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Failure investigation of a hydraulic hose for service in liquid carbon dioxide at high pressure

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by

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Summary

Internal defects were observed during inspection of a flexible hydraulic hose for the transport of liquid carbon dioxide (CO_2) from a tank to an experimental installation at Thornton. The hose was replaced by a new one and the failed hose was sent to Shell Global Solutions, Amsterdam, to perform a failure investigation. Aim of this investigation was to find the root cause of the failure and to confirm the capability of such hoses to operate under supercritical CO_2 conditions, up to 150 °C and at pressures up to 150 bar in the next phase of testing.

The failure investigation revealed that the blister on the internal surface of the hydraulic hose was caused by permeation and accumulation of liquid CO_2 from the internal side through the nitrile rubber liner wall. CO_2 liquid has accumulated at a location of poor adhesion between liner and the reinforcement and has created an internal annulus. When pressure was dropped and temperature increased during un-coupling of the hose the liquid transformed into gas, the annulus expanded and forced the liner to build the blister at the internal surface of the hose.

It is likely that the blister on the cover was created at a weak spot at the interface reinforcement – cover by the same forces.

Multiple layer composites are susceptible to accumulation of gas between liner and parent material when the adhesion between the composite layers is insufficient (non-bonded areas). Nitrile rubber is not suitable for operation at temperature above 100 °C. It is recommended to replace the composite hose by a single layer metal hose.

Amsterdam, December 2010

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1. Introduction

Internal defects were observed during inspection of a flexible hydraulic hose for the transport of liquid carbon dioxide (CO_2) from a CO_2 tank to an experimental installation. The hose was replaced by a new one and the failed hose was sent to Shell Global Solutions, Amsterdam, to perform a failure investigation. Aim of this investigation was to find the root cause of the failure and to confirm the capability of such hoses to operate under supercritical CO_2 conditions, up to 150 °C and at pressures up to 150 bar in the next phase of testing.

2. Objectives

Find the root cause for the failure of the hydraulic hose.

Provide an alternative materials option for dense phase CO_2 service.

3. Scope of work

- Visual inspection of the failed hose and documentation of the failure;
- Dismantling and macroscopically and microscopically examination of hose components (indications for explosive decompression, internal cracks, voids);
- Identification of polymers components and determination of characteristic material properties (by IR, DSC);
- Comparison of the identified polymers with that specified by the manufacturer and confirmation/disclosure of the selected polymer materials to be potentially suitable for the envisaged service (dense phase CO_2, temperature up to 150 °C and pressures up to 150 bar).

4. Results

4.1 Material to be investigated and service conditions

Manufacturer of the hose is Rapisarda (Italy), code: DIN AS PIR 4SPEN856-4SP 51 165 bar WP, EMMI sample number UM.10.005. According to GSUK-PTD/HMHM is the internal liner of the hose made of nitrile rubber and the hose is recommended to be suitable for dense CO_2 service up to 150 bar and a temperature down to -40 °C. The failed hose has not seen temperatures below zero.

The dimensions of the as received hose are listed in Table 1.

Table 1 *Dimensions of the hose as received*

Dimensions	mm
Length (as received)	1430
Length coupling	130
OD	70
ID	50
WT internal rubber liner	3
WT steel wire reinforcement	4
WT external rubber cover	2

Typical service conditions of the hose are listed in Table 2 (selection).

Table 2 *Temperature and pressure data of the CO_2 medium during operation*

Temperature (°C)		Pressure (barg)		Maximum rate of pressure change
Max.	**Min.**	**Max.**	**Min.**	**barg/sec**
16.4	14.5	80.8	62.2	0.2
18.7	10.7	132.1	53.8	2.0
29.3	19.0	144.8	62.3	1.6
27.4	19.2	125.7	60.3	1.4
7.9	7.0	42.4	38.8	0.2
26.5	16.0	148.3	52.1	2.6
24.3	7.7	147.1	40.4	8.8
9.6	3.2	153.5	39.3	3.3
19.4	10.4	42.4	43.1	0.9

4.2 Visual inspection of the failed hose

Defects were observed in the internal liner of the 2 inch flexible hose during endoscopical inspection by GSUK-PTD/HMHM (Figure 1) and on the external surface (blister Figure 2). The hose was cut at the location of the internal defects and send to Amsterdam for failure analysis.

The received hose shows both a blister (Ø 10 mm) on the outer cover as well as a separation of the internal liner at the cut end (Figure 3). The separation occurs between the rubber liner, a fibre cloth and the steel wire reinforcement, over a length of approximately 40 mm.

The internal of the hose was inspected with an endoscope and no additional defects or blisters have been observed.

4.3 Examination after dismantling of the hose

4.3.1 Blister on rubber cover

The blister was clearly visible when de hose arrived in Amsterdam and it was observed that it disappeared during the time between arrival and preparation.

Cross sections were cut through the blister (Figures 4 and 5). Between the internal liner (black part at the bottom) and the external cover (black part at the top) four rows of steel wires imbedded in a red coloured rubber matrix came into view. This rubber matrix is very soft and flexible. There is no tight adhesion between the wires and the red rubber matrix. The separations visible around the wires most likely were applied by deformation of the red rubber during cutting. In the cover a crack is visible. The crack runs along the top row of the steel wires and ends into the rubber.

In the vicinity of the blister (distance about 400 mm) a 7 mm long slit is observed in the middle of the liner (Figure 6) that seems to have an internal connection to another tiny incision (1 mm) on the internal surface of the line (arrow).

4.3.2 Separation between liner and steel wire reinforcement at the hose end

The liner separation is extended over an area of 35 x 35 mm and it is estimated that the total area of separation may be twice as big, including the contra-part of the hose that remained at Thornton. The separation is marked by a convex area on the internal side of the liner (Figure 7) indicating that this area had been blown up.

At the interface liner – steel wires a fibre fabric is applied which usually adheres very tight to the liner (see white spots in cross section Figure 5). The separation occurred just between this fabric and the liner.

4.4 Scanning Electron Microscopy (SEM)

Specimens of the separated part of the internal liner were prepared for SEM to identify defects that typically would occur as a result of Rapid Gas Decompression (RGD).

4.4.1 Morphology of the surface of a specimens prepared with a sharp knife

No micro-cracks were observed in the microstructure that would give an indication for RGD related defects.

The cut surface of this specimen shows a plastically deformed matrix of rubber (fibrils) with spherical particles (Figure 8). These particles most probably are carbon black fillers and the relative large amount of these particles is an indication for a relative high hardness of the rubber.

Shore A hardness of the liner was measured to be 90; the hardness of the cover could not be determined in a reliable way.

4.4.2 Morphology of an artificial brittle fracture (cryogen preparation)

No micro-cracks were observed in the microstructure that would give an indication for RGD related defects. At a high magnification elongated voids are visible in a brittle polymer matrix (Figure 9). The maximum void size is 1 by 5 microns and all voids are aligned in the longitudinal direction.

4.5 Identification of the rubber type

4.5.1 Infrared analysis

Pieces of the liner and the cover were investigated by infrared analysis. Unfortunately the quality of both IR spectra was disturbed by the presence of filler material which made analysis of the rubber components unreliable. The filler material most likely consists of a mixture of calcium carbonate and carbon black. With some reservations, the IR spectra seem to match with the presence of butadiene and acrylnitrile and there are indications that the rubber is a copolymer of butadiene/styrene/acrylnitrile.

4.5.2 Thermal analysis

Differential Scanning Calorimetry (DSC) has been performed to determine the glass-transition temperature (Tg) of both the liner and the cover. The Tg of the liner was measured during a second DSC scan at -38 °C and that of the cover at -45 °C (see Figure 10).

The curves of the Thermo Gravimetric Analysis (TGA) are show in Figure 11. The external cover contains about 11 % (w/w) of oils, plasticizers and other additives, about 34 % polymers and two types of fillers (total about 55 %). The internal liner contains about 18 %(w/w) oils, plasticizers and other additives, 11 % polymer and about 82 % fillers.

5. Discussion

According to the monitored temperature and pressure data appeared the CO_2 medium in the hose in most cases in the liquid state and not in the supercritical phase (see Figure 12).

Both the liner and the cover layer of the hose did not show any indications for chemical degradation. Therefore, chemical degradation of the rubber can be excluded as failure.

Visual inspection of the entire hose surface and examination by electron microscopy of the liner did not show multiple damage features that typically would occur as result of rapid gas decompression damage. The micro voids, which were observed by SEM examination, are strictly aligned in the axial direction of the liner and are most likely created during the extrusion process.

One exception is the tiny slit observed in the liner in the vicinity of the cover blister. This slit could be caused by rapid gas expansion.

RGD resistant grades of rubber in general are hard rubbers; a high hardness can be reached by the addition of large amounts of reinforcing filler particles to prevent the growth of voids and gas bubbles. The large amount of fillers in both the cover and the liner which is confirmed by TGA and observed with IR is an indication that a RGD resistant rubber has been used for the liner. This is verified by the measured high Shore A hardness of the liner.

5.1 Possible failure scenario

Looking at the morphology of the separation failure in the liner it is likely that at this area the liner had been blown down. The reason for the blow down was the permeation of liquid CO_2 through the liner wall and the accumulation of that liquid which created an annulus between liner and reinforcement, most probably initiated at an adhesion defect at that location between liner and fabric. When pressure was released the temperature increased and the CO_2 liquid transformed to gas, resulting in expansion in the annulus and the development of the blister observed during the initial endoscopic inspection.

Gas (or liquid) permeation and accumulation of gas in an already present cavity was most probably also the cause for the blister observed in the cover. The fact that this blister faded away over time indicates that gas permeation and expansion is an important reason for the visibility of such kind of failures. As soon as the gas volume is reduced again by permeation the blister is not visible anymore but the cavity between the cover/reinforcement is still present and may form a weak spot in the cover.

Taking in account that both the liner and the cover blisters diminished after gas pressure release it can be concluded that such failures could potentially be present at more locations of the hose.

As long as the internal blisters could diminish they will not form a problem for the functionality of the hose. However, if the gas pressure difference between annulus and hose internal becomes critical, collapse of the liner may occur with inherent consequences. This situation is undesired and should in all cases be avoided.

As the general high temperature compatibility of nitrile rubber is limited to 100 °C it is recommended to not use the hydraulic hose above this temperature.

It is recommended to replace the composite hose by a single layer metal hose as gas permeation will always occur through plastic components, especially at elevated temperature. Multiple layer composites are susceptible to accumulation of gas between liner and parent material when the adhesion between the composite layers is insufficient (non-bonded areas).

6. Conclusions

- The blister on the internal surface of the hydraulic hose most probably is caused by permeation and accumulation of liquid CO_2 from the internal side through the nitrile rubber liner wall;
- CO_2 liquid has accumulated at the location of poor adhesion between liner and reinforcement and has created an internal annulus;
- During transformation of the liquid into gas and the expansion of the gas volume in the annulus at elevated temperature and the drop of the internal pressure the liner was forced to build the blister at the internal surface of the hose;
- It is likely that the blister on the cover was created at a weak spot at the interface reinforcement – cover by the same forces;
- Multiple layer composites are susceptible to accumulation of gas between liner and parent material when the adhesion between the composite layers is insufficient (non-bonded areas);
- Nitrile rubber is not suitable for operation at temperature above 100 °C.

7. Recommendations

It is recommended to not use the hydraulic hose above a temperature of 100 °C and to replace the composite hose by a single layer metal hose.

Amsterdam, December 2010
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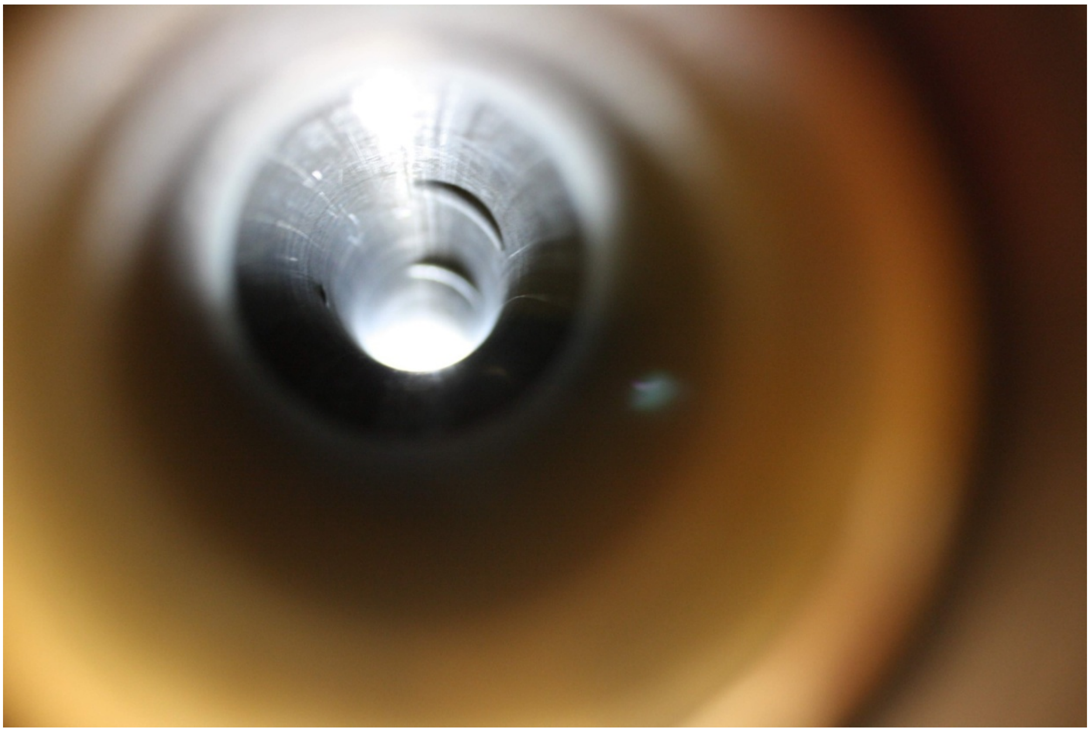


Figure 1 *Defects (blisters) observed at the internal side of the hose*

Figure 2 *Defect observed at the external side of the hose*

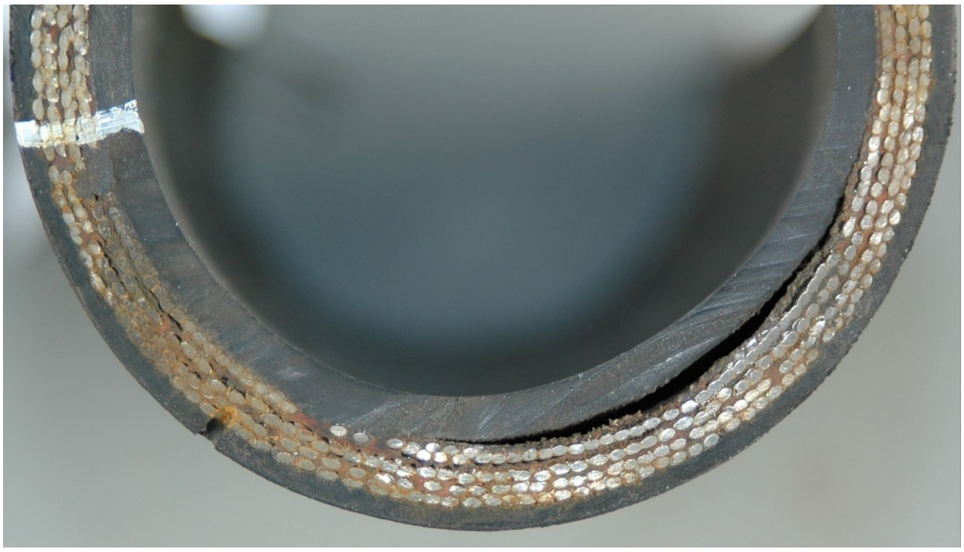


Figure 3 *Separation of the liner, hose cut through the location shown in Figure 1*

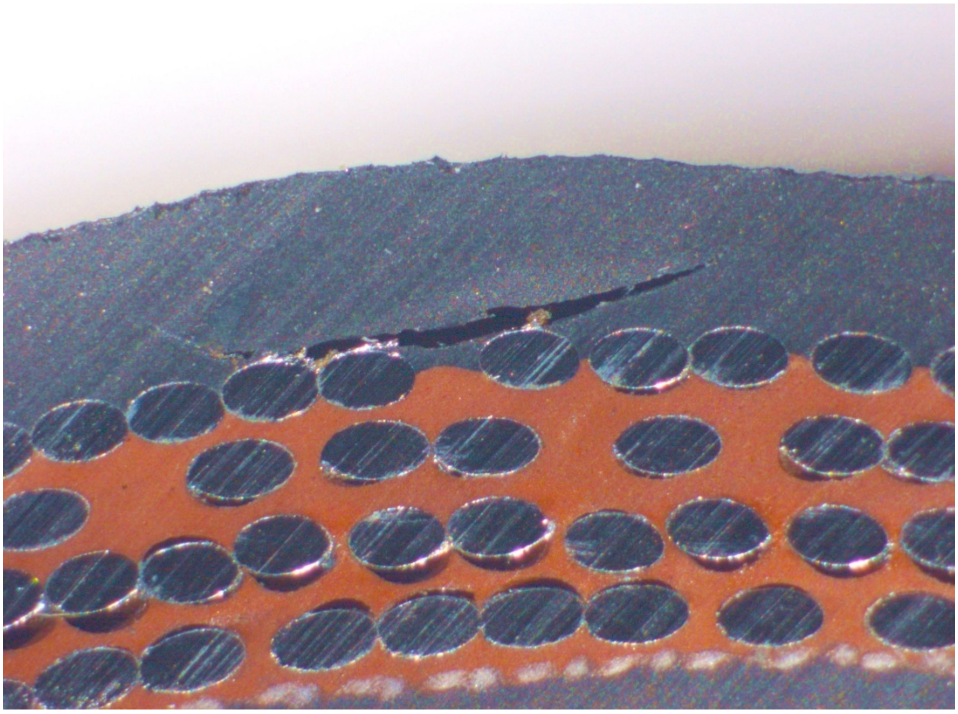


Figure 4
Cross section through the blister in the cover showing the shallow blister and underneath a separation of the rubber from the reinforcement, Part 1

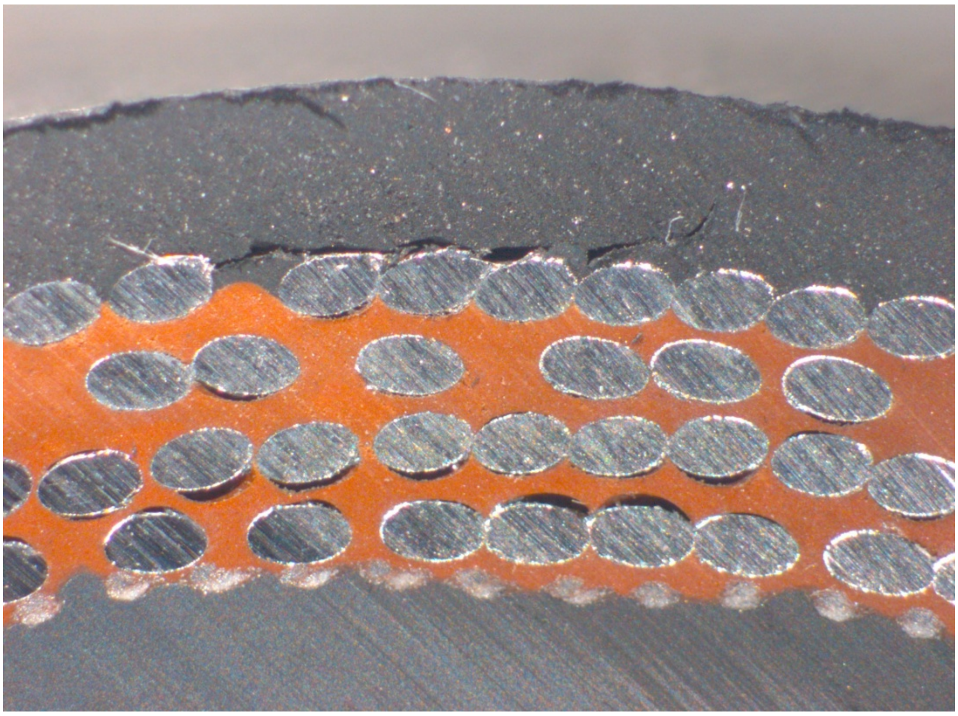


Figure 5 *Cross section through the blister in the cover, Part 2*

Figure 6
Defect in the liner, observed in the vicinity of the blister; the larger slit is obviously connected to a tiny incision on the internal surface of the liner

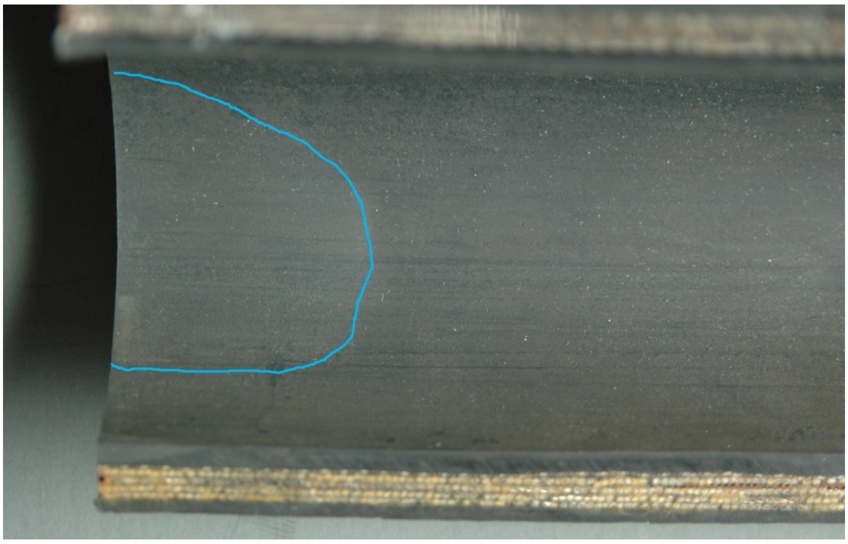


Figure 7
Internal of the liner at location with the separation shown in Figure 3; the convex area marks the extended of the separation

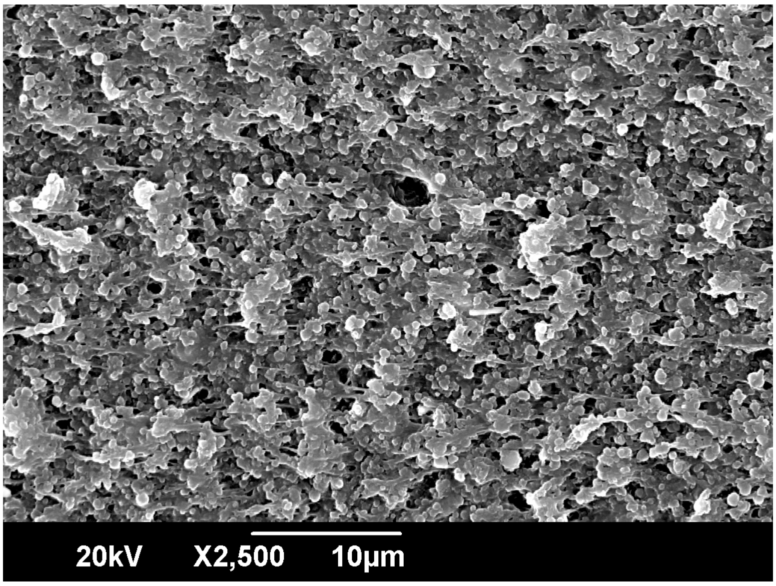


Figure 8
SEM investigation: typical structure of the liner; filler particles in plastic rubber matrix

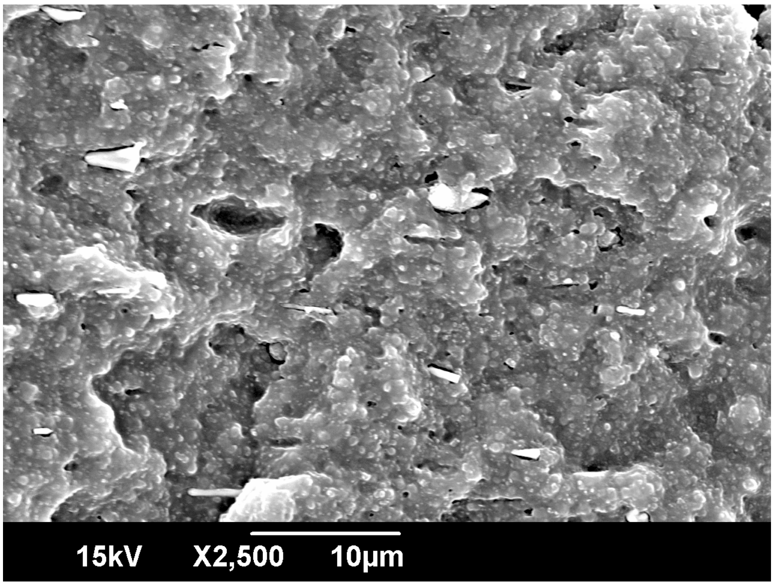


Figure 9
SEM investigation: artificial fracture of the liner showing brittle rubber matrix and voids aligned in the axial direction of the hose

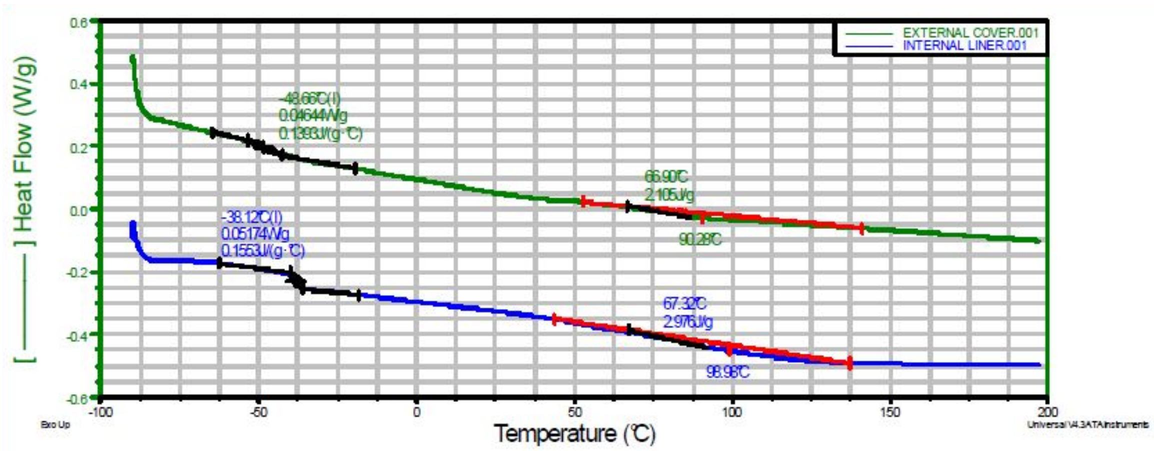
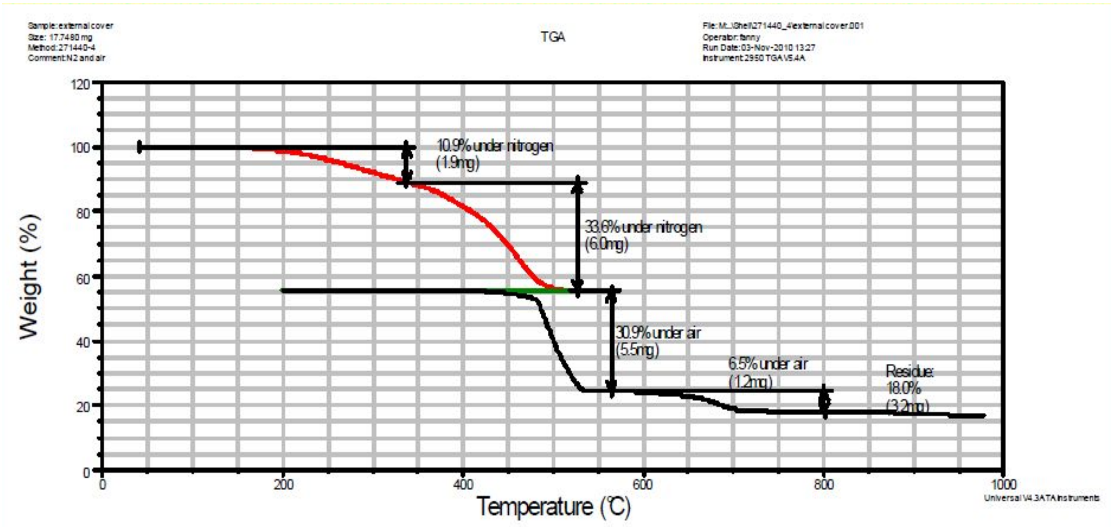
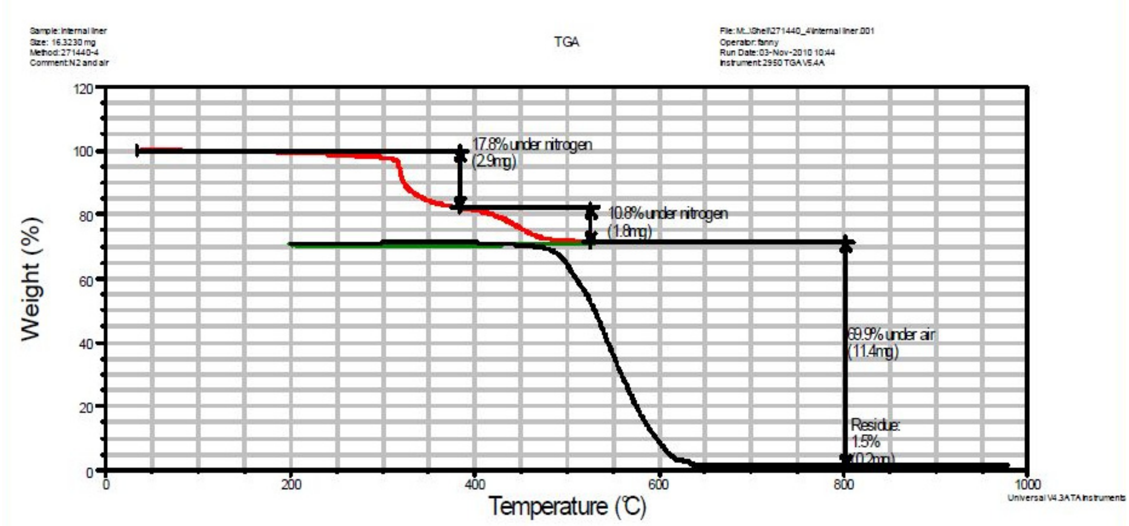


Figure 10
DSC scans from the cover and the liner showing glass-transition at -45 °C and -38 °C respectively



(a) *TG analysis 'external cover'*



(b) *TG analysis 'internal liner'*

Figure 11
TGA curves showing the different compositions or both rubbers; remark the relative large filler content of both rubber

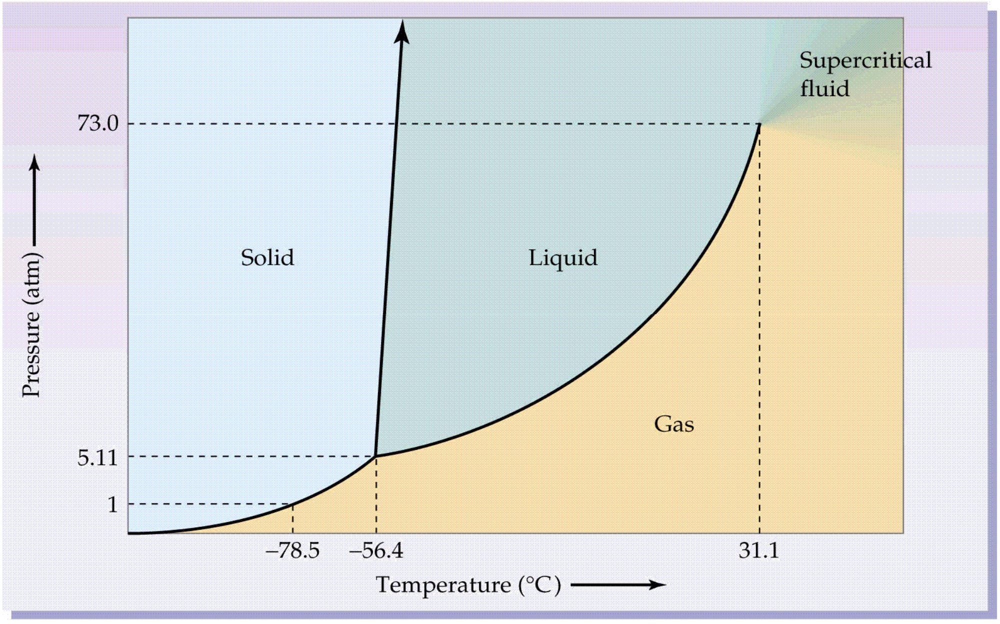


Figure 12 *Temperature/pressure phase diagram of CO_2*

Bibliographic information

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