the volume gas is removed, the water begins to come off the surfaces, and makes up most of the remaining gas load.

As we continue to pump down to lower pressures, the character of the gas load changes. Other gases begin to make up the major portion of the gas load, as you can see in this table:

RESIDUAL GAS LOADS

Pressure (Torr)	**Major Gas Load**
Atm	Wet air
10^{-3}	Water vapor (75%-95%)
10^{-6}	H_2O, CO
10^{-9}	CO, N_2, H_2
10^{-10}	CO, H_2
10^{-11}	H_2

As we continue to pump, the mean free path of the gas molecules beomes longer and longer. We have gone from the viscous flow range into the molecular flow range. Remember that in molecular flow, pumping occurs only when the molecules randomly move into the pumps of their own accord. We now need high vacuum pumps.

base pressure

The vacuum system can be pumped to lower and lower pressure as long as the throughput of our pump is greater than the gas load. When the throughput equals the gas load, the pressure stabilizes. We have reached the *base pressure* for our system. Reaching base pressure means that in the high vacuum range, the following happens: the system pressure goes down only as long as the gases make it into the pumps faster than they come off the walls and other surfaces; then, when the gases come off the surfaces as fast as they make it into the pumps, the pressure for that particular system will go no lower.

outgassing
virtual leak
real leak

When you find that you cannot "pump down" to the typical pressure for your system, you may be seeing the effect of an increased gas load. We call this *outgassing*, or a *virtual leak*, or perhaps just a leak. Why the increase in the gas load? A dirty system—more molecules on the walls to be pumped off. (It may also be because of a *real leak*—a crack or hole in the system.)

We have briefly reviewed the purity levels that we achieve in a vacuum system and the way that gases are pumped. Let's now go on to consider how these are affected by the materials that we use in a vacuum system. So, in this next part of our discussion, we will touch on a few important properties of the materials that go into a vacuum system.

Materials

Materials used in vacuum work often need to withstand wide changes in temperature. This is because the equipment is often baked to drive gases off the chamber surfaces and into the pumps. In other cases, chilling to very low temperatures helps to produce a good vacuum. Some equipment is exposed to both high and low temperatures.

thermal expansion coefficient

Materials change in size when their temperatures change. This size-to-temperature relationship is called the *thermal expansion coefficient.* These changes in size are different for different materials.

Materials with different expansion rates are often joined to each other in vacuum equipment. During bakeout, the materials change in size at different rates. This causes strains at the points where they are joined.

The strain distorts the joints and can result in—you guessed it—leaks! Unless, of course, something is done ahead of time to prevent them. We'll continue this part of our discussion later.

Another major problem in vacuum performance is the outgassing rate of the materials in the system. A rough surface has a larger surface area for gases to stick to than a smooth surface does.

The problem is that this kind of sticking is only temporary. The partially trapped gas slowly comes off the walls after roughing is complete, making it difficult to achieve good vacuum performance. Also, some materials just naturally outgas more than others.

So, we want the materials used in our vacuum system to have several characteristics:

- Wide temperature tolerance
- Similar thermal expansion rates
- Low outgassing rate

There are other characteristics that may be of importance to us. We may need an electrical conductor or insulator, high strength, a thermal conductor or insulator, a non-magnetic quality, elasticity, low volatility, low chemical reactivity, radiation resistance, and probably others as well. With these characteristics in mind, let's look at some of the materials used in vacuum systems.

Stainless Steel

Stainless steel (or SST), 304 SST in particular, is widely used. It is a high-strength material that stands up to the wide temperature changes experienced in vacuum work. We need the high strength to withstand the air pressure trying to collapse our vacuum container.

Also, SST doesn't oxidize easily, so its surface remains smooth. This means that it doesn't produce large surfaces for gases to stick to. Therefore, it doesn't outgas much. It may be joined by welding or brazing.

A large work chamber made of "easily" machined 304 SST will usually have many ports welded to it. It may have pieces attached by brazing as well. Some stainless steels are non-magnetic, which is important in some applications.

Copper

Our chamber may have OFHC™ copper used as gasket seals or as plumbing to carry materials in and out of the chamber. (OFHC means oxygen-free, high conductivity.) Because of its careful refining process, OFHC copper contains very little oxygen. Therefore, it doesn't outgas much.

OFHC copper can also take wide changes in temperature. This is important because copper is often baked at very high temperatures, then chilled to very low temperatures. It is a very good electrical and heat conductor.

Brazing and welding are common methods of joining copper to copper and copper to other materials. OFHC copper is an ideal gasket material because it is relatively soft. Also, it contains very few microscopic leak paths (micropipes), which would prevent production of high and ultrahigh vacuum levels.

The ability of copper to conduct heat makes it an excellent choice for cryogenic applications. Copper is used in liquid nitrogen traps and cryogenic pumps. It is not as inert (non-reactive) a material as we might like. As a result, we usually nickel-plate copper to improve its chemical resistance. We use copper to handle large heat loads, as in cooling a sputtering gun or evaporative source.

Of course, copper is widely used because of its electrical conductivity. We use it to get any amount of electrical energy into our vacuum system.

Ceramics

We use ceramic materials (alumina, in particular) to contain electricity. Ceramics have excellent insulating properties, both for electricity and heat. We routinely braze the ceramics to the other materials in the system.

Ceramics are fragile but have great compressive strength. Their thermal expansion rate (also called coefficient of expansion) is very low.

Kovar

We use an intermediate material, such as Kovar™, to join glass to metal and ceramic to metal. Kovar has a coefficient of expansion which is between that of ceramics and stainless steel. By brazing the Kovar to the ceramic, then the Kovar to the metal, we get a vacuum-tight seal that remains leak-free even when exposed to extremes in temperature. Kovar is the trade name for an alloy composed of 54% iron, 29% nickel and 17% cobalt. It is magnetic.

Elastomers

Elastomers are materials that are flexible but not compressible. As such, they are very good to use for gasket seals. Because they are soft, they fill the gaps between mating surfaces and make leak-free joints. Their elasticity, or the ability to spring back to their original shapes, makes them reusable in most cases.

The permeability of elastomers can be a problem in some systems, particularly UHV systems. In these systems, the additional gas load caused by permeation causes problems. Elastomers in general are quite permeable to helium. Leak checking with helium can give slow indications of small real leaks when the source of helium is actually helium that is slowly diffusing through the seal into the system.

Buna-N™ is a common elastomer that is used because of its resistance to helium permeation and because it is inexpensive. It works very well in seals that are not going to be heated above 80°C. It is essentially a synthetic rubber material.

Viton™ is an excellent elastomer which is widely used for O-rings, valve seals, bonnet gaskets and chamber L-gaskets. It outgasses very little, so it can be used for both high and ultrahigh vacuum work. Viton will withstand temperatures up to about 150°C.

Polyimide™ is substituted for Viton where higher temperature tolerance is required. Polyimide will remain elastic up to about 200°C. It is a stiffer material than most elastomers. Therefore, it requires higher sealing pressure to assure leak-free operation. It is also more resistant to radiation than most elastomers. But it has the disadvantage of absorbing water.

Silicone compounds also withstand high temperatures, have poor outgassing rates and are quite permeable to helium and water. They are used in vacuum furnace work because of their temperature tolerance.

Teflon™ is also a good elastomer. However, it is also a plastic-like material, meaning that when it is deformed it tends to remain deformed. We call this "cold flow." When we try to use Teflon as an O-ring, it flows slowly out of the seal, even at room temperature. This, of course, results in a leak at the seal. Teflon is widely used to seal pipe thread joints and ferrules in flexible couplings. The threads help hold the Teflon in place and therefore stay leak-free. Teflon is quite permeable to helium. It can withstand temperatures to about 150°C.

Viton, Polyimide and Teflon are all fluoropolymers. They should not be overheated or burned due to the possibility of toxic gas production.

We have seen quite a collection of materials here. These are, of course, not all the materials that are used in vacuum systems. Special processes may require materials that must meet quite different or special requirements.

Our requirements for vacuum systems in general expose some materials to very low temperatures, others to very high temperatures, and some materials to both. We must allow for the changes in the size of materials when their temperature changes.

We must also be concerned about the surface texture and how it will affect the outgassing rate into the system. Remember that the gases stored or adsorbed on the walls become our major problem under molecular flow conditions.

We may be quite concerned about the vapor pressure of solids under the conditions present in our vacuum system. Recall the graph of vapor pressure of the elements from chapter 1; the graph suggests that there are elements such as lead, zinc and cadmium which may have a vapor pressure which is too high for vacuum system use. This means that brass parts and cadmium-plated screws are not to be used in a high vacuum system. Their vapor pressure will be high enough to prevent us from reaching operating pressure, particularly if we will be using high temperatures in the process.

The same consideration should be used in determining what organic materials should be (or not be) used inside the vacuum system. Materials with high vapor pressure will cause higher gas loads. This typically means that the system will be unable to reach the desired pressure.

Joining Techniques

We have already mentioned several joining techniques in talking about materials. Welding and brazing are very commonly used in construction of vacuum systems.

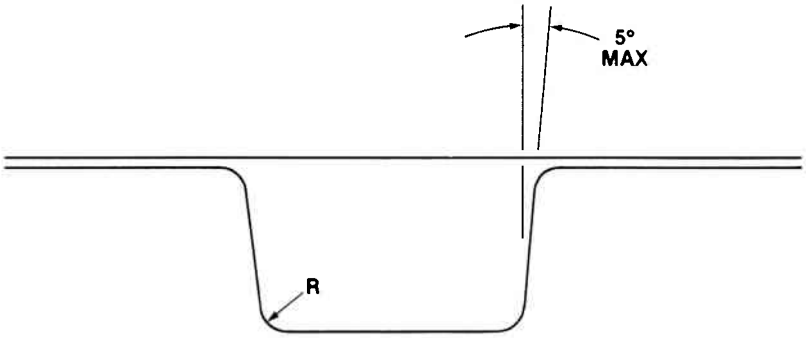
When we need to be able to take part of our system apart and put it back together leak-tight, we use flanges or couplings to join the system components. Let's take a look at the various types of flanges that can be used.

Flanges

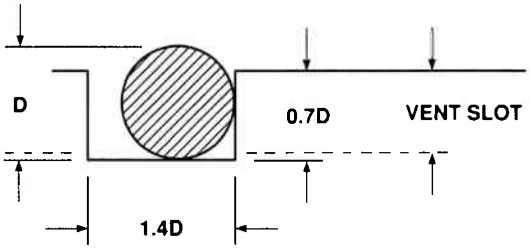
Flanges enable us to connect (join) the system parts in a reasonable and convenient manner. They also make it possible to quickly connect feedthroughs for purposes of controlling and monitoring system operation, and to maintain the system when trouble occurs.

Elastomer-Sealed Flanges

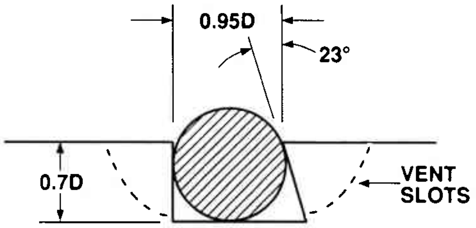
Elastomer-sealed flanges are used where there is little objection to the use of an elastomer, mostly based on temperature considerations and, perhaps, outgassing.



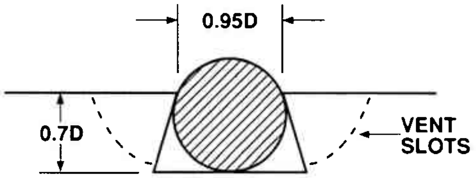
The groove in the flange for the O-ring should have sides that slope outward to a maximum of 5°. The groove should also have a radius on the inner corners equal to about two-tenths of the O-ring diameter. The surface finish of the seal area should be at least 32 microinches. The outer edges should be smooth, to avoid scratching the O-ring when making the seal.



The depth of the groove provides for deformation of the O-ring to about 70% of its unsqueezed diameter. This gives enough elastomer material to make the seal without overstressing the O-ring, but not so much as to force it out of the groove where it might be pinched or cause excessive outgassing. A vent slot is usually machined across the face of the groove to eliminate trapped volumes and for leak detection.



A. DOVETAILED O-RING GROOVE

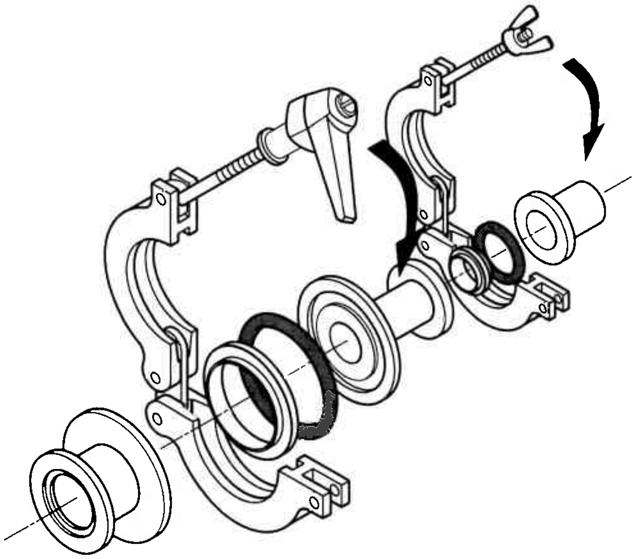


B. DOUBLE DOVETAILED
O-RING GROOVE

DOVETAILING OF O-RING GROOVES

The O-ring groove can be dovetailed to help retain the O-ring. You may also see fully dovetailed O-ring grooves in cases where a gas flow process might otherwise blow the O-ring out of the groove. Dovetailing, or keystoning, is also useful for retaining the O-ring against the force of gravity. The O-ring groove must be relieved to prevent pockets of partially trapped gas from becoming sources of virtual leaks.

Another popular type of elastomer flange is the KF™ flange. As marketed by Varian, it is known as the KLAMP-FLANGE™.



KF FLANGE ASSEMBLIES

The flange is of standard ISO 2861/1 design, consisting of two symmetrical flanges, a center ring to support and position an

O-ring, and a clamp that allows assembly without any tools. KF flanges are quite convenient to use in rough and high vacuum systems.

Here are some suggestions you will find useful when working with O-rings:

1. When preparing to make a flange connection, be sure to clean and dry the groove and the flat mating surfaces. Check the sealing surfaces for scratches that cross the seal area.
2. Lightly lubricate the O-ring with a vacuum grease such as Apiezon-L™. Then, wipe off most of the grease with lint-free paper before making the connection. Keep in mind that the O-ring makes the seal, not the grease. The grease makes the O-ring slip and helps it to conform to its groove.
3. Don't apply a lot of helium to an O-ring when leak checking. You will get a small, slowly increasing signal as the helium permeates the O-ring and goes into the vacuum chamber.
4. If you reuse an O-ring, visually inspect it to make sure it has no small cross-wise cracks or nicks that might leak. If it has swollen because it has been exposed to solvents or excess heat, do not reuse it. It is best practice to replace used O-rings with new ones.
5. Leak checking O-ring sealed flanges with solvents affects the O-rings (acetone is an example). The solvent slowly works into the O-ring and on into the system, causing an outgassing problem. The O-ring may even tend to dissolve in the solvent and become gummy and sticky.
6. O-rings will absorb water and will cause outgassing of water vapor. Baking the O-rings will minimize this problem.

Let's go on now to discuss another variety of flanges, the metal-sealed variety.

Metal-Sealed Flanges

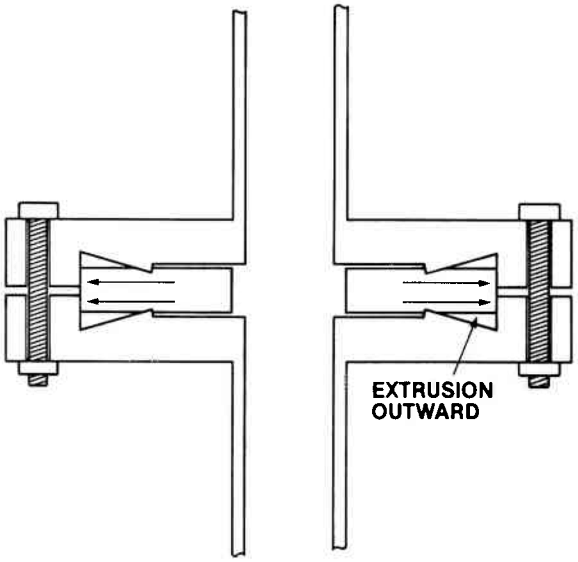
Metal-sealed flanges accomplish the same task that elastomer-sealed flanges do. They do have certain advantages over elastomer seals, however. They may be used at much higher temperatures—up to 500°C. They have low outgassing rates. They are more expensive to install than elastomer-sealed flanges.

The Varian ConFlat™ flange has become the vacuum industry's standard flange. Let's take a more-detailed look at this flange and the features that have made it a reliable and dependable flange.

The process starts with the selection of the 304 stainless steel. Either cross-forged or electroslag remelted (ESR) steel is used.

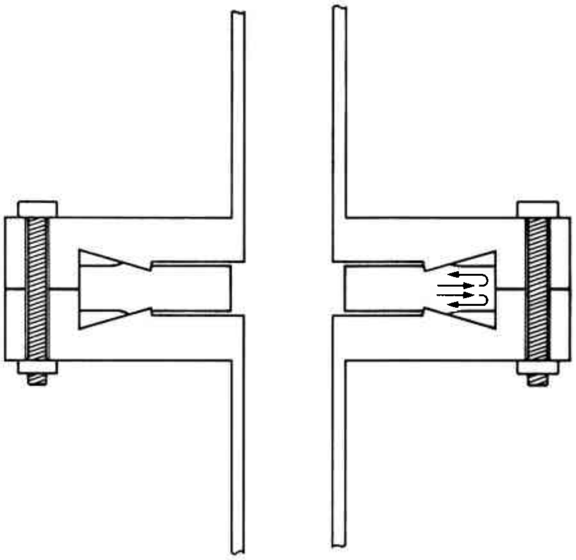
Care is used in selecting the material to eliminate leak paths caused by inclusions or micropipes in the material.

The design has built-in, long-term reliability because it captures the gasket. This prevents the gasket from flowing away from the seal area, even under the most extreme temperature changes. Let's look at the design of the flange, particularly the knife edge.



PARTIAL SEAL

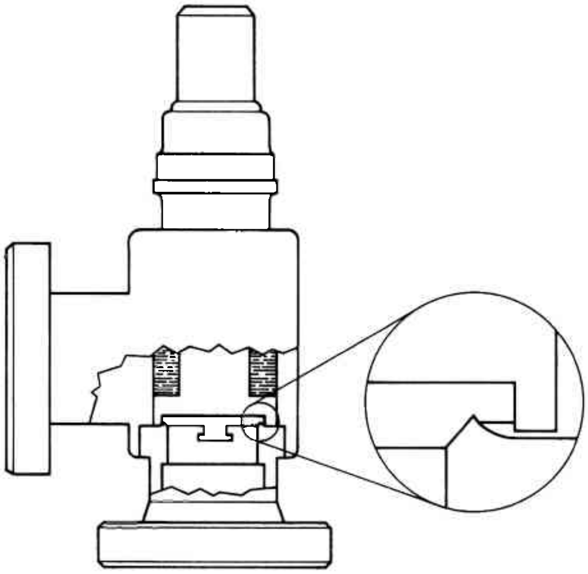
As the flange knife edge begins to bite into the copper gasket, the gasket material flows outward until it butts up against the flange supporting surfaces. At that point, additional outward movement is no longer possible. This keeps the material from flowing away from the sealing surfaces.



COMPLETED SEAL

As the flanges are bolted face-to-face, the gasket material is actually forced inward. This situation develops a tremendous pressure at the sealing edges. In fact, this gasket-capturing geometry develops close to 200,000 lb/in.^{2} where the knife edges and gasket come together.

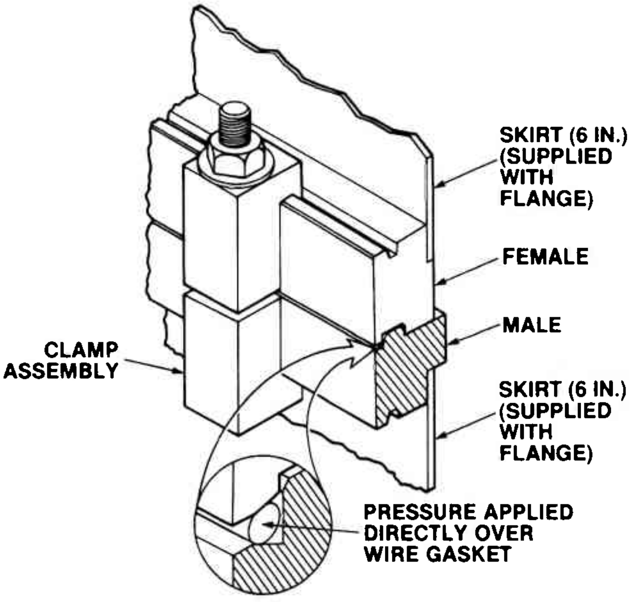
The ConFlat flange seal-capturing geometry is so reliable that it is used in many metal-sealed devices, such as valves and compression ports.



ALL-METAL VALVE

The figure shows an all-metal valve, made entirely of stainless steel and copper. The sealing surface inside the valve has a knife edge which cuts into the copper button to seal the valve closed. A valve of this sort can be baked to 450°C if necessary.

Another variation of the all-metal flange is the Wheeler™ flange, which is shown at the top of the next page.



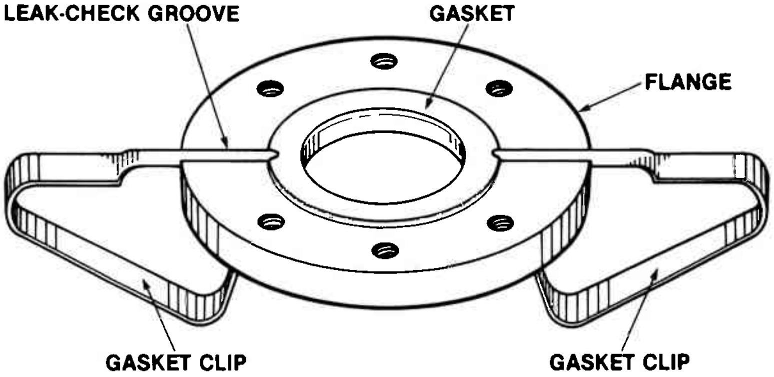
WHEELER FLANGE

This flange is used for larger diameter seals. It seals by forcing a copper wire into a compressed area under pressure, much like the ConFlat flange. The force is applied with large C-clamps. Wheeler flanges may be used reliably up to 450°C.

Here are a few suggestions to think about when using all-metal flanges:

1. The knife edges of fixed flanges are protected because they are set back behind the flange faces. The knife edges are exposed on the rotatable flanges and can be damaged if they are not handled properly. Protect the knife edges by covering them with plastic caps. Never place them on a bench or surface where they may be scratched.

2. The grooves milled across the flange mating faces are helpful. They can serve two functions: (1) for a quick leak check of the flange joint, just spray a little helium into the slot when leak checking; and (2) they will also allow the use of gasket clips to hold the gasket in place while you bolt the flanges together.



3. The bolts used on the ConFlat flanges have a twelve-point head on them. This is not done to frustrate you. It indicates that a high-strength steel has been used for that bolt. A good hardware supplier has the 1/4 in. or 5/16 in. twelve-point wrenches you need to hold these bolts while you tighten them. Lubricate these bolts to prevent them from seizing up (galling). We recommend a high-temperature lubricant such as Fel-Pro, C-100 or C-5A.

4. Don't reuse the copper gaskets. The time spent replacing a leaky used gasket that you just installed is much more expensive than the price of a new gasket.

5. Examine the knife edge of the flange, especially on rotatable flanges, to see if there are any nicks or scratches across the knife edge. The general rule is that if you can feel the scratch with a fingernail, it will leak. The flange should not be used. Some scratches may be burnished out.

6. Tighten the flanges metal-to-metal to insure a good seal and that any mismatched forces are carried through the flange faces, not the gasket.

Let's go on now to look at other joining techniques.

Cold Welding

Cold welding, or pinch-off, is a common method of sealing OFHC copper tubes such as those on ion pumps and vacuum tubes. After the components are baked and pumped out, the connecting tubes are pinched off. This crushes the tube walls together so tightly that a leak-tight seal is made. This keeps the devices under vacuum.

When you open a pinched-off seal, use a tube cutter. Don't use a hacksaw or some other cutting tool that generates particles. The particles will be drawn into the sealed-off device as the seal is opened. Remember that the copper particles are good conductors, and may land so that they will short out the device (of course, at the worst possible time).

Brazing

Brazing makes good vacuum joints. Brazing is a high-temperature soldering technique that is done in a hydrogen-filled furnace. The hydrogen atmosphere prevents oxidation at the joints, needs no flux, and allows for careful temperature control. You may have brazed something using a torch for heat. This is also a common process. But unless it is very carefully done, it results in strains which will develop into leaks. It may also leave flux residue which will outgas excessively.

Components that will be brazed are prepared by assembling them in a jig or holder with the brazing material placed between the parts. The brazing material is in the form of wires or gaskets.

They are then placed in the hydrogen furnace under very closely controlled temperature conditions. This is done so that no local heating and stresses occur where the parts are joined. This helps to keep the joints leak-free.

Gold or copper-based alloys are generally used as braze materials. It is possible to do multiple-step brazing by using alloys of varying composition and therefore different melting points.

Brazed joints may be quite strong. However, the material itself may have problems. We commonly use brazing to join ceramics to Kovar™ to stainless steel. The ceramic material is quite sensitive to shocks and tends to crack easily. Treat your brazed joints with care; you will have fewer leaks as a result.

TIG Welding

Tungsten-inert gas (TIG) welding is a widely used joining technique. It is a form of arc welding that joins parts by fusing them together without the use of filler materials or flux.

A TIG welding torch concentrates extremely high power at the joints. This makes it easy to weld stainless steel, which melts between 1,450°C to 1,550°C. TIG is also good for joining molybdenum, which melts at 2,620°C.