What is required to Engineer, Test and Qualify Swelling Elastomers Seals.

Whilst swelling elastomers have gained and continue to gain general acceptance as seals in Oil and gas wells little is known by the industry on their development and the necessary requirements to create engineered and tested elastomers and seals. This document was prepared to show what was done to engineer elastomers and seals at Ruma Products for its OBLIQUE series of swelling seals.
Swelling Elastomer Seal Development Standards and Tests

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Preamble
This brief overview was created to illustrate the various phases required to engineer firstly an elastomer and then the swelling seal that would use this elastomer in an actual well. It will touch upon the various steps in the process, the various international standards used to qualify various tests undertaken and lastly the quality requirements on the final product. This requires the whole aspect to be split into basically 3 fundamental sections.

1. Elastomer Engineering. Here the elastomer is engineered to meet fundamental requirements such as operating temperature envelope, motive fluid specification, swell requirements.

2. Seal Engineering. By simply making a seal swell it does not follow that it automatically will seal. Seals need to be engineered to create the desired effects and create the seal operating envelope.

3. Manufacture. While seal engineering determines the shape or form of the seal, manufacturing procedures and production equipment has to be created to ensure that the final product complies with all the requirements (physical as well as quality) to ensure compliance to the desired.

Ruma Products (Oblique is the trademark of one of the product ranges of Ruma Products) is unique in the industry in being able to design and compound its own elastomers, design its own swelling articles made from those elastomers and then have its own facility to manufacture these articles. This uniqueness outs itself in the fact that we are ISO 9001-2008 Certified for swelling seals. The knowledge required to do this and the test facilities are all in house as is our research and development programs.
Elastomer Engineering.

Elastomers are complex highly viscous hydrocarbon based materials. They come in various very specific forms such as EPDM, NBR, HNBR etc each having unique specific characteristics. However in its pure raw form the compound is not a useable product. It is comparable to the flour used in baking. In baking you have to add ingredients to the flour to create an edible tasty product. In reality just adding the ingredients to the flour does not make an edible tasty product. It has to be mixed correctly and then baked to create the final tasty product. Elastomers are the same. The raw elastomer has a number of different chemical ingredients added to it which after baking (called vulcanisation in elastomers) give a mechanical or chemical characteristic to the end product.

Each of these ingredients inter reacts with either another ingredient or with the base elastomer to create the desired effect. Whilst an experienced compound engineer knows what the potential effect of the various ingredients can be the final product still has to undergo a plethora of tests to monitor the mechanical and chemical properties of the compound. These ensure the created result has both the mechanical strengths as well as the ability to withstand the effects of time and operating environment.

The tests required and used are the following

- Swell Buttons Test (same as Chemical Resistance)
- Flow characteristics Mooney: NEN-ISO 289-1, measure the Mooney-viscosity of uncompounded or compounded rubbers, this is used to give information on the mechanical characteristics requirements to be able manufacture from the compound.
- Vulcanization (rheometer): DIN 53529-T3/ISO 6502, measure the vulcanization-characteristics; the change in mechanics of the rubber as a function of time at the required temperature.
- Vulcanization product: DIN 53505/ISO 868, determination of the mechanical characteristics of vulcanized rubber on flat surfaces.
- Temperature responding DMA: NEN-ISO 6721-11: Determination of dynamic mechanical properties including the glass transition point at different temperatures and conditions.
- Tensiometer at different operating temperatures: ISO 37: Determination of the change in mechanical properties (such as tensile stress-strain properties) at different temperatures. This enables engineering data be gathered and used in the design of articles made of the compound.
- Bonding and Bonding Engineering Development: internal test (see Work-instructions Oblique page 27). Bonding is the chemical gluing of the elastomer to a substrate. This can be anything from Glass-fiber to exotic metals. Depending on the metallurgy this can be many different bonding agents for the same rubber to many different base metals. It can be up to three or four different layers of agents.
- Wear Resistance: ISO 34-2: Determination of tear strength
- Aging: ISO 188: Accelerated ageing or heat-resistance tests
• Chemical Resistance: NEN-ISO 1817: Determination of the effect of different liquids on the mechanical properties of elastomer and their aging effects at different temperatures.

All of these tests enables us to understand how the elastomer will react to the manufacturing process and then later as an article in well conditions and how the elastomer react to the process of manufacturing and use.

**Elastomer Swell Engineering**

Because the elastomer is being required to swell it needs to have specific ingredients to create the diffusion gradient that creates the swelling. Whilst we talk about swelling in the oil field in water and/or oil. Different forms of swelling are available outside of these two, for example in Caesium Formate Brines, Acidic fluids etc. For the two common motive fluids the swell has to be engineered as it the interaction between the different elastomers and the motive fluids under the influence of Temperature. These result in a huge test programme based upon multiple samples to create

• Swell Repeatability. Because the elastomer consists of a collection of ingredients the quality of which together with the tolerances used in their measuring and mixing can result in variations in product performance. Tests are conducted to ensure that tolerances in the process are in place to ensure that the results are not different per batch of elastomer mixed.
• Swell Curves: this is the shape of the swelling versus time at differing conditions and temperatures.
• Elastomer Swell Envelope: In swelling the salinity and the temperature inter react to create a three dimensions envelope inside which the swelling characteristics can be used to create sealing.

**Water swelling Elastomers**

No Formalised standards exist for swelling elastomer swell measurements. Our in house program was created to measure the swell in a standardised way, to ensure swell repeatability and enable swell engineering data to be created which forms the backbone of the sealing engineering database. Put simply if you cannot measure it correctly you cannot engineer the desired solution.

**In-house test for Water Swelling Elastomers.**

We measure the hardness, volume, weight and dimension changes of:

• Elastomer Discs (for volume swell), 35 mm diameter and 6 mm thickness
• Elastomer on Plates (for thickness swell), 13 mm rubber on 3 mm steel plate, size 5 x 5 cm

We place this samples in controlled saline solutions, varying from De-mineralized water in steps up to saturated brines, and under different temperatures from 20°C to 250°C in steps of 20C.
Each of the sample sets will be measured on volume, weight increase and dimension changes after set exposure time period of 1, 3, 7, 14, 28, 60, 100 days for each of the salinity steps.

After every measurement step we refresh the brine solution as this alters slightly due to the effects of swell.

This enables us to make produce accurate swell curves, with the time on the x-as, and the volume-change (or for engineering the thickness-change) on the y-as. This is an example: Which shows the types of effect on swell given the influence of temperature for a given brine with various temperatures against time.

![Fig 1 Typical swell curves](image)

**Fig 1 Typical swell curves**

Fig 1. The resulting hundreds of different points enables the three dimensional data to be integrated into an engineering model that is used for seal design.

The biggest influence of swelling is that of the vulcanization and the quality of the rubber being created. This requires considerable elastomer understanding and working. There are two consequences to incorrect vulcanisation.

1. If not vulcanized enough the correct cross linking will not be achieved resulting in a substantially reduced mechanical characteristic

2. If over vulcanized the elastomer aging occurs and this can result in substantially impaired characteristics.
Imagine it in the same context as baking, if under baked the ingredients are raw, if over baked oxidization occurs and the burnt product is uneatable. This is exactly the same for elastomers.

**Heat Survey: (for an example: see Work Instructions Oblique, page 24)**

During the development of a water swelling elastomer we perform a heat survey on the rheometer to determine what the vulcanization program will be for vulcanizing the elastomers in the autoclave.

![Heat survey rheometer graph](image)

**Fig 2** Here you see an example of a heat survey on the rheometer.

Note: blue line is the vulcanization curve of the elastomer. Green line is the programmed temperature increase.

The above graph illustrates how the temperature build-up of an autoclave (this is the oven that the elastomer is “cooked” in) builds up and is modelled by the rheometer in the green line. The blue line is the measured effect of vulcanization that the rheometer actually measures.

**Elastomer High Temperature Seal test Unit:**

*High temperature rubber swell tester:* the purpose of this equipment is to determine, at a maximum temperature of up to 300 degrees Celsius and a maximum pressure of 200 bar, the
swelling characteristics and the basic inherent sealing quality of swelling rubber. The misconception is that because it will swell it will seal. All we can say for certain with a swelling elastomer is that if it does not swell it doesn’t seal. How well the elastomer will swell at elevated conditions depends upon a number of factors designed originally into the elastomer. Things like bonding, tear strength, swelling characteristics etc. all interact with each other and it is this interaction and the compromises made with them that determine things like how well it seals. How long it will remain sealing and how it ages under sealing are very important elements tested for. The more extreme these conditions, the longer it takes to develop the base elastomer, the optimum degree swelling of the elastomer and the resulting desired characteristics.

In house we place a steel bar fitted with a water swelling elastomer into a high pressure vessel. A temperature is chosen (max 300°C), the test salinity selected and the amount of swell that the elastomer needs to swell to simulate seal off. The elastomer will then start to swell and will seal off. Once seal time has been achieved to seal off the pressure will be increased (max 200bar) until the seal starts to leak. Note that these are compound tests not product tests such as a swelling seal, they give insight into the elastomer mechanics not sealing.

By monitoring the point of the pressure increase until it fails.

Here is a figure with some dimensions of a test sample that goes in the pressure vessel.
Note that these dimensions are not created or geared to suit the well, but are created to enable a temperature pressure cycling of the elastomer and understand how the variables interact with the elastomer over time.

**Shape of the Elastomer**

The physical shape of the elastomer has a major impact on the sealing characteristics of the seal. We have a number of confidential ways (I hope that you will understand why we would prefer not to share with you in writing) that we use to look into the difference a shape has on the effects of swelling and also then sealing that this would create. These help us to model the design of a seal for a customer’s requirements.

**Full Scale Testing**

In all of the above the goal has been to create a wide envelope elastomer and determine its swelling operating envelope. However swelling is not sealing, to be able to create a seal we need to look into how well the elastomer will seal and by looking at the relative performance determine how we need to develop the elastomer to seal. We look at depth into the effective sealing so as to modify the operating envelopes and then using the physical mechanical data to design a seal to suit well conditions. Once all of these parameters are known we have a computer automated tester that will then robustly allow us to test seals up to 4.5 inches at elevated conditions to qualify the seal made from the qualifies elastomer. The unit is computer controlled to enable it to conduct pre-determined test cycles without operator input safely 24/7, the cycles being up to 300 Celsius.
Manufacture

The last phase of all the work is to manufacture products for customers all based upon the above development work. It is as vital a part of the development cycle of a seal as the development itself. The best developed seal is of little importance if the manufacturing system is sub-standard or flawed. Our system consists of the following:

- ISO Certification of Oblique and the procedures and processes.
- ISO Certification material Suppliers, all our raw ingredient suppliers are ISO certified.
- Supplier Auditing, We have an Auditing program for our suppliers to ensure compliance to our requirements and procedures.
- Oblique Quality Plan. All our products are manufactured in accordance with a formal Quality Plan. In this quality plan each phase of manufacture is via work instructions along with a barcode monitoring system.
- Pipe Shot blasting Requirements. Procedures are in place that manage the blasting to suit different metallurgies of base pipes/sleeve cores etc.
- Bonding Coating requirements. Procedures are in place that enable different elastomers to be bonded to different metallurgy pipes, even where multiple elastomer seals are applied to the same piece of pipe.
- Elastomer pre Use QA/QC requirements. Each elastomer batch (elastomer is mixed in batches of 500kg) is tested for quality compliance before it is authorized for use and is then placed into the stores. Prior to it being used in manufacturing on a product a second series of tests is done (including swelling) to ensure that the elastomer still complies to requirements after storage. The elastomer is authorized for use after is complies with the requirements, before it can be used in manufacturing. Every batch of elastomer has a sample removed and stored in a deep freeze (this step gives an unlimited shelf life) for future reference if tests are required for future reference on articles used in wells.
- Bonding Agent QA/QC: All bonding fluids are tested and supplied with batch traceability prior and during use. (Note: All bonding used is Lead (Pb) free, no bonding containing Lead is used)
- Vulcanisation checks: The computer that is programmed for the autoclave vulcanisation management program creates and stores the actual standard vulcanisation curves and values. The autoclave is tested weekly for correctness of values and is fitted with secondary measurement equipment to confirm the primary control values.
- Bonding Tests: every batch of products going through a vulcanisation is fitted with destructive samples for bonding confirmation.
- Product QA/QC checks: All articles are checked and measured for dimensional correctness and are dimensionally recorded on certificates together with an individual serial number per item. These certificates have to be signed off before release for shipping. Copies of all certificates are stored for ten years for future reference.
- Equipment QA/QC. All equipment, measuring equipment, lab equipment etc. area part of a monitoring system where certification is managed and maintained for calibration and other purposes.
• Facility Audits: Management conduct random facility audits as routine. Additionally as part of the ISO certification the facilities, data management and data storage are audited as part of the re-certification and Certificate renewal.
• Full Traceability: All produced articles are fully traceable from batch elastomer to delivery to customer.
• Step Manufacturing procedures: Every step in the manufacturing process whether it is raw elastomer to loading on the transport is covered by procedures.

Customer Seal Design

Whilst all of the above deals directly with a seal at some stage of its creation, a very important part of the seal is how it is actually being used. The customer has a requirement and this needs to be met. Whilst it would be nice if one size fitted all and one elastomer fist all applications this is an impossible dream. In most cases a degree of optimisation of designs is required. This is for a number of reasons. Firstly no two wells are the same and secondly because of differences in hole sizes.

Seal centring

With all swelling packers contact with motive fluid will cause swelling to occur radially increasing the element diameter over time until it contacts the bore of the space it is contained in. In both vertical and horizontal applications it is almost certain that the seal will NOT be central to the bore hole. In a vertical application because of effects of gravity it will be potentially closer to the centre of the bore hole. In a horizontal application the seal will almost certainly always be on the low side of the hole. This is compounded by the fact that in horizontal applications the bore trajectory can porpoise as well as corkscrew on its way through the various layers. These all effect the relative position of the seal to the bore in the length direction as well as in the radial directions.

To aid centring in both Vertical and Horizontal positions our seals are supplied with 2 running guides, they function to a degree as Centralisers as well as protective guides during running.
The larger the guides diameter the better they help the seal centralise in both horizontal end radial conditions. When the seal is not centred the lower section is not swelling correctly and the top has to swell more to compensate for this. In the

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<th>inch</th>
<th>Base Pipe</th>
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<td>Rubber OD</td>
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<td>inch</td>
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<tr>
<td>Running Guide OD</td>
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<tr>
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<table>
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<th>Calculations</th>
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<td>6,5</td>
</tr>
<tr>
<td>Apparent washout size</td>
<td>8,25</td>
</tr>
</tbody>
</table>

Table 1

Table 1 is an illustration of the changes caused by low side offset on what we call *apparent hole sizes* that need to be taken into account with swelling seal design. It is only intended as an illustrative table to show how crucial the effect of centring could be, but indicates the effect of low side lay and different size of running guide can have because of the centraliser effect.

The discussion above centres on the mechanical condition and sizes existing in the bore. However this is only a half of the full discussion, the physical mechanics of the chosen elastomer are the second half of the situation. The diffusion gradient existing in the elastomer determines both the speed of swell (time to first contact) as well as the time to full seal swell. A rule of thumb is the hotter the motivation fluid the quicker and greater the swell, the more saline the fluid the slower the swell. Temperature and salinity are therefore crucial in the selection of the correct elastomer type as well as the correct OD of the seal.

**Customer Engineering details**

As a part of the design process it is important for us to model the intended use, relevant well parameters and details to determine the conditions, swelling that is required and times involved. This is important because often different elastomer operating envelopes overlap. This enables us to select the most effective elastomer envelop for a given well/Field and to dimension the elastomer to be the most effective for that well as well as then design the dimension of the end rings to suit the centralisation of the seal.
Fig 3. This is Data sheet customers are requested to fill in so that we can look towards creating the correctly dimensioned and designed seals for.
Seal or Packer

One of the most complicated problems with swelling seals is that they are in the mind’s eye of many users, packers and in fact most suppliers are selling them as such. Whilst they do actually fulfil the function of an open-hole packer they cannot be full addressed as packers. Packers qualification is covered functional by ISO 14310. It is a document developed to standardise the testing and qualification of mechanical packers. Its function is to standardise all the potential tests done.

Mechanical Packer Testing

History:

A little over 10 years ago a new standardized testing document was created that looked past manufacturers and operating companies own, often very different and conflicting, testing requirement of mechanical packers. The idea of this document was to still have a fit for purpose testing criteria but to define the severity of the purpose for the testing as well as the severity of the testing. This new document ISO 14310 was intended to do that. It combined a pragmatism approach with engineering to arrive at a set of meaningful tests and understandable results.

Mechanical packers

Conventional mechanical packers consist of two important section, the slip system and the sealing system. They work by mechanically energizing the slips to both hold and lock the packer in place as well as to compress the elastomeric seal deforming it, and through this deformation creating a seal. In turn the seal being deformed now also functions as a sort of spring and helps to lock the setting force in place and help to lock the slips in place. How Oval is the casing as well as the surface finish are now however crucial for the effective functioning and long term functioning of the packer.

This all gives the seal elastomer a dual function, that of a dynamic seal as well as a force lock. It is therefore important that the seal elastomer is of sufficient mechanical and chemical quality to do this and this forms an important part of the testing of a packer. To understand this role we need to understand what an elastomer is. An elastomer is a super viscous hydrocarbon fluid. The majority of these elastomers are actually man-made although they do occur naturally being created by plants. These base elastomers are however highly elastic (read stretchable) but have very poor chemical and mechanical characteristics.

In their raw form they have a very limited use. By however adding chemicals and applying heat and pressure to them, they change and gain both chemical and mechanical characteristics. The quality of the vulcanization is very important to the quality of the final product. As with most things in the creation of the elastomer enhancements of properties on one side detracts from them on another. The harder an elastomer is, the less elastic it becomes and the more a force needs to be applied to deform it. The less it deforms the worse it seals, the softer it is the quicker it wears or extrudes under pressure. Finding the correct balance is therefore for seal elastomers very important.
It is therefore vital that in the testing of a mechanical packer that the elastomeric seal is subjected to a considerable range of tests. When an elastomer is compressed and this compression is held for a period of time, after the compression is released the elastomer does not actually return to its original size, this is called “the compression set” and its degree is dependent upon the quality of the seal elastomer. This means that if the elastomer wears on the contact face, due to cyclic loading, that the force in the slips can be reduce due to the reduction in compression and the results of compression set, the combination of this then can lead to movement in the slips and wear here which again leads to more wear on the seals. The extensive testing under cyclic and temperature loading in ISO14310 is intended to test and look into this.

Swelling Seals

Swelling elastomer seals are however different, there are no slips, there is no mechanical force applied to the elastomer. The sealing force is generated by way of a diffusion gradient created by chemicals within the elastomer and this is the same for both oil and water swelling elastomers. The seal is held in place in the well by the chemical bonding of the elastomer to the pipe and by the friction of the elastomer against the casing or formation caused by the induced diffusion gradient. The seals ultimate strength is the tear strength of the partially swollen elastomer.

What this means is that failure modes of swelling seals are different to those of mechanical packers, there are no slips to wear, there is no compression set in the swollen elastomers, how oval is the hole and OCTG plays a different role now, although it may still effects sealing. This has to be therefore reflected in a different form of testing designed at addressing the weaknesses, as did those test of the ISO 14310 for mechanical packer testing.

What are the strengths of swelling packers, this is maybe difficult to define but it is however easier to ask what can be their weaknesses. Because swelling elastomers are the product of chemical engineering it is important that the chemical aspects are understood and addressed, however these do not need to be addressed in a pressure testing environment but are quiet easy to address in a laboratory.

- Chemical bonding tests,
- Tear strength tests of both swollen and un-swollen elastomer
- Running Guide holding tests
- Contact pressure tests.

Whilst each of the above might seem insignificant a number of things have to understood to bring their significance into perspective

Chemical Bonding

This is the most important factor in a swelling seal, it is what hold the elastomer to the pipe. It is crucial for this but has a number of important factors. It is as the name suggests a chemical process, so it is effected by a number of things. The base metal the elastomer is attached to differs
with things like stainless steels and high nickel metals so what works with ordinary steel or iron is not always effective with another base metal. The type of base elastomer is also crucial so Nitrile is not always bonded in the same way as an Aflas or Viton elastomer might be. However the standard of pipe preparation is probably even more crucial, the quality of the grit-blasting and the surface finish and cleanliness are vital. Once blasted the primers has to be applied within an hour so that re-oxidation does not occur. Once the pipe has been coated with a primer the bonding agents may be provided and cured at a later stage. The correct bonding agents for the operating temperature is very important here too, aging of the bonding occurs at higher temperatures so this has to be born in mind. It is often harder to engineer a good and sound bonding than it is to create a sound swelling elastomer and it is as important as the swelling seal itself.

Note: A NUMBER OF THE OLDER COMMERCIALLY AVAILABLE BONDING SYSTEMS CONTAINED LEAD, BECAUSE THIS IS POISONOUS AND ENVIRONMENTALLY DANGEROUS THIS IS REPLACED IN NEWER DEVELOPED BONDING SYSTEMS. HOWEVER IT IS STILL COMMERCIALLY AVAILABLE AND USED IN SOME AREAS BECAUSE IT IS CHEAPER. Bonding is a commercially sensitive process, most suppliers will not supply specific details of their own process so it is important not to push to get this information, but it is important to test for the effectiveness of the process.

Tear Strength tests

Ultimately tear strength determines the effectiveness of the swelling seal, both for the bonding as for the swelling. It gives insight into the quality of the elastomer as well as the effectiveness of the vulcanization. This later is important as a great part of the cost of making a product is in the vulcanization process, incorrectly done and the elastomer has poor mechanical properties, poor swell and even with the best bonding a poor bonded characteristic as the elastomer has to be as strong as the bond otherwise it will easily tear loose. Additionally whilst the diffusion gradient causes the swell it is the vulcanization process that regulates the mechanical properties that give it both in speed and in ultimate swell. With the vulcanization process the correct use of steam or air vulcanization is crucial to ensure that the steam dryness fraction does not affect water swelling elastomer ingredient performance.

Running Guide Holding Tests

After the bonding the only other mechanical part that forms a part of the swelling seal system would be a running guide, some manufacturers use these others don’t. As these are fixed with grub screws it is possible to do a hold test and check when they slip and start to release.

Contact Pressure tests

This is a test some suppliers do to indicate the type of pressure that a swelling seal can exert in an enclosed space, it gives a good indication of the type of holding force a swelling packer can exert. By knowing the unit force per surface area it is possible to calculate the order of size that of the sealing forces that the packer can hold.
The Seal testing

The above tests give a good indication of the quality of the chemical engineering that is a part of the design of the seal and its potential longevity in actual well conditions. If the seal were now to be built into a test facility the above can be firstly cheaply and safely confirmed. The seal supplier having additionally supplied lab data on swell to first contact and then the time required to achieve full anticipated sealing pressure data. This can be confirmed with pressure tests.

Bear in mind that few of the “swelling seal manufacturers” are actual elastomer engineering companies and subsequently buy their elastomers in from compounders and then later have them installed by rubber product manufacturers. These seal manufacturers have knowledge on the final product as most will have conducted test, few will have any in depth knowledge on the elastomer, its compatibility and testing itself.

After conducting the seal test there is little to be gained from extensive long pressure tests as the forces at play, even when loading and temperature cycling is involved, because of the flexibility of the elastomer is taken up by the seal flexing. Additionally the system being different to a mechanical packer has no slip mechanism so that load changes can have no effect on any part of the system other than tearing either the elastomer or the bonding. Having conducted pressure tests to see its holding ability a number of other tests can however be done.

Test to failure and then let the seal heal to see how good is its healing capacity. As most of the seal is still un-swollen in the test set up it is possible for a damaged seal to reseal, something which conventional seals cannot do.
Contact Details

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