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POLYCARBONATE-BASED CRYOGENIC SEALING

P. Wertelaers

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POLYCARBONATE-BASED CRYOGENIC SEALING

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Abstract

The possibilities and difficulties of realizing cheap and robust cryogenic sealing, based upon a polycarbonate rectangular-section ring squeezed in between flanges with a 'nose' profile, have been examined. We summarize the properties and limitations of bis-phenol-A-polycarbonate, and compare these to our sealing requirements. Computations of the flange's nose indentation are discussed. We derive from it estimations on cool-down shrinking problems, tightening forces, and remaining elasticity. Flanged assemblies have been extensively tested in a liquid nitrogen environment: shock quenches from room temperature as well as long-term exposures to the liquid. Helium permeation through the seal has been measured as a function of temperature. A test in a liquid helium environment (4.2 K) has been done. We further discuss experiences with the production of large-diameter seals, from plastically deformed rods, butt-welded with a solvating agent at one or more places on the resulting ring's circumference. Finally, we present first impressions on a promising alternative welding method.

Résumé

Nous avons exploré les possibilités et les difficultés de la réalisation d'une étanchéité cryogénique, économique et robuste, basée sur un joint en polycarbonate. Ce joint, un anneau de section rectangulaire, serait écrasé entre des brides équipées d'un profile de 'nez'. Nous récapitulons les propriétés et les limitations du polycarbonate de bisphénol-A, et nous les confrontons avec nos exigences d'étanchéité. Des simulations numériques de l'impression du nez dans le joint sont présentées. Nous abordons les problèmes du rétrécissement au froid, des efforts de serrage, et de l'élasticité résiduelle. Des ensembles bridés ont été soumis à des essais poussés dans l'azote liquide : des trempes aussi bien que des expositions au liquide de longue durée. La perméation de l'hélium à travers le joint a été mesurée, et cela en fonction de la température. Un essai à l'hélium liquide (4.2 K) a été effectué. Nous présentons ensuite nos expériences avec la réalisation des joints de grand diamètre, à partir de tiges cintrées, soudées avec un solvant à un ou plusieurs endroits sur la circonférence. Enfin, nos premières impressions d'un procédé de soudage alternatif de grand potentiel sont discutées.

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1 MOTIVATION

CERN, the world's largest particle physics research facility, has since long been the scene of considerable activity in cryogenics.

Superconducting magnets for detectors first appeared in the bubble chamber era. They are applied in many contemporary and future detectors and targets. Another application in the detector field are the noble liquid calorimeters.

Particle accelerators are now becoming cryogenic sites of unprecedented size and complexity. 288 superconducting accelerating cavities are necessary for the energy upgrade of the 27 kilometre Large Electron-Positron Collider. The Large Hadron Collider presently under construction, to be lodged in the same tunnel and thus of equal length, will be fully equipped with superconducting bending and focusing magnets. There has been a continuous effort to realize and improve viable and economical dismountable connections.

Several types of cryogenic sealing systems are on the market. A few of them have gained large-scale application.

Indium-based solutions suffer from a lack of elastic bounce-back capacity, necessitating special precautions to preserve compression force. Realizing such seals requires a certain 'artist's touch'. They have never gained a true industrial status, and are used less and less.

CF[®]-type assemblies are known from the ultra-high vacuum world. A knife-type profile on the flanges is driven into a flat, square-section, metallic seal, usually made of copper. In that case, high demands are put on the flange material and the flanges are expensive. The bolting overhead is considerable. Standard equipment exists for nominal diameters up to some 200 mm. The solution is definitely not suited for very large assemblies.

The Helicoflex[®] seal consists of a soft metallic mantle (mostly unalloyed aluminium) wrapped around a torus-shaped, hard spring. The seal is circular in cross-section, at least before tightening. It gets squeezed to a near-elliptical shape. There is some elastic bounce-back. The principle is elegant; requirements on the flanges are modest. The bolting overhead is non-negligible, yet feasible. However the solution is proprietary, and large seals are very expensive. There are different opinions on the ability to re-use a seal. The aluminium tightness mantle can easily be damaged.

The work reported upon here has been an attempt to find cheap seals, that can be produced 'on site' if necessary, and which are industrially robust, limiting the requirements on flanges and bolting.

One could furthermore think of special applications like electrically insulating bridges.

A topic of specific interest is the ability to pair together flanges out of different materials. This pairing problem is one of the major issues when trying to develop cryogenic equipment (partially) out of aluminium.

2 ON POLYCARBONATE

In a broad sense, polycarbonates constitute the family of thermoplastic polymers containing a -O-CO-O- (carbonate) group, Fig. 1a. The only significant member, hereinafter simply referred to as polycarbonate or PC (Fig. 1c), is the result of a phosgenation process on bis-phenol-A, Fig. 1b. The material is also known under trade names such as Lexan[®], Makrolon[®] and Merlon[®].

Note the high density of intractible aromatic rings in the molecule's backbone, resulting

in a remarkably high glass transition temperature T_g of about 150°C. Temperatures up to 120°C can easily be sustained, and creep resistance at room temperature (RT) is outstanding for an amorphous (see below) thermoplastic. Melting sets in gradually at about 225°C, albeit with a very high melt viscosity, which evidently decreases with increasing temperature. Burning and thermal degradation problems become apparent from about 280°C onwards.

The side groups, methyl and oxygen, are not very bulky, yet the material shows little tendency to crystallize. Commercial products (mostly injection moulded) and semi-products (mostly extruded) are completely amorphous and show excellent transparency (safety glass).

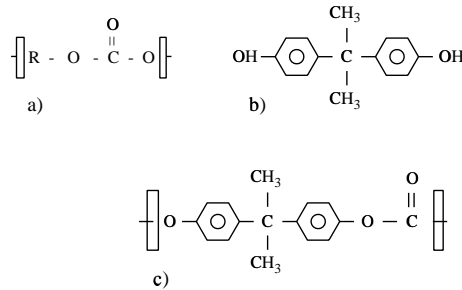


Fig. 1: a) Polycarbonates in the broad sense; b) Bis-phenol-A; c) Commercial polycarbonate (repetitive unit).

A slow cool-down from T_g or a prolonged ‘annealing’ treatment (.. 135°C ..) will nevertheless cause crystallization. This should be avoided. Not only do the products get opaque and stiffer and harder, they lose much of their ductility as well.

Because of this amorphous nature, the rubbery state ($T > T_g$) is characterized by a very small modulus (Fig. 2) and marginal yield and creep strengths. PC products get so soft above T_g that even their own weight becomes problematic.

Room temperature is already very low compared to T_g : the material is well into its glassy state. One hopes to find little extra trouble when cooling down further to nearly 0 K, but this is not entirely the case:

- The relative increase in elastic modulus is far greater than e.g. for metals (Fig. 2). It is however moderate compared to other thermoplastics.
- One or more so-called secondary-state transitions occur, undoubtedly linked to a loss of side-group mobility. The first such transition is sometimes quoted at around –50°C. A corresponding drop in impact strength is reported as well.

Thermal expansion is depicted in Fig. 3. Locking PC to metal at RT, and then cooling down to near 0 K, induces roughly a 1% mechanical strain in the polymer. Thermal conductivity is low: about 0.2 W/(m·K) at RT, decreasing for decreasing temperature in the usual fashion for amorphous thermoplastics.

Water absorption in the glassy state is very low. Humidity can nevertheless be a notorious enemy:

- The absorbed water results in spectacular — and fast — bubble formation when heated to above 160–170°C. Drying seems to be tedious, requires high temperatures (120°C) and may take many hours for pieces more than just a few millimetres thick.

- Melting humid feedstock is known to give brittle products. At temperatures below 120°C there are no known adverse effects on PC products which have undergone usual, ‘ambient’ moisture absorption. And this apparently also holds for cryogenic seals.

The C=O region is somewhat polar (has a small electric dipole moment); this opens up the possibility of heating (welding!) by dielectric losses. PC is dissolved in agents such as methylene chloride (CH_2Cl_2).

The material is fairly resistant to a number of agents, but is readily attacked by some others. Especially noteworthy is the environmental stress cracking in the presence of oils, greases, solvating agents. The cleaning of products with ethanol should NOT give problems.

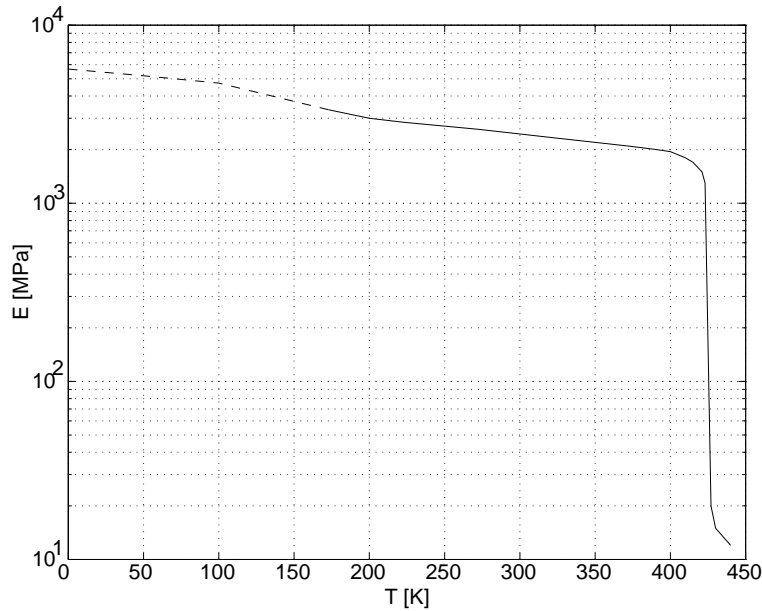


Fig. 2: Small-signal tensile elastic modulus as a function of temperature (literature compilation). Data below about 200 K are diffuse. Poisson’s ratio is quoted to be 0.37 and 0.39 at 4 K and RT respectively.

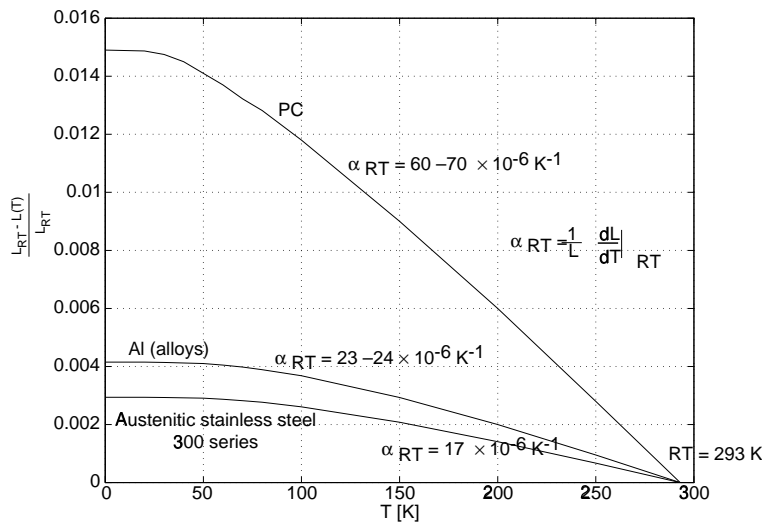


Fig. 3: Thermal expansion of PC compared to some relevant cryogenic construction metals (literature compilation).

3 DEFORMATION, DUCTILITY, TOUGHNESS, CRAZES, CRACKS AND FRACTURE

There are basically two modes of plastic deformation in a glassy, amorphous thermoplastic:

- (bulk) shear yielding;
- crazing.

Shear yielding is a constant volume process and responds mainly to the shear contribution in the stress state.

Figure 4 depicts a stress-strain curve and specimen ‘life’ resulting from a tensile test where shear yielding was possible. The elongation at break reaches attractive values, and one classifies the behaviour as ‘ductile’.

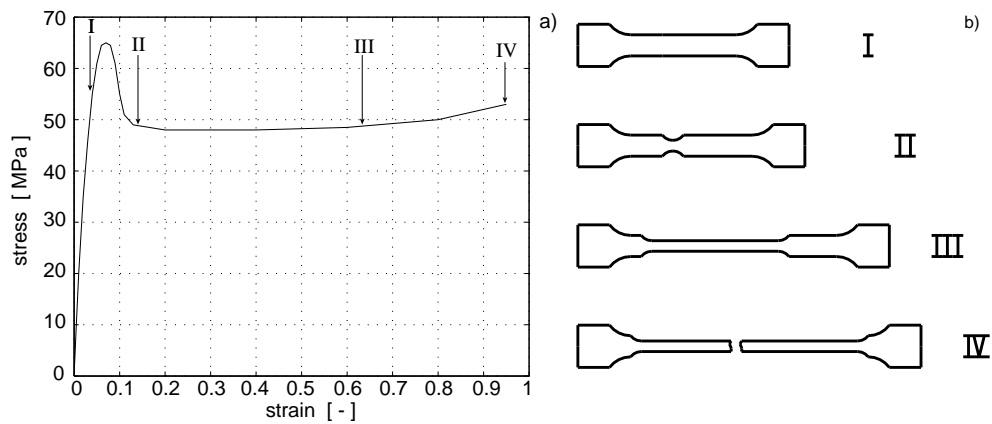


Fig. 4: Uniaxial tensile test on PC at moderate strain rates (order $10^{-2} - 10^{-3}$ per second), at room temperature: a) engineering stress versus engineering strain, typical curve; b) tensile specimen at different instances.

The very early formation of a local neck is a direct consequence of a mechanically unstable plastic deformation onset: the engineering stress (and thus the total force as well) decreases after having passed a maximum value (which is called the ‘yield stress’ by convention). This ‘strain softening’ is typical for many (ductile) thermoplastics. Further straining results in a slight orientation strengthening, visible as a local rise at the end of the curve. This strengthening eventually stabilizes the necking, and makes the neck expand through the specimen before final rupture. The failure mode in this case is mostly shear tearing or a sort of microvoid coalescence. The strain softening instability tends to localize (the start of) plastic deformation. So-called shear bands are sometimes visible on the surface of a strained object. They try to orient themselves to the directions of maximum shear, Fig. 5. Pronounced, sharp shear bands may initiate cracks at their crossings, and are to be avoided.

The (much) more embarrassing deformation mode is crazing, a phenomenon typical for polymers (though there exists uncertainty about its occurrence in materials other than glassy amorphous thermoplastics). A craze differs from a crack in at least two ways (Fig. 6):

- It is much smaller, especially thinner, and its edges (tips) are very sharp.
- The craze is not empty. So-called ‘fibrils’ of extended chains, drawn from the bulk material, span the gap and give it a certain load-carrying capacity.

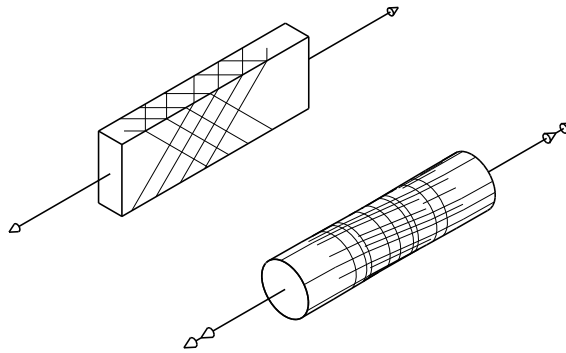


Fig. 5: Shear bands for different types of loading.

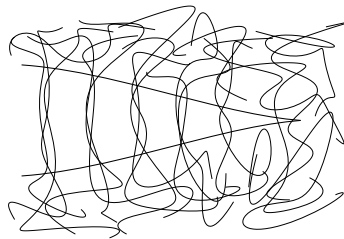


Fig. 6: Artist's view of a craze.

If crazing occurs, then real cracking can never be far away. Crazing may not at all be a brittle event from a material scientist's point of view. However, catastrophic failure due to crazing–cracking IS regarded as brittle from a more mechanical viewpoint when only a very small portion of the total volume was capable of absorbing plastic deformation energy.

For crazes to form or to grow (increase in volume), the hydrostatic component of the stress state must be positive, so tensile. There is a clear tendency for a craze (and a crack) to orient itself perpendicular to the direction of the largest principal (tensile) strain.

Whether or not (unfortunately more often than not) an object favours responding in crazing terms rather than bulk yielding, depends on several factors.

- Material. Tensile tests carried out on samples out of polystyrene (PS) or polymethylmethacrylate (PMMA — also known under trade names such as Perspex[®] or Plexiglas[®]) result in low rupture strains: crazing! The outcome for PC is quite different, as discussed above. This apparently big difference has been the subject of research and controversy, yet a foolproof explanation does not exist. We must however emphasize that the difference is not as big as it seems. Crazing and bulk yielding are competing mechanisms. Bulk yielding happened to set in first, under the specific conditions, for PC, but some alteration may give a completely different result. Even PC is quite prone to crazing!
- Material again. The higher its molar mass, the higher the density of molecular entanglements. The risk of craze formation reduces and toughness benefits (but reaches an upper bound).
- PC is known to be sensitive to 'strain crazing'. A design rule says to limit long-term tensile straining to below 1% at RT. The temperature dependence of this limit value is unknown to the author.

- Aggressive agents can have a very significant effect. Remark: nitrogen and argon are reported to have some adverse influence on PC at about 80 K [1].
- Stress state: very important! Multi-axial tensile stresses — which can easily occur at sharp edges — are very dangerous. PC's plane-strain fracture toughness K_{Ic} is reported to be only $2.2 \text{ MPa}\sqrt{\text{m}}$ (RT value) [2]. The crazing tendency is firmly reduced as the hydrostatic stress component vanishes, or even better, becomes negative (compressive). Under the latter condition, even PS and PMMA exhibit shear yielding capability!

The influence of strain rate and temperature on the fracture behaviour is a difficult issue [2–4]. Plane-strain fracture toughness increases for temperature decreasing from ambient to some 77 K, with some crack propagation rate dependence at the latter temperature. At 4 K, again a more modest value, apparently not rate dependent, is found. A (sudden) drop in blunt notch impact 'strength' for decreasing temperature has been reported, but be careful with the interpretation!

Much of the test work (see further) has been devoted to inventorizing crazing tendency in our specific sealing application, and under conditions which are typical for cryo-sealing.

Polymers are not able to deform plastically at extremely low ($\ll 77 \text{ K}$) temperatures, where they are essentially brittle (failure mode: cleavage).

Rupture strain of PC is reported to be 3.3% at 4.2 K [3]. This may not seem much, yet the value is among the highest of all polymers mentioned.

4 THE FLANGE PROFILE - INDENTATION BEHAVIOUR - COMPRESSION - BOLTING

It was judged useful to apply a seal with a simple, rectangular cross-section. Each hard flange would then have to drive a local impression (an indentation) into the softer seal, much like CF assemblies.

The two flanges are not in direct contact with each other. A PC sealed assembly becomes an electrically insulating bridge, when precautions on the level of bolting are taken. Such a bridge has been installed in the NA48 experiment (liquid krypton cryostat).

One would be tempted to make a 'coarser' (deeper and larger) impression than CF. This has the clear advantage that we become much less sensitive to material and surface imperfections, for both flange and seal. The economic potential is evident; the assembly is more 'robust'. The lower stiffness and yield strength of the polymer seal (compared to the metallic CF seal) make it possible.

A rounded flange indentation profile (a 'nose') has been chosen, rather than a sharp CF-like 'knife'. From an emotional viewpoint (!) sharp never fits well to polycarbonate (not even when the stress state right under the knife could turn out to be triaxially compressive). In practice a nose flange could prove more robust than a knife one; we would like to be able to work with flanges, which are cheaper than the conventional, forged, vacuum-remelted 316LN!

Figure 7 confronts the traditional CF geometry to the used PC-sealed solution.

Some further details on the 'nose' are given in Fig. 8. It must firmly lock the PC seal when cold, in spite of its tendency to shrink about 1% more (in absolute terms) than the flange metal, see Chapter 2.

This also means that mechanical, circumferential, tangential, tensile straining of about 1% will be imposed on the polycarbonate, when cold (at least, on the bulk part of the seal — the situation is much more complex in the vicinity of the impression). The consequences of this are that

- we have to carefully watch whether strain crazing develops. Indeed, this 1% is just the design limit, at RT, and there is uncertainty of how much constant strain PC can withstand at lower temperatures — see Chapter 3;
- the weld(s) in the seal has(have) to be strong enough!

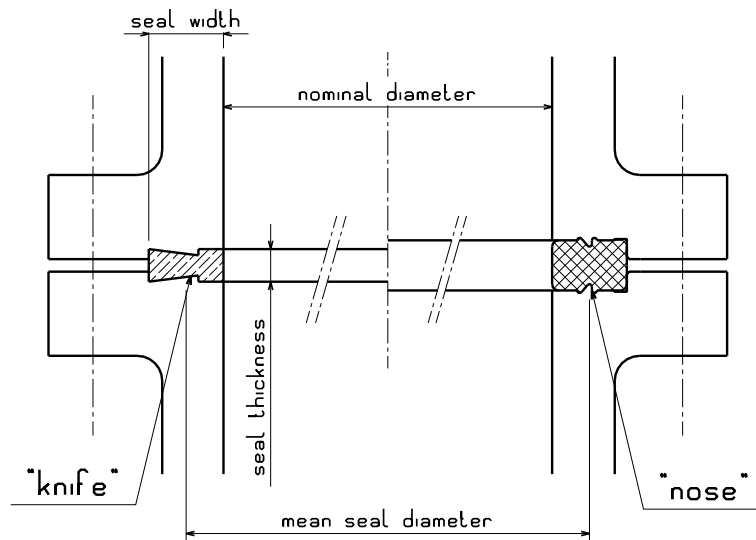


Fig. 7: CF assembly (left) and PC-sealed assembly (right), and some language conventions.

A rounding radius of 0.2 mm on the nose tip seems reasonable for non-extreme nominal diameters (between 35 mm and, say, 2 m). A consequence of this bluntness is a steep initial increase of the compression force (as a function of deflection). This handicap is relative, as the total indentation depth is considerably greater than the rounding radius.

Limiting the conical edge angle to 40° means limiting the increase of compression force. The situation is different for CF, especially for the larger nominal diameters!

It is practical to define the nominal compression stroke such that the bulk of the seal surface touches the flange surface, the latter acting as an ‘arrest’. Were the dips at the nose’s roots absent, then the inevitable rounding due to the cutting tool’s shape would result in a steep increase in compression force before actually attaining the arrest surface.

The layout is such that the final machining — of the nose — can be executed on a CNC lathe ‘in one go’, with one tool.

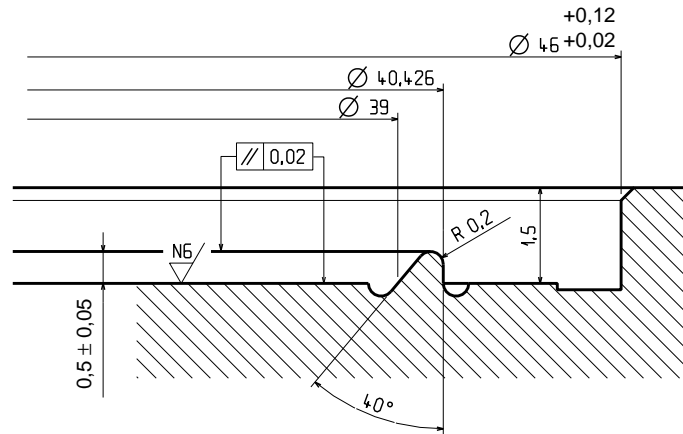


Fig. 8: Typical execution drawing for a flange, nominal diameter 35 mm (the flange of the ‘small device’ — see below). This geometry may not be the optimal one.

If the seal is able to withstand the 1% induced straining when cold, then it should certainly not have any trouble pairing together flanges made out of different metals. The imbalance in their thermal contraction is typically a per mille effect in absolute terms, see again Fig. 3. However, for very large diameters one has to care about additional seal shearing, Fig. 9.

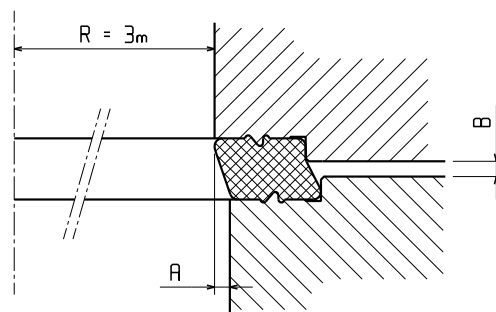


Fig. 9: An aluminium (upper) to austenitic steel (lower) union of 6 m nominal diameter; cold. The difference in shrinking between the two metals is of the order of one per mille, resulting in a shearing effect $A = 3$ mm approximately! The gap B has to be designed sufficiently wide, and consequently the seal becomes very thick. The precautions for the bolting are evident.

Numerical simulations of the seal compression are very cumbersome, due to its extremely non-linear character. However, in order to learn at least something, we nevertheless did perform some finite element modelling, albeit with important simplifications:

- A simplified geometry was modelled (and executed as hardware for measurements), Fig. 10.
- A somewhat simplified material (PC) stress-strain curve was used.
- Material again: Von Mises yield criterion; associated flow rule (Prandtl-Reuss); isotropic strain hardening rule. It was not possible to model a pressure-modified Von Mises criterion — more realistic for polymers — in the finite element code we used (Ansys).
- The nose indentation was modelled using a discrete number of gap elements.

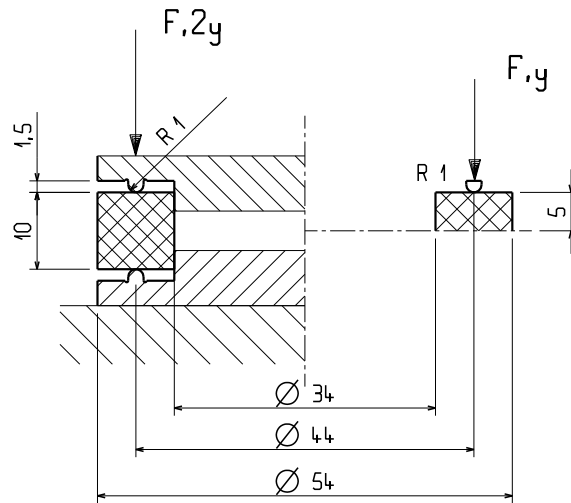


Fig. 10: Axisymmetric finite element model, further exploiting top-down symmetry (right), and the corresponding set-up for experimental verification (left) of the indentation behaviour.

Figure 11 depicts what happens with the seal after a moderate indentation, and Fig. 12 compares the resulting force-deflection dependence with measured curves.

Figure 13 represents the compression/decompression measurement of the ‘real’ geometry used for the ‘small device’. The forces are important, in some way comparable to those of a Helicoflex[®] seal. However the nose is rather big for a 35 mm assembly.

Figure 14 shows the bolting details of the ‘small device’. The moderate tightening forces do not necessitate at all such heavy bolts. They have merely been chosen in order to mount elastic washers with adequate force and deflection ranges. The device has been used for difficult helium tests, and we wanted to be really sure of having sufficient compression at all times.

On the other hand, the ‘large device’, depicted in Fig. 15, was only equipped in a poor man’s fashion, without any external elastic element (in spite of having austenitic steel bolts for aluminium flanges), and therefore handicapped in some sense. This big assembly served as test jig for deformed-rod/butt-welded seals, see Chapter 7. It only underwent simple liquid nitrogen testing. No accidents — like vacuum collapse in the cold — occurred in spite of the handicap. There are signs, however, of a potential performance increase (helium permeation!) in the case of less loss of seal compression force in the cold, see Chapter 5.

The flanges can, in the author’s opinion, be made out of any type of 300 series austenitic steel of correct standard elaboration quality. The mechanical properties of 304L are more than sufficient. Care has to be taken, however, in the case of aluminium products: a good mechanical performance is mandatory. Not only is the nose tip subjected to high stresses (Fig. 11a), one has to think of scratch sensitivity as well. The highly alloyed members of the 5000 (AlMg) family seem acceptable; 6000 (AlMgSi) alloys can be used as well, when hard (aged), but be careful about weldability. High-end UHV bolts are most likely not necessary, and have not been used.

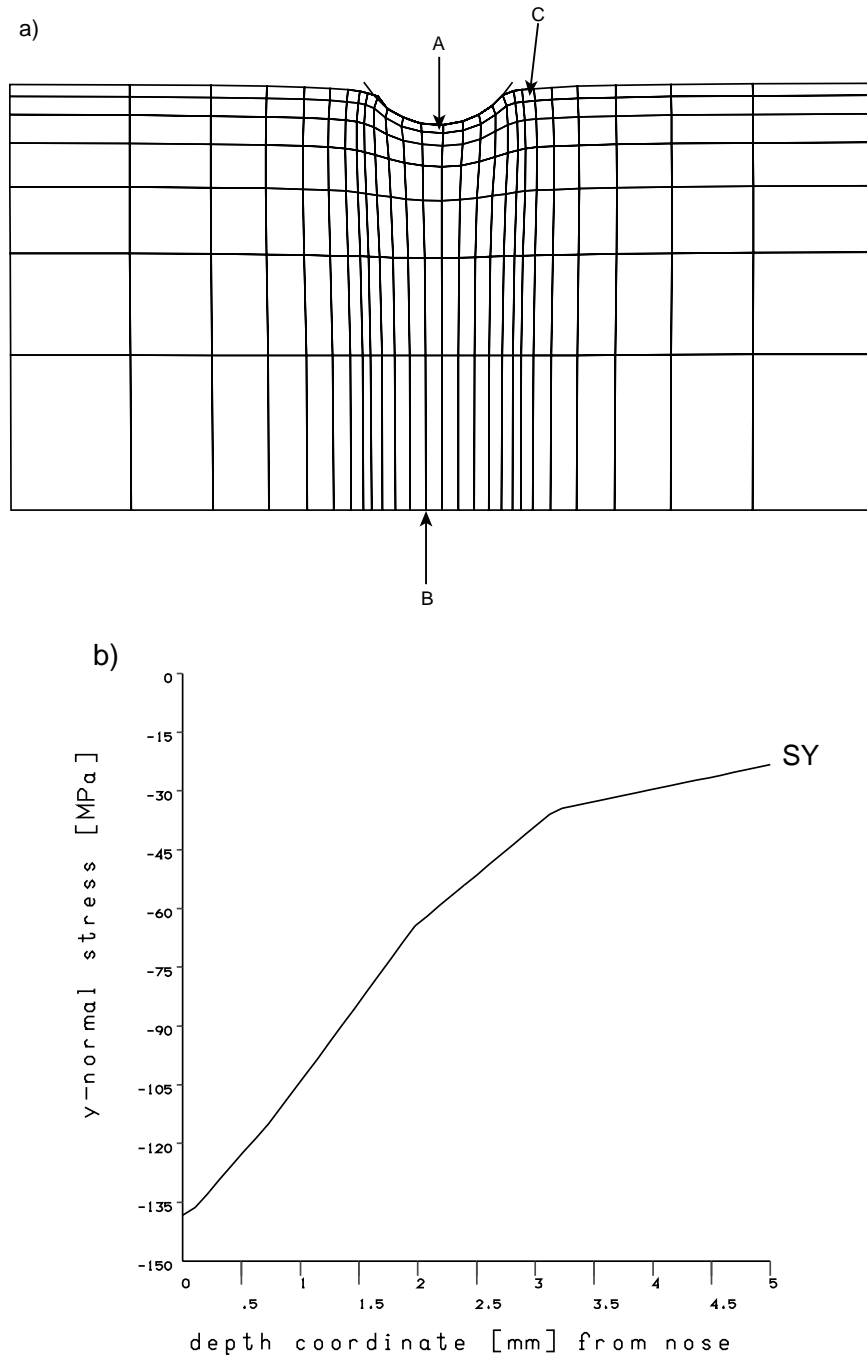


Fig. 11: Finite element model results; the nose entered 0.5 mm. The model's output is in true stress values, not engineering. a) The cross-section of the (half) seal. The rotation axis is far to the left. Only the skin of the nose is visible. Just below the nose (zone A) the equivalent plastic strain attains some 45%, and all three principal stresses are strongly compressive (ranging between -143 and -100 MPa - not conflicting with 65 MPa yield strength!!). Tensile stresses do not occur, except in the seal's core region (zone B), and at the free surface just outside the nose (zone C): radial stresses in both zones. b) The compression in the seal, from nose tip to seal core. A considerable amount of elastic energy has clearly been stored.

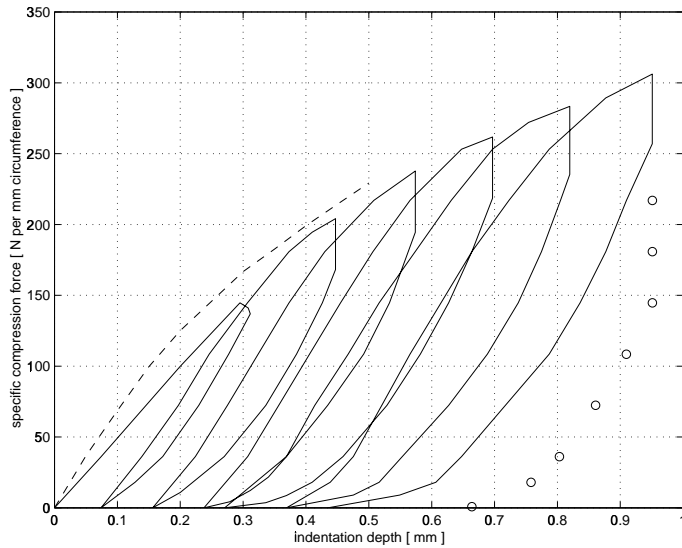


Fig. 12: Compression force versus indentation depth, the latter according to the simulation's convention (one-half, or, per flange). 0.5 mm in these units means that the two flanges have come 1 mm closer. — Measurements (solid): 'up' and 'down'. The machine was hand-driven, deflection rather than force being imposed. The test took about 15 minutes and the 'breaks' in between (vertical line segments) lasted some 30–50 seconds. Relaxation and anelastic/visco-elastic effects are eye-catching: you could virtually measure anything! The measured elastic bounce-back is enormous, but probably because the compression lasted only minutes (dotted: speculation of what might happen after much longer hold to maximum indentation). — Computations (dashed): only 'up'. The disagreement with the measurements is understandable, given the extreme non-linearities and the imposed simplifications on material constitutive behaviour.

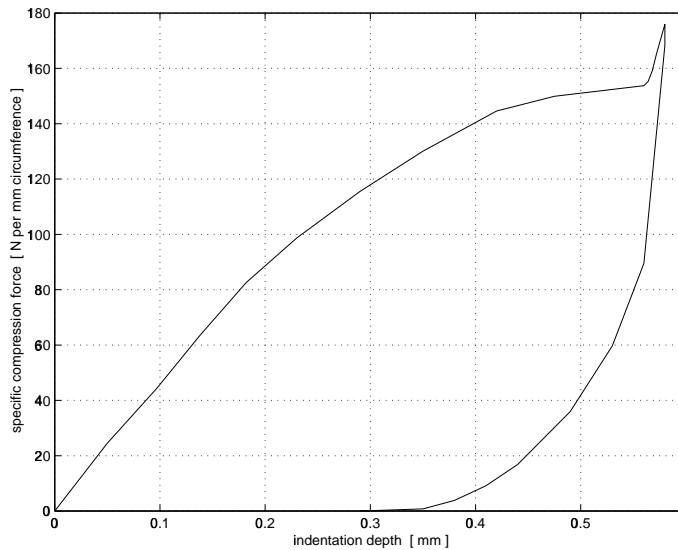


Fig. 13: Specific compression force versus indentation depth, measured with the blind flange of Fig. 8, and on a seal with half-thickness (Fig. 10) 2 mm. Again 'up' and 'down'.

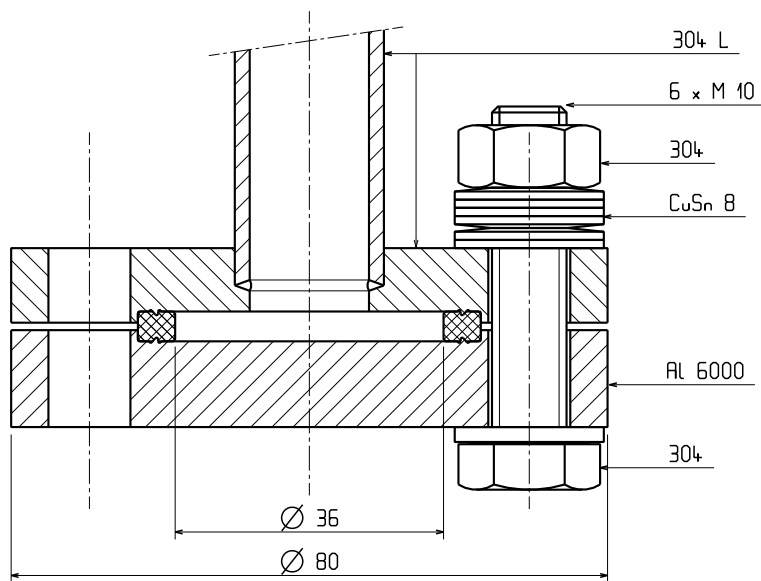


Fig. 14: The 'small device' bolted together.

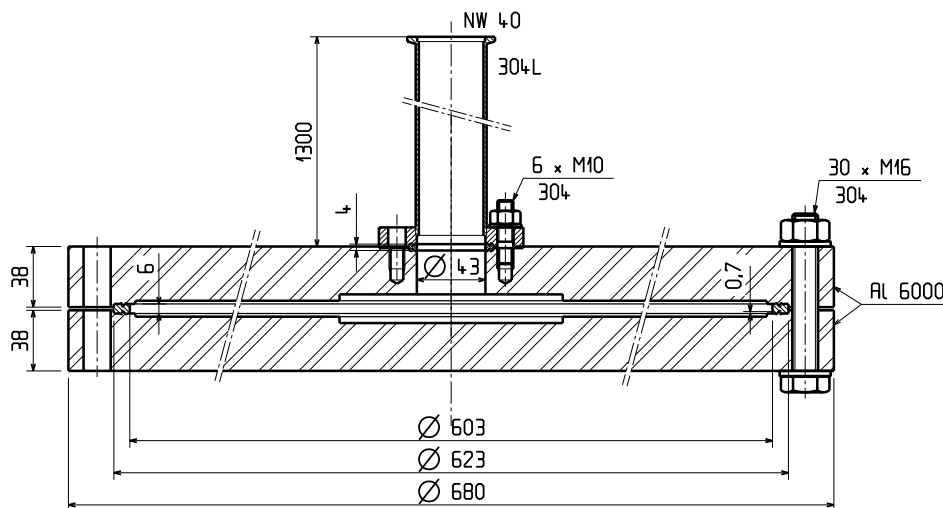


Fig. 15: The 'large device'. The noses are somewhat coarser: they have to cope with seals with less precise geometry (thickness variations, etc.).

5 LIQUID NITROGEN AND LIQUID HELIUM TESTS

The large device was put in a liquid nitrogen bath and has been tested with three seals, Fig. 16. The tube was long enough to be able to stick out of the open dewar; the valve on top of the tube — in order to isolate the device in case of trouble — remained at RT. Thanks to the very long flexible line, the device could be lowered into the bath while remaining connected to the pump/detector. Very brutal quenches from ambient to 77 K could thus be executed and carefully monitored.

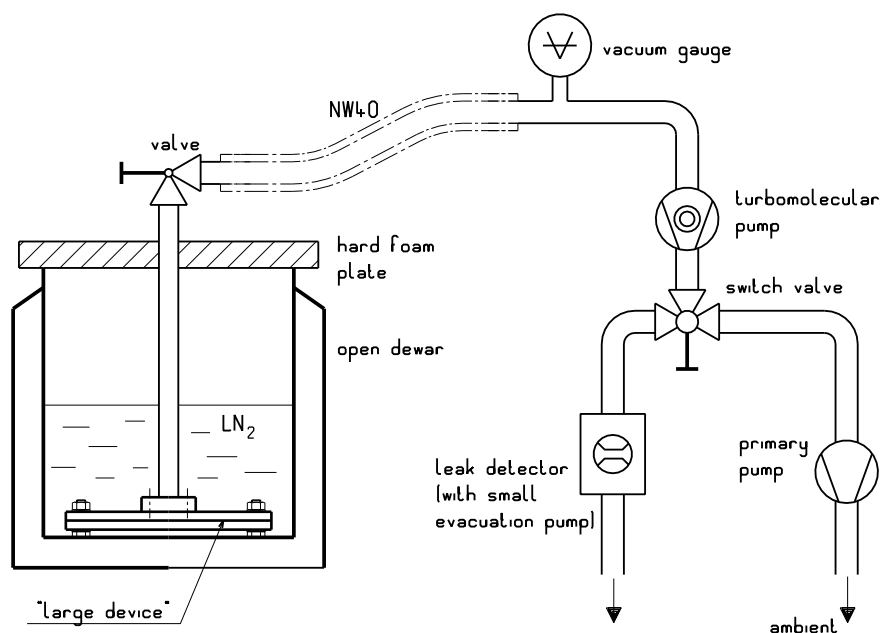


Fig. 16: The test set-up for the large device.

Note that the main flanges are made out of aluminium (6000 - aged), and the bolts out of austenitic steel; there has been no additional flexibility in the bolting. Note also that the connection to the austenitic steel tube is done by PC sealing as well; here there are no spring-loaded bolts either. Much of the compression force under a certain bolt gets lost when the following bolt is tightened afterwards to compress the seal further. The bolt is too stiff, and can hardly store any elastic energy. All bolts should be tightened synchronously in order to avoid this handicap, which is also present when compressing an O-ring, a Helicoflex, or even a CF. This has not been done. Simple, sequential tightening with only one pair of hands typically took four to five rounds.

Whether gently cooled down or quenched, the vacuum never collapsed. In general, the gauge reading descended from typical 10^{-5} down to 10^{-6} mbar or even better, and the helium background from some 1.5 to 0.5 times 10^{-9} mbar l/s. Sometimes the vacuum worsened temporarily on the way down to 77 K. This could have been due to some minuscule seal motion/deformation. Another theory would be additional surface outgassing due to oxide layer damage. The big aluminium surfaces had been machined, yet had not undergone the proper surface treatment for high vacuum afterwards.

Helium was sprayed around the device at regular intervals, before and during cool-down, whilst cold, and after eventual warm-up. When cold, this was done either by lifting the device somewhat out of the bath (but not entirely out of the dewar - humidity!), or by spraying into the bath. Sometimes a slow but significant reaction (many 10^{-8} mbar l/s) was registered after a generous helium spray. The bolts were then firmly re-tightened in the cold state. After this, the performance improved significantly. This re-compression has been done for the first and second seals, not for the third.

The first seal has been used twice, i.e. has been dismantled and re-mounted, compressed and cooled down again. It was still more or less tight (the vacuum didn't collapse), but performance had clearly degraded, sometimes in an awkward way. We have to mention that the ability of re-using has never been among our primary goals and we do not recommend such practice.

The second and third seals underwent long-time testing. They each remained in the device for about two months. The second seal was cooled down from RT three times, and was cold (and in contact with LN₂) for about one month altogether. The third seal was kept cold all the time.

A circumferential cracking at the outside of the indentation was visible on the dismantled seals: very light and shallow in the first seal, deeper and sometimes deviating outward to nearly-radial direction in the second one, Fig. 17. This may not be so surprising: see Fig. 11a. However, nothing of the sort was visible on the third seal. Apparently, the extra deformation by the firm re-compression, under unfavourable conditions of a low temperature and nitrogen environment (Chapter 3), had been just too much.

The small device was prepared for service in a warm-cover helium cryostat, Fig. 18. A first seal was ‘in’ for many months, and had been cold (to 77 K) a few times. One little edge-surface crack was found. The edge had been chamfered before, but this chamfer was rather sharp, not rounded.

The second small seal was rounded by hand. It underwent permeation tests down to 100 K and the 4.2 K LHe test afterwards. It had been ‘in’ for about three months. A third small seal has been ‘in’ for about two months, and it was permanently exposed to LN₂. Both second and third seals are free of any visible damage.

The performance of the small device during LN₂ testing (not in the He cryostat) was without comment, probably thanks to the spring-loaded bolts. Regarding the 4.2 K test in the cryostat, the leak detector did not respond at all — on top of the inherent set-up background signal of 1×10^{-9} mbar l/s — to the LHe environment.

Conclusions:

- one has to round the seal’s edges;
- cheapest bolting is fine, spring-loaded bolts are better;
- quench is no problem: no tightness collapse, not even temporarily;
- the nose shape is not yet optimal.

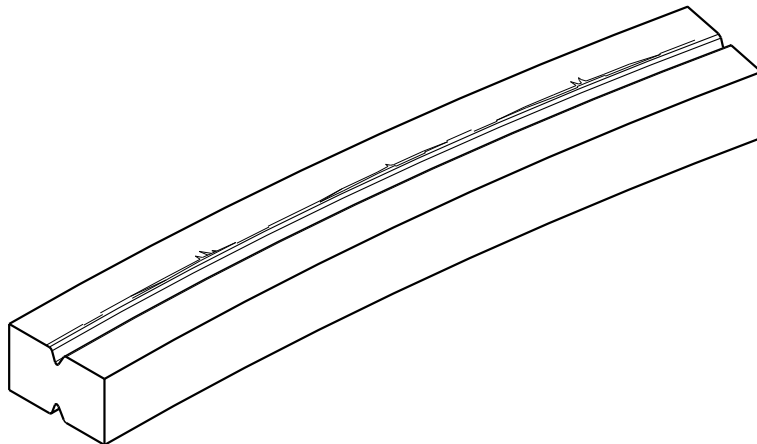


Fig. 17: Traces on the second large seal after the test; less pronounced on the first seal, absent on the third.

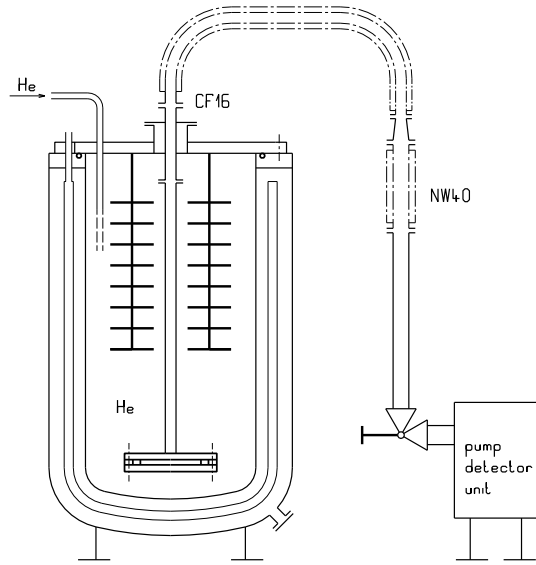


Fig. 18: The test set-up for the small device in the helium cryostat. Level and temperature gauges are not shown.

6 HELIUM (GAS) PERMEATION

Polymer materials are never perfectly tight; a certain amount of gas will permeate through them. Especially small molecules, like helium and hydrogen, might have little trouble.

A nice overview is given in Ref. [5], where values, and their temperature dependence, for a few common materials are given as well. We quote the essential:

- The total flow of a certain gas leaking through a plate of a certain material is given by:

$$Q = P \frac{A}{d} \Delta p ,$$

where A is the exposed surface (m^2), d is the plate's thickness (m), and Δp the driving force, being the difference in (partial) pressures (Pa). Q is the steady-state flow, and would conveniently be expressed in vacuum engineers' units: $\text{Pa m}^3/\text{s}$ ($= 10 \text{ mbar l/s}$). The proportionality factor P is the material's permeability for that specific gas, in m^2/s .

- Permeability is the product of solubility and diffusion, both being temperature dependent following an Arrhenius law. Consequently, for the permeation,

$$P = P_0 \exp\left(\frac{-E_p}{RT}\right)$$

in which E_p is the activation energy and R is the universal gas constant. An Arrhenius-type temperature dependence is characterized by a saturation for very high temperatures, and a very swift decrease for decreasing temperatures (rushes to zero faster than any power of T). Polymers are very tight at sufficiently low temperatures (provided that they have not degraded by cracking, etc.).

- Permeation for helium and hydrogen is of the same order of magnitude. The values for methane are significantly lower.

- Semicrystalline thermoplastics globally have lower permeation rates than amorphous, because of better average chain packing in the crystallites.

On our request, Forschungszentrum Karlsruhe carried out helium and hydrogen permeation measurements on 3-mm-thick PC plate, for temperatures ranging between +22 and -80°C. As for their previous tests, the Arrhenius fit was always excellent. It yielded [6]:

$$P = 6.4 \times 10^{-12} \text{ m}^2/\text{s at } 20^\circ\text{C for He (} 10 \times 10^{-12} \text{ for H}_2)$$

$$E_p = 16.565 \text{ kJ/mole for He (23 for H}_2) .$$

The 3-mm-thick plate needed about two hours to reach steady-state conditions at RT, after the helium was admitted. Reaction rates at lower temperatures would be much lower.

Based upon this knowledge, one could make a very rough estimation on the helium leak rate through a sealed assembly. It is exposed to pure helium gas at atmospheric pressure on its outside, and it is evacuated inside (see Fig. 18). We assumed (see Fig. 7):

- as effective surface A : $\pi \times$ mean seal diameter \times seal thickness.
- as effective thickness d : the seal width. So we assumed that the entire seal arrest surface, not only the nose, was sealed off!!! The ‘estimation’ looks very much like a speculation!

The measurements to compare with were carried out on the ‘small device’ with spring-loaded bolting (see Fig. 14).

Figure 19 shows the measured helium signal (dots/crosses) and the ‘estimated’ leak (solid line).

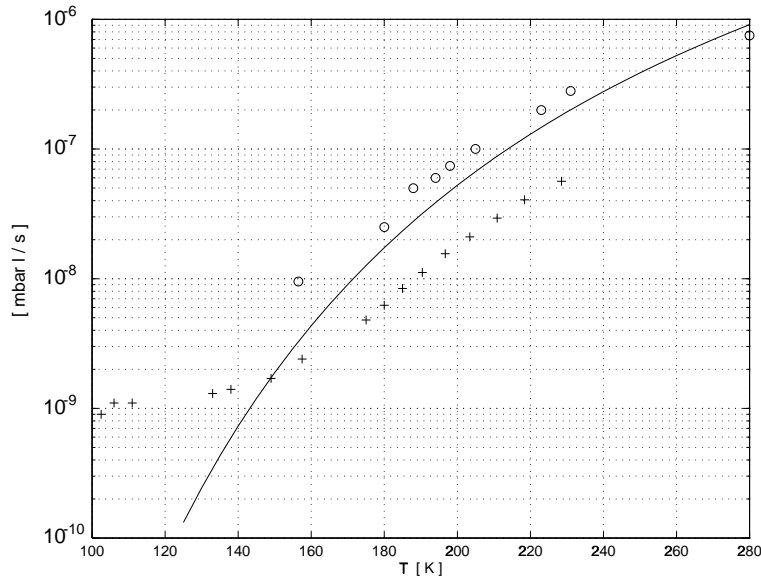


Fig. 19: Helium leak rate, estimated from PC permeation data, through a seal (solid line), confronted to the measured leak detector signal (dots/crosses). The measured signal is in fact the sum of seal permeation and (parasitic) natural background of the set-up. Seal dimensions: 36 mm inner diam., 46 mm outer diam., 8(!) mm thick. Beware the extreme loading: pure helium at atmospheric pressure!

The first test (dots) was characterized by a 1×10^{-8} mbar l/s natural background. For the second test (for which a new seal was mounted - crosses) we installed a leak detector with a pumping

unit of higher performance. This resulted in a 1×10^{-9} mbar l/s set-up background, enabling us to graze somewhat deeper. The seals were chosen very thick in order to boost the signal. Nevertheless it proved useless to descend deeper than some 120 K.

The 10^{-6} mbar l/s order of magnitude at RT seems huge, but one must keep in mind that this is the response to very artificial, extreme conditions. Furthermore, this response was very slow, very progressive: it took about a working day to reach the 10^{-6} steady-state value.

We have to mention, without going into details, that there are many potential sources of trouble when carrying out such an extreme test.

7 PRODUCING LARGE-DIAMETER SEALS OUT OF A ROD

For diameters above one metre, it becomes impractical to machine a complete seal out of one plate, if the plate exists at all! So one would have to weld pieces together. A practical solution would now consist in starting from a piece of long rod, with the final seal cross-section, and bending it to a circular segment.

7.1 Rod deformation

The material's ductility (see Fig. 4) enables a plastic deformation (roll bending) at RT. It turned out convenient to work on a bending press with four rollers. Two operators are necessary when no additional rod guiding tool is available, in order to avoid snap-and-twist. Rate-depending yield strength, visco-elastic and rate-depending anelastic problems, etc. make it impossible to get a product of well-defined geometry out. Figure 20 gives an impression of the problems: the product is 'alive'.

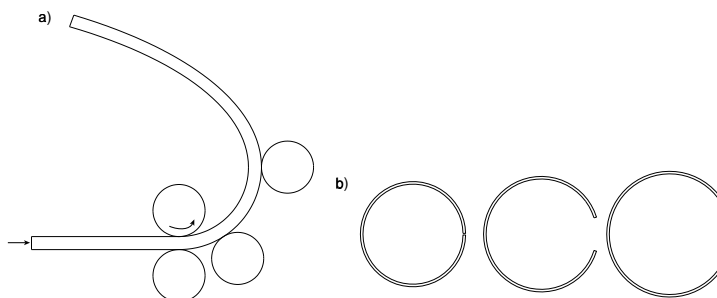


Fig. 20: a) Plastic deformation of a rod on a bending press with four rollers. b) The rod's behaviour afterwards: after minutes/hours/days.

This behaviour does not really jeopardize good seal making, one just has to adapt to a job which is quite different from metal working. We had the unfortunate reflex of rolling on very small radii of curvature, to pre-compensate for the relaxation. We thus imposed a significant (plastic) strain on the innermost and outermost material 'layers'. The strain is actually somewhat bigger at the pulled outside than at the compressed inside, because polymers have a lower yield stress in tension than in compression, often encompassed in a pressure-modified Von Mises yield criterion. Shear bands — under $\pm 45^\circ$ (see Fig. 5) — were the logical result of this excessive straining, Fig. 21. The edges should have been rounded before the rod was bent.

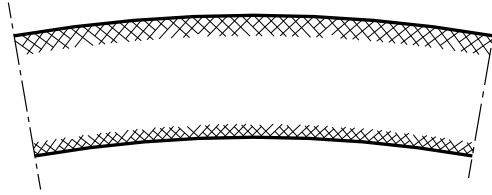


Fig. 21: Potentially dangerous shear bands after bending, pronounced at the outside. Extreme rolling curvatures and sharp product edges are the causes.

A more gentle way of limiting free relaxation would be to put the product into a ‘jacket’ right after rolling, and to limit the excess of deformation during rolling. Furthermore, a very low bending-press speed would be convenient.

The rod’s terminals are to be cut away after forming, the length to be cut off being two roller diameters at least. In practice, the position at which to cut away will of course be determined by the desired circumferential length. Furthermore it is important to have flat end faces, perpendicular to the rod’s axis tangent - see sections on welding. Execution by milling, with the help of the jacket, seems the most adequate solution.

Summarizing, we can state that segments can indeed be realized, virtually without capital costs. We have also learned how things should NOT be done. Remark: bending at elevated temperatures is believed to give more trouble than gain.

It remains now to join the ends together. Each of the two following paragraphs describes one specific welding method.

7.2 Methylene chloride welding

This process looks much like gluing, and indeed it is often mentioned under a ‘gluing’ header. This is not correct from a physico-chemical point of view. Elsewhere, the joining method is sometimes referred to as ‘solvent cementing’, another rather unfortunate term. Methylene chloride (CH_2Cl_2) is a highly volatile liquid, which dissolves a number of amorphous thermoplastics, among which is polycarbonate. Ethylene chloride (less volatile) and chloroform are other dissolving agents, albeit not quite as powerful, and so we haven’t used them. If the surface of a PC product is wetted by a dissolving agent, then a thin layer of polymer molecules gains mobility, is liquid in some respect. But the agent disappears very soon from it, and the layer returns to a solid. The surface has changed in appearance, because it had been ‘melted’. More specifically, most often there is bubble formation.

The step to a method of joining two pieces together is evident. The contact area is temporarily ‘melted’ (actually dissolved) after which the assembly solidifies, polymer molecule cohesion being re-established. The strange substance (the dissolving agent) disappears (more or less), which is why ‘welding’ is the most appropriate term.

It is however not so evident to produce high-performance welds, and this in a reproducible fashion. Methods as usually described in material application brochures, have not been retained. We have instead followed the strategy developed for the realization of transparent targets for BEBC (the Big European Bubble Chamber):

- Butt welds only: contact area flat and perpendicular.
- Do not wet either of the two pieces to be welded.

- Hold a third object, a ‘leaflet’ of PC (sheet 1 mm thick), in a bath of solvent for typically half a minute. The leaflet is now more than wet: it is virtually saturated, and weak!
- Remove the leaflet from the bath and place it rapidly in between the two pieces to be joined.
- Push the assembly together hard: at least 600 N per cm² of contact area. Again, do it very swiftly. Clearly, this has to be done in a press.
- Leave the assembly in the press, under load, for several hours (6–12).
- Let it dry and relax for a few weeks (at least three).

Speed is important: pressure must be applied within 15 s of removing the leaflet from the bath. One could dream of cold solvent and cold products, but an enhanced moisture pick-up would normally be the result, unless one works in a conditioned space. This had been done for the BEBC targets, keeping humidity at 40% relative. An investigation on the influence of water was beyond the scope of our project. Whatever may be, working within the quoted time limit is feasible for seal production. Risks of bubble formation are at the origin of requirements on speed and pressure.

The leaflet is sticking out on all sides. In principle, the welded seal would now be entirely machined, in order to obtain flat surfaces. We have, however, tried to keep the rod(s) intact, so as not to machine, and to treat the weld zone by hand instead. Most of the surplus material left by the leaflet is cut away with a sharp pair of scissors, and the finishing is done by filing. With this filing, we also smooth the left-to-right transition due to thickness/width variations in the rod(s). The resulting seal geometry is not perfect, and therefore one would apply somewhat bigger (deeper) flange noses than would be the case for machined seals. The leaflet bits have indeed to be cut away gently, and not pinched off: the violent blast of the latter creates shock waves and cracks.

Figure 22 depicts the different stages in the process.

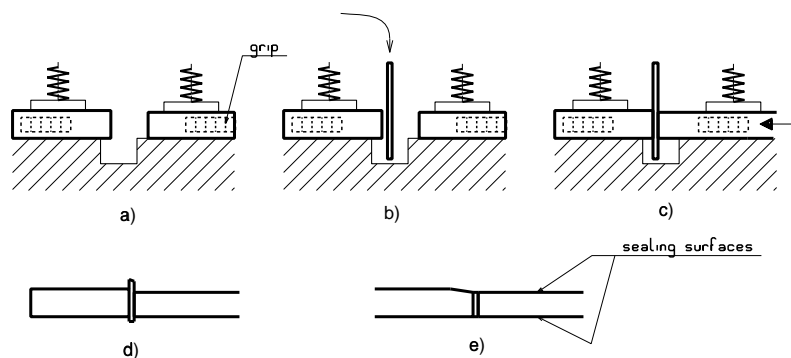


Fig. 22: Methylene chloride welding (the seal’s rotation axis is vertical): a) align and lock the rod ends in the press, open the press; b) insert the wetted leaflet in the gap; c) close the press and apply pressure; d) cut away the pieces of leaflet sticking out; e) smooth the surfaces with a file.

Evidently, the sealing surfaces at the weld zones would be longitudinally polished with fine sandpaper: a swift and non-delicate operation. However, we have rather made the weld

zones transparent (again), by putting a droplet of methylene chloride on the surface and pushing a ‘mirror’ against it, again very rapidly. The result is not very predictable, and it serves no purpose for seal making. It did enable us, however, to examine the welds ‘optically’. We had apparently been very successful in avoiding bubbles. On the other hand, tiny cracks, longitudinally oriented, were sometimes visible - at least under the microscope. They occurred in the very thin cross-section layers which had been effectively wetted by the solvent during welding, Fig. 23.

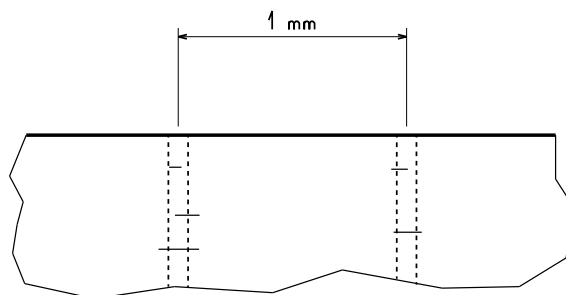


Fig. 23: Tiny cracks at the interfaces, at or near the free surface.

If the weld is less strong in tension (it mostly is) than the base material, then eventual failure will occur at the welds. The bulk of the product (base material) will at that instance not have undergone any appreciable plastic deformation. This is a consequence of the strain softening behaviour, see again Chapter 3. The failure would be brittle from a mechanical viewpoint. (We cannot dream of rolling a welded rod!) This handicap is not dramatic at all in sealing applications. Once the seal is ready, it would not be exposed to extreme tensile straining. However, we do have to cope with a certain tensile straining when cold, namely about 1%, due to cool-down shrinking imbalance, see Chapters 2 and 4.

Tensile specimens have been prepared with the welding press which was also used for seal production later on. They had the same cross-section and the same surface finish as the seals. Some were broken at RT, others in a LN_2 bath. On average they showed acceptable performance, yet there was a noticeable and somewhat worrying spread in the results. The three seals prepared with the necessary care all survived (see Chapter 5). Another one, which was mounted hardly two days after welding, did indeed break on the very first cool-down attempt.

Compression has proven unproblematic: the weldings showed no signs of suffering from the flange nose indentation.

Clearly, the whole process is not suited for large-scale production. However, the technology exists, is operational, and can be deployed with very low investment costs.

Nevertheless there is still room for research on performance improvement. Indeed, there is evidence that too much solvent remains captured in the core of the welded interface — even after months at RT for $6 \times 10 \text{ mm}^2$ cross-section. This contamination would give problems (bubbles) in a bake-out. There is evidence as well of the existence of a serious biaxial tensile stress state, confined very locally to the cross-sections which had been wetted (the leaflet). The reason for this is probably a swelling of the leaflet during solvent absorption. Upon drying, it wants to shrink, which it cannot do freely because now it is ‘frozen’ onto the base material. There is little hope of relaxing these stresses in time, due to the very high creep resistance of PC at RT.

7.3 Dielectric welding

A more classic welding approach, through real melting as a result of heating, can be attractive if the heated zone is not too local and sharply defined. A very concentrated melt zone, with sharp temperature gradients in the heat affected zone, may and will cause internal stresses.

Conventional thermal welding methods such as heated mirror and hot gas can be ruled out: the product quality would undoubtedly be insufficient. The geometry of our seals forces us to exclude ultrasonic or friction weldings.

Realizing diffuse and controllable heating (melting) in an interface to be welded is almost impossible with any of the classic thermal welding methods. Dielectric heating is, in the author's opinion, the only valid candidate.

A firm, triaxial compression of the interface zone during and directly after heating, seems and has proven to be essential. The pieces have to be narrowly enclosed by a tunnel-like jacket, and they have to be pushed axially:

- to have good interface contact during warm-up before melting;
- to avoid (humidity) bubbles, see Chapter 2;
- to limit risks of void formation in the very hot core of the material — this is a consequence of cool-down shrinkage: material has to be ‘filled’;
- to limit the reduction of area in the welded zone due to shrinkage (quite comparable to the application of post-pressure in injection moulding).

Dielectric heating is power dissipation within the material as a result of damping effects in an oscillating applied electric field. A treatise, even introductory, on this exciting discipline is beyond the scope of this text. Suffice it to mention the formula for the specific power dissipation:

$$\frac{dP}{dV} = \frac{1}{2} \hat{E}^2 \omega \epsilon_0 \epsilon_r' \tan \delta$$

in which dP/dV = heat generation per time unit, per unit volume of material;

\hat{E} = amplitude of applied, oscillating electric field;

ω = angular frequency of the harmonic oscillation;

ϵ_0 = vacuum permittivity;

ϵ_r' = material's relative permittivity (more precisely, the real part of the complex permittivity);

$\tan \delta$ = material's ‘loss factor’, a measure of the energy dissipation per cycle, and this in relation to the ‘stored’ energy.

The most straightforward dielectric heating/welding method is the capacitive: the objects to be heated are put in between two plates, constituting a ‘capacitor with dielectricum’. The method is popular for welding PVC foils together. The low-frequency handicap (typically 27 MHz) is compensated for by high-voltage amplitudes and narrow plate gaps. Our seals are fairly thick objects; furthermore we would need a jacket around them. Capacitive heating has not been retained as the appropriate method.

The Parts Processing Team of Philips' Centre for Manufacturing Technology suggested performing a number of welding tests with their microwave equipment. The electrical component of an electromagnetic wave, generated in a ‘cavity’, is used for dielectric heating [7]. The

domestic microwave oven works on this principle, the water in the food often being the main dielectrically dissipating substance.

Figure 24 depicts part of the set-up, which fabricated simplified specimens, not entire seals! The cavity is of the TM_{010} type, yielding an axial electric field, the amplitude of which is constant along the axis, and varies radially according to a Bessel function of first type / zero order, with its first zero at the cavity wall. These relations are evidently valid only inside the (empty) cavity. The cavity is made out of aluminium, and can be readily machined out of a block. The resonance frequency of the first fundamental standing wave — this is the mode in which the cavity is employed — depends on the cavity's dimensions, but also (somewhat) on its loading. It has to be tuned — with reduced power — before the actual welding operation commences. It is of the order of 2.45 GHz. The covers are dismountable in the Philips' cavity, for obvious practical reasons. The electrical contact there must be good, because the microwave current passes through the screwed interface. One could, for seal welding, design a cavity which opens into 'dees', giving a non-critical interface.

Polycarbonate doesn't seem to be a material with very high losses, and literature on its dielectrical properties is scarce. One must keep in mind that those properties are essentially dependent upon both frequency and temperature. In any case, more than enough power turned out to be available: one minute at full power is sufficient to thermally destroy the PC.

Axially, things are just the way we want them to be. The field is about constant over a long distance - about the cavity height. Nevertheless, there will be an axial temperature gradient with a maximum in the cavity centre, due to heat conductance and — more importantly — thermal inertia effects. Melting sets in at the centre, and a broader, diffuse melt zone is obtained when heating is continued, at a lower power level.

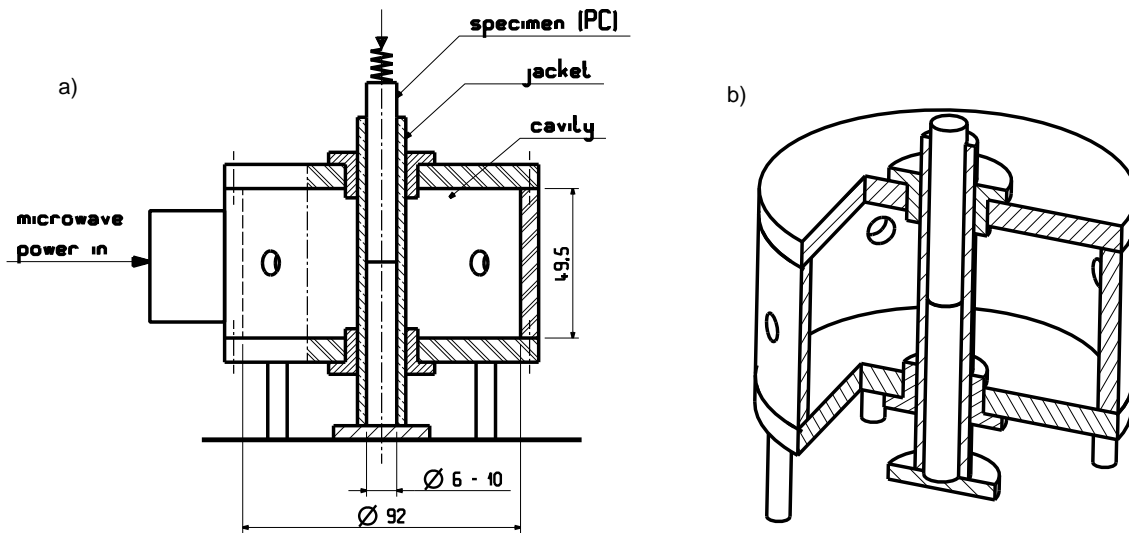


Fig. 24: The microwave cavity, loaded with a specimen-in-jacket. A few little holes in the cavity's mantle permit the process to be monitored with the aid of a video-camera, if the jacket is transparent. External compression of the specimen is essential.

The main problem to solve with this technology is that of the radial gradients. The material deep inside, near the axis, suffers from over-heating, at a moment when the surface may not have even started to melt. Filling the cavity with a dielectric load (PC + jacket) modifies the field distribution in that the maximum at the axis becomes more pronounced. Moreover, if

the jacket is made out of low-loss material (it usually is), then the PC surface is handicapped additionally, because it transmits heat to the cold jacket.

The combination of jacket and external compression has proven effective in avoiding humidity bubbles. However, the assembly clearly tends to develop a ‘bell’ at or near the hot spot. The reason could be our failure to supply material to the melted zone, supply prompted by solidification and cool-down shrinkage. The required compression could turn out to be of the same order as that for methylene chloride welding.

The transmitted power has to be varied during the welding process; it must be sharply diminished after core melting has set in.

At present we are trying to pre-heat the jacket with a polyimide ‘glow plug’. In any case, an ideal jacket material which would

- be hard and rigid at temperatures up to 320°C at least;
- have a very low thermal expansion coefficient;
- have very low thermal conductivity;
- have significant dielectric losses, or, if not, be dielectrically ‘inert’ ($\epsilon_r = 1$) AND have low thermal capacity;
- preferentially be optically transparent,

has not been found yet.

Our research has been very limited, but, in spite of the difficulties encountered, this method would undoubtedly be a serious candidate in the case of series production of large PC seals.

8 WHY POLYCARBONATE?

There is absolutely no proof that other polymer materials would NOT work.

Below we list the reasons why we considered PC to be a good compromise:

- Thermosets are hard and intractible; they are to be ruled out.
- Elastomers (rubbers) are in their rubbery state at RT, but upon cooling down they inevitably get glassy, hard, and prone to cracking. Moreover they shrink a lot.
- Most of the familiar semi-crystalline thermoplastics have their (main) glass transition temperature below RT. This means that the amorphous regions in between the crystallites are in their rubbery state at RT. The consequences are good deformation capabilities and toughness, again at RT! Below the glass transition temperature they usually become very brittle; polymer crystals have (far) fewer slip systems than cubic metal lattices. They are also quite stiff. Below 100 K, POM, the polyamides, HDPE, and even LDPE and PTFE have higher moduli than most of the amorphous thermoplastics (such as PC) [3].
- There is not much choice among the amorphous thermoplastics. PS and PMMA are brittle; PVC is less tough than PC. Moreover, PVC is no longer very fashionable with respect to fire safety.

- PC has been intensively studied in terms of deformation and fracture, even at low temperatures. This is not necessarily true for other polymer materials!
- The high temperature resistance of PC would enable a modest bake-out of sealed assemblies (bake-out tests were beyond the scope of this project).
- Its transparency enables an optical quality control (and an optical monitoring of microwave weldings).

The main topic of care with PC is its tendency to develop strain crazing.

Poly(butylene terephthalate) (PBTP) could perhaps be studied as an alternative material.

9 OUTLOOK AND SUGGESTIONS

- Even more severe long-term testing is needed in order to increase confidence with respect to strain crazing.
- Only high molar mass material should be used. Attempts to injection-mould smaller seals should be discouraged; it would be better to machine them. Once more, the edges must be rounded! For larger seals, standard rod material would have to be made available, extruded on the final dimensions, and with rounded edges. Ideally, very long rods would be available, necessitating only one weld.
- The total radial force, with which the flange nose keeps the seal in place in spite of thermal expansion coefficient differences, is proportional to the seal's cross-section area. It could pay off to limit/minimize this area somewhat.
- Elegant 'springs' for the bolting of high-end applications have still to be found. The existing umbrella washers, which fit to bolts of reasonable diameter, are largely insufficient: their stroke is too small, and so is the force needed for total squeezing.
- The nose geometry (shape) will have to be optimized. Instead of the used $-40^\circ/0^\circ$, one could try $-20^\circ/+20^\circ$, in the hope of reducing the crack tendency of Fig. 17 whilst still keeping the seal in place during cool-down. Furthermore, it is questionable whether the dips at the nose's roots were such a good idea after all, especially in combination with spring-loaded bolts.
- Performance of methylene chloride weldings may increase after a careful annealing treatment. The welded object would be put in a tunnel-like jacket and compressed axially. The jacket's centre — where the weld is — would be heated, but its ends would remain below 120°C in all cases. The jacket's centre temperature would be increased slowly, from RT up to some 120°C , with firm compression. This permits CH_2Cl_2 outgassing. Afterwards, the compression force would be lowered and the jacket's centre would be heated to above PC's glass transition. A very efficient stress relief is the logical consequence. As an extra bonus, one would now make more profound molecular entanglements around the interfaces [8]. The tiny cracks reported upon (Fig. 23) might have a fair chance of healing, see again Ref. [8].
- Clearly, it would be nice to produce real seals with microwave welding. If the jacket has to be out of one piece, it could be broken afterwards.

- The influence of pump-oil contamination on strain crazing needs to be examined.
- Is it conceivable to transfer the experience gained in this project to cryogenic valves?

10 CONCLUSION

Our objective is believed to be within reach.

Ductility and transparency of PC are exploited. The increase in stiffness and hardness on cooling down is relatively modest, and this further advocates the use of this material. Its main mechanical limitations (poor ductility at very low temperatures, notch sensitivity, strain crazing) have not adversely affected our application, and thus hopefully remain of no concern. Its high glass transition temperature would probably permit a modest bake-out.

The tightness profile (‘nose’) on the flange is rather coarse, and this relaxes requirements on both seal and flange (surface and material imperfections). The nose seems to adequately keep the seal in place in spite of the latter’s important thermal expansion coefficient. Consequently, the seal can easily pair flanges out of different materials. Tightness is not perturbed during or after a cool-down quench. The hydrostatic component of the mechanical stress state in the squeezed seal is predicted to be compressive. The remaining ‘bounce back’ elasticity in the compressed seal is very attractive compared to classical solutions. It more than compensates the through-thickness thermal contraction of the PC seal. We have proven that a sealed assembly can indeed be operated without additional flexibility in the bolting equipment. Compression forces are important, yet compare favourably to conventional cryo-proof bolted assemblies.

The tightness quality is limited, at least at room temperature. The revealed gas permeation rates are typical for amorphous thermoplastics. However they decrease rapidly with decreasing temperature. High-vacuum quality sealing can easily be achieved as from 120 K (LKr) downwards.

A cost-effective way of producing large-diameter seals has been demonstrated. With the necessary care cheap rod material can be rolled to obtain circular segments, or ultimately, a complete ring with only one dislocation. The ends can be butt-welded with a methylene chloride based process pioneered at CERN. Seals have been realized in this way and have shown acceptable performance. Another technology, dielectric welding in a microwave cavity, seems to offer very attractive perspectives.

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