

Practical research: dry dust filter cartridges after use in hydrogen and natural gas

A comparative analysis of the effect of hydrogen versus natural gas on filter cartridges

Stephanie van Ooijen

Regular inquiries by natural gas suppliers and institutes showed a great need for certainty about the H₂ resistance of filtration products. Theoretical research did not provide the desired assurance, so it was decided to perform a comparative analysis of filters used in practice.

1. Introduction

During the last 50 years, material resistance of steels, sealing materials and plastics in H₂ has been extensively studied, mainly in the USA. The results have since been published in numerous papers. Testing mechanical properties in a hydrogen atmosphere is time consuming since a possible influence will only be shown after a longer period of exposure. Although this topic is becoming increasingly important in the filtration sector, information on hydrogen compatibility of individual materials is hardly ever confirmed by the respective manufacturers or only given with considerable reservations.

The typical hydrogen problem of extremely strong

diffusion through or in all materials is hardly relevant for a filter cartridge. All sealing functions are limited to the retention of particles and not to gas tightness. Hydrogen is not necessarily the biggest challenge for filter cartridges; there are additional factors that can cause complications, such as operating temperature, combinations with reactive substances or gases such as oxygen and pyrophoric dusts.

An investigation was conducted to provide concrete evidence of this. For this purpose, three VoTech filter cartridges were made available for testing. Sample 1 is in new condition and has not been used for filtration purposes. Sample 2 was used for one year in 100 % hydrogen gas (60 °C) with heavy tungsten contamination. Sample 3 was used for one year in dry natural gas. Sample 3 differs in construction due to an additional outer support, end caps and steel quality.

2. Test procedures and results

The requirement towards the DTNW ÖP institute was to comparatively evaluate whether there are visible material deviations between individual components of the used samples compared to the sample in new condition. In addition, the components of the contaminated samples were examined for general damage caused by regular operation. Sample 3 differs slightly from samples 1 and 2, but the filter material is the same.

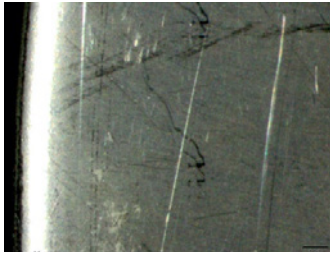
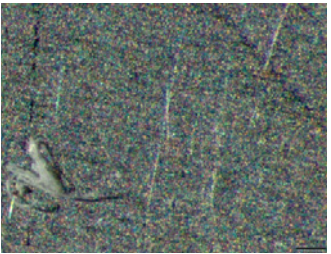
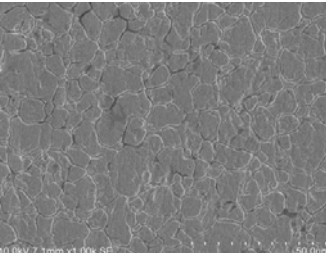
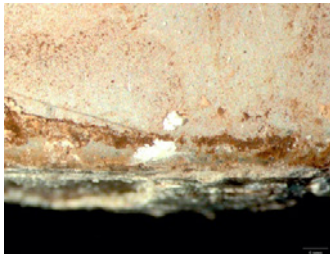
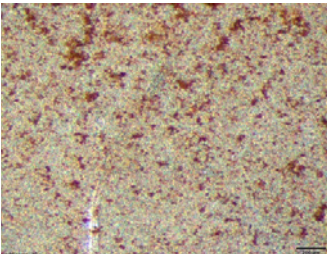
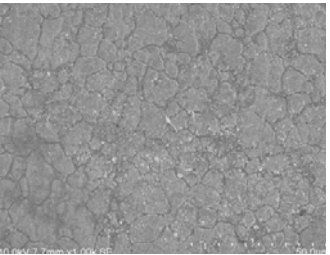

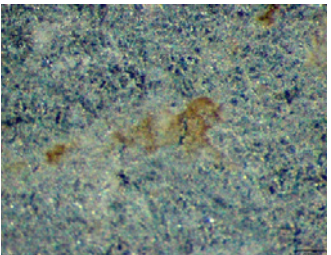
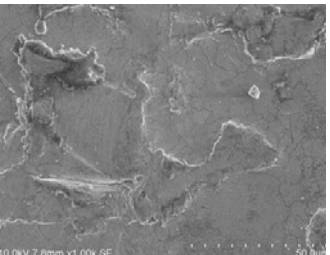
Test methods:

1. Microscopy: observation at different optical magnification.
2. SEM: scanning electron microscope.
3. Tensile properties of textile fabrics (filter material) according to DIN EN ISO 13934-1, Determination of maximum force and elongation at maximum force using the strip method, date of issue: 2013-08.
4. Standard climate: DIN EN ISO 139 (20 ± 2 °C, 65 ± 4 % relative humidity)
Strip width: 50 mm (*cut parallel to the fold)
Condition of test specimens: adjusted



Figure 1: Filter cartridges

Table 1: Summary of photos of the surface of stainless-steel end caps of samples 1 and 2, resp. the metal surface of sendzimir galvanized end caps of sample 3

SMP no.	Microscopy		SEM
1 (metal end cap)			
	6.5x magnification of the surface	40x magnification of the surface	SEM magnification of the surface
2 (metal end cap)			
	6.5x magnification of the surface	40x magnification of the surface	SEM magnification of the surface
3 (metal end cap)			
	6.5x magnification of the surface	40x magnification of the surface	SEM magnification of the surface

Clamping length: 200 mm
Test speed (along filter axis): 100 mm/min
Preload force: 5 N
Number of test specimens: 5
Number of rejected test results: 0

Sample identification:

Sample 1: Filter cartridge, previously without use / new condition.
Sample 2: Filter cartridge, one year use in hydrogen gas.
Sample 3: Filter cartridge, one year use in natural gas.

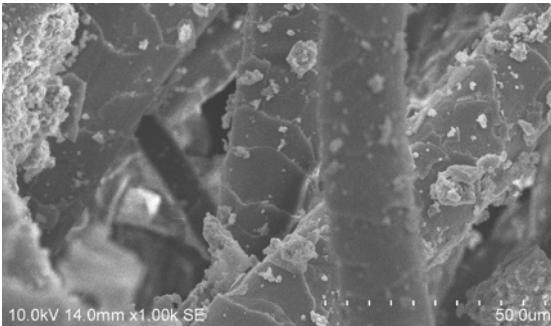
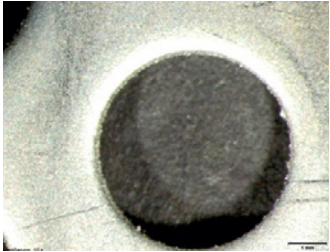
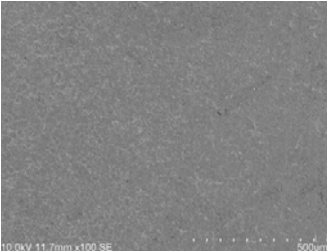
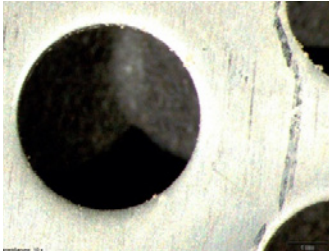
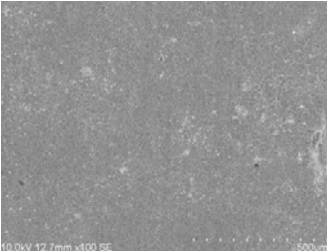
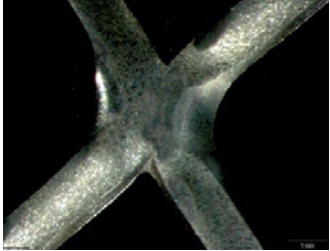
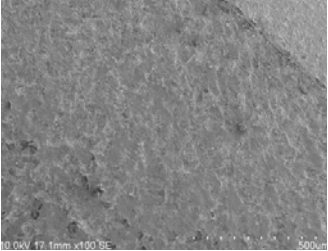


Figure 2: Fibers of the wool felt.

Table 2: Summary of photos of the support cores (oncoming flow side); perforated sheet steel for samples 1 and 2, expanded metal for sample 3

SMP no.	Microscopy	SEM
1 perforated sheet steel stainless steel 304		
	10x magnification of the surface of the core	SEM magnification of the surface of the metal core
2 perforated sheet steel stainless steel 304		
	10x magnification of the surface of the core	SEM magnification of the surface of the metal core
3 expanded metal sendzimir galvanized		
	10x magnification of the surface of the grid	SEM magnification of the surface of the grid

Seals: acrylonitrile butadiene rubber (NBR), hardness 80 shore.

Inner core: austenitic stainless perforated sheet steel 304 (material 1.4301).

Sample 3:

End caps: Metal sheet, sendzimir galvanized.

Seals: Wool felt, animal natural fibre.

Inner and outer core: expanded metal, sendzimir galvanized.

The examination started with the visual impression of the materials of the used filter cartridges (samples 2 and 3) in comparison to the new and unused filter cartridge (sample 1) with regard to parameters such as morphology of the surfaces, damage and changes of the filter materials, seals and the adhesive compound. This was performed by visual evaluation by eye and magnifying glass at various magnifications, as well as by a scanning electron microscope (SEM). The filter material had to be inspected from both sides, as it comes into contact with the respective gas from both sides. Examples of possible changes / damage would be cracks in the filter material, the seals or the adhesive. Chemical changes (e.g., a rough attacked surface) were also examined comparatively.

The components to be tested on the samples were:

Samples 1, 2 & 3:

Flow direction from outside to inside:
Filter material (pleated/folded): 100 % polyester non-woven, upstream side with PTFE membrane.
Bonding compound: two-component polyurethane.

Samples 1 & 2:

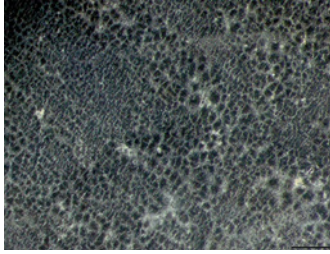
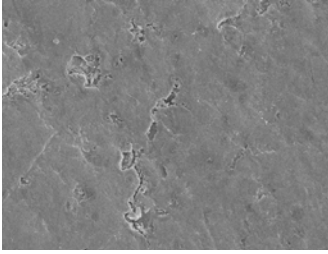
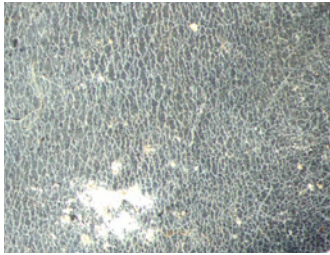
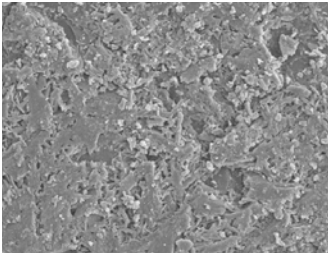
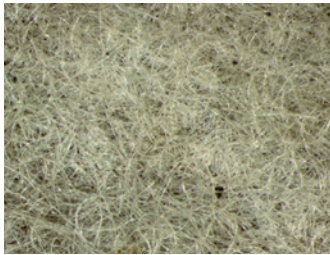
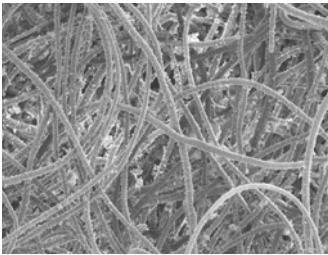
End caps: stainless sheet steel, austenitic stainless steel (material 1.4301).

3. Results of examinations of metal parts

3.1 Support cores and end caps

The selection of materials used in the samples was based on many available studies and publications on the resistance of metal and polymer and hydrogen embrittlement. According to numerous publications, stainless steel 304 offers sufficient protection, stainless steel 316 has no added value (with respect to use in H₂); it is only more expensive. The diffusion of hydrogen into the metals and the resulting hydrogen

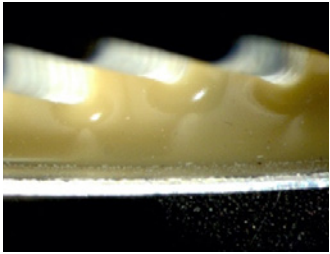
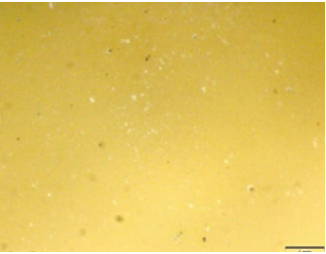
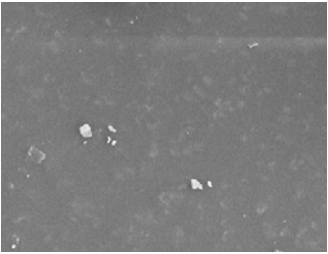

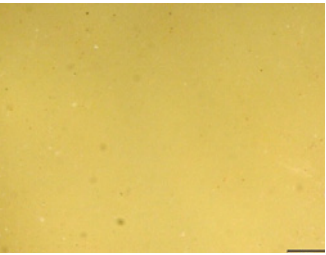
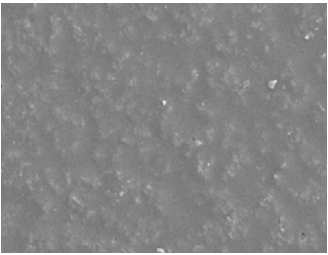
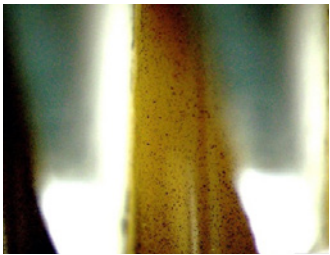
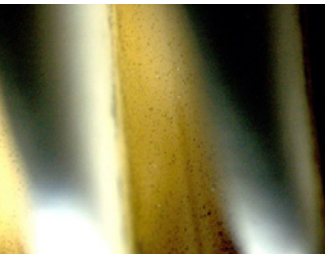
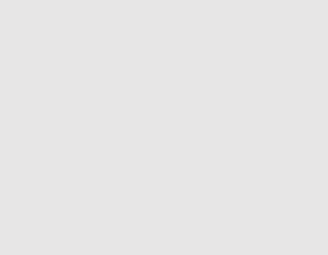
Table 3: Summary of photos of the gaskets applied on samples 1, 2 & 3

SMP no.	Microscopy	SEM
1 gasket nitrile NBR		
	10x magnification of the surface of the gasket	SEM magnification of the gasket
2 gasket nitrile NBR		
	10x magnification of the surface of the gasket	SEM magnification of the gasket
3 gasket natural fiber		
	10x magnification of the surface of the felt gasket	SEM magnification of the felt gasket

embrittlement can cause fractures or even tears under pressure. Table 1 summarizes the images of the metal end caps and the metal surface of the samples. Viewing the covers of the samples in the microscope images shows an overall relatively smooth and undamaged surface for the sample when new (sample 1) with only occasional superficial scratches. In a direct comparison to sample 1, the surface of the metal end cap on sample 2 is very comparable. The surface is visually covered with a grainy / powdery and slightly brownish deposit i.e., tungsten carbide particles carried by the gas flow. The surface underneath is undamaged and at most exhib-

its the superficial scratches already detected on sample 1; no structural damage is observed. The surface of sample 3 is covered with fewer deposits than sample 2 and overall is somewhat rougher/matt than that of samples 1 and 2. Apart from slight superficial scratches, no damage is visible here either in the areas examined. SEM images of the metal sides of the metal end caps from the samples are shown at identical magnification. Sample 1 shows a scale-like structure that is only visible at this magnification. Sample 2 shows a comparable structure with additional particles on the surface. These particles were most likely deposited there during operation in hydrogen gas. Structural damage such as scratches, ruptures and holes were not found on the examined surfaces of samples 1 and 2. Due to the difference in material, sample 3 cannot be directly compared with the sample in new condition. The surface here seems smoother and covered with some particles, but also on sample 3 there is no structural damage to be discovered after operation in natural gas. Table 2 summarizes the images of the perforated stainless-steel core of samples 1 and 2 and the galvanized expanded metal core of sample 3 on the upstream side. The inner core of sample 1 shows a smooth and clean surface without damage. The surface is slightly shiny and the edges of the perforations are clean. Only occasionally superficial scratches occur. Neither damages nor material changes of the surface are detected on sample 2, the edges of the perforations are unchanged. Overall, no change can be observed. The composition of parts in sample 3 differs from the configuration of the other 2 samples. The outer core of galvanized expanded metal has been examined but could therefore not be compared to the stainless

Table 4: Summary of photos of the bonding compound applied in the end caps of samples 1, 2 & 3

SMP no.	Microscopy		SEM
1 adhesive both ends			
	6.5x magnification of the bonding compound between metal and filtering material	10x magnification of the extensively applied adhesive mass in the end cap	SEM magnification of the bonding compound
2 adhesive both ends			
	6.5x magnification of the bonding compound between metal and filtering material	10x magnification of the extensively applied adhesive mass in the end cap	SEM magnification of the bonding compound
3 adhesive both ends			
	10x magnification of the adhesive mass at the edge of the filter between metal and filter material	10x magnification of the adhesive mass at the edge of the filter between metal and filter material	

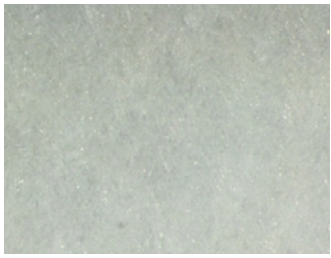
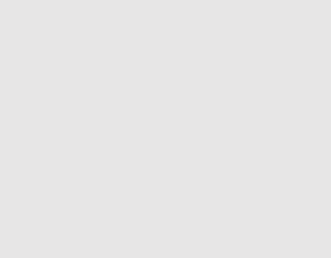
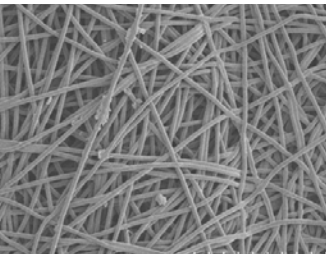
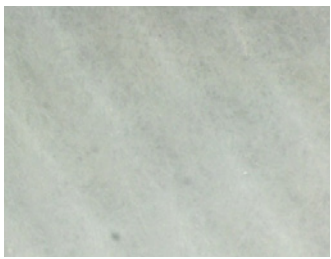

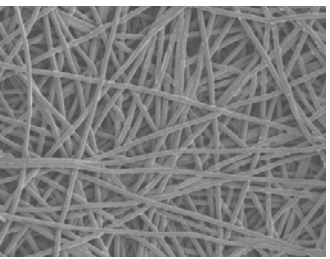


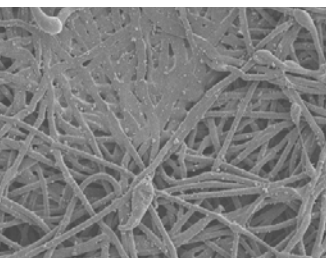
perforated core of sample 1 or 2. The surface of sample 3 also looks undamaged and the edges of the metal strands are clean. Structural damage is not detected. The SEM images of the perforated plates show a similar scale-like surface on samples 1 and 2 as was previously visible on the metal cover of the filter cartridge. No visible material damage to the examined areas can be observed here either. Here, particles are not visible on these surfaces of the perforated cores, since it was mounted at the clean side of the cartridge. As mentioned before when referring to table 1, the composition

of parts of sample 3 again differs from the other samples and also with this sample, there is no visible damage that may have resulted from use in the natural gas flow. Individual particles are visible on the surface here because the core was installed on the outside of the filter and thus exposed to the particle-loaded gas stream.

4. Results of tests on synthetic parts

Phthalates are used primarily as plasticizers for polymers, and the saturated hydrocarbon compounds in hydrogen or natural gas cause the polymer to become

Table 5: Summary of photos of the inner side (downstream side) of the applied filtering material in all three samples

SMP no.	Microscopy		SEM
1 Filtering material downstream side			
	10x magnification of the inner side of the filter medium (area)		SEM magnification of the fibers at the inner side of the filtering material
2 Filtering material downstream side			
	10x magnification of the inner side of the filter medium (area)	10x magnification of the inner side of the filter media (pleat)	SEM magnification of the fibers at the inner side of the filtering material
3 Filtering material downstream side			
	10x magnification of the inner side of the filter medium (area)	10x magnification of the inner side of the filter media (pleat)	SEM magnification of the fibers at the inner side of the filtering material

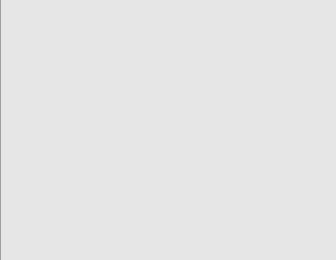
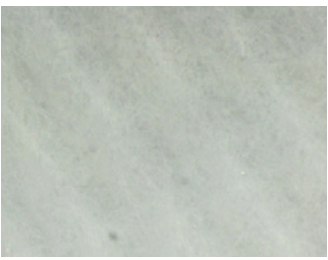
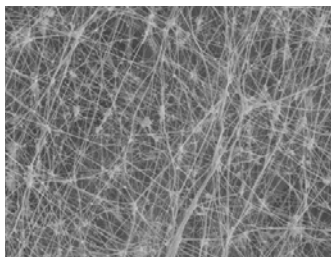

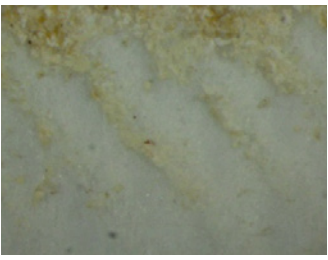
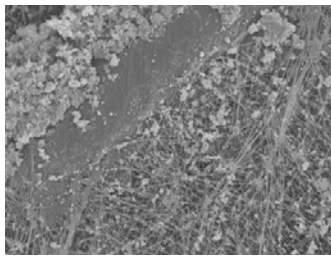
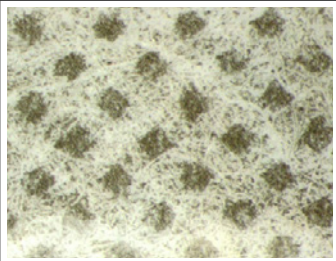
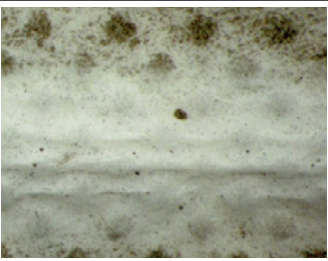
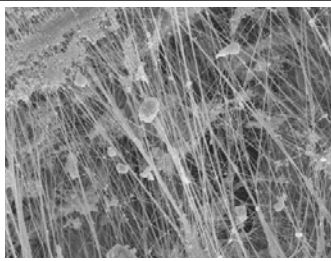
brittle. The possible subsequent fibre breakage can cause the filter material to be damaged and the separation efficiency to be impaired. Therefore, this was the most important part of the study, especially when applied in hydrogen gas.

4.1 Gaskets

Table 3 summarizes the images of the gaskets of the different samples. The acrylonitrile butadiene rubber (NBR) gasket for test sample 1 is optically undamaged at 10x magnification except for a very superficial crack in the material. However, the cracking does not go into depth and makes the surface appear somewhat textured. The

surface is also very clean. For sample 2, no significant differences can be seen in a direct comparison. Individual particles are visible on the surface as an overlay, but they were probably deposited there during operation or transport. Otherwise, also this surface is undamaged except for the very superficial fractures, which again are not deep and thus only makes the surface appear textured. The previously described different design of sample 3 also encompasses different gasket material. Instead of a gasket made of an elastomer, a gasket made of wool felt is applied. This gasket and the fibres it contains are also visually undamaged and no destroyed or protruding fibres can be seen. The white colour of

Table 6: Summary of photos of the outside (upstream side) of the applied filtering material in the three samples

SMP no.	Microscopy		SEM
1 Filtering material upstream side			
		10x magnification of the outer side of the filter media (area)	SEM magnification of the fibers at the outer side of the filtering material
2 Filtering material upstream side			
	10x magnification of the outer side of the filtering material with filter cake (area)	10x magnification of the outer side of the filtering material with filter cake removed (area)	SEM magnification of the fibers at the outer side of the filtering material
3 Filtering material upstream side			
	10x magnification of the outer side of the filtering material with filter cake (area)	10x magnification of the outer side of the filtering material with filter cake removed (area)	SEM magnification of the fibers at the outer side of the filtering material

the fibres is still visible inside the gasket material. However, the fibres at the surface of the gasket are slightly darker in colour, caused by deposition of dust particles on these fibres during operation in the gas flow. The SEM images of the NBR seals of samples 1 and 2 show some superficial fractures, but these do not extend into the depth of the material. Additionally, for Sample 2, there are many particles visible on the surface that were likely deposited there from use in the hydrogen gas and/or transport. Structural damage for sample 2, which could result from use compared to

sample 1, is not observed at the examined areas. A higher magnification of the wool felt (Fig. 5) reveals the scaly structure of the individual fibres together with individual particles. Wool is typically characterized by a scaly structure on the surface and can thus be distinguished from for example polyester fibres which have a smooth and homogeneous surface. The particles can be traced back to the use of the filter in the gas flow. There are no damaged fibres or fibre ends observed at the examined areas. It can be concluded that operation in the natural gas flow has caused no damage.

Table 7: Test results maximum tensile strength on applied filter media in samples 1, 2 & 3

SMP SMPno.	material direction	max. tensile strength [N]	variation coefficient [%]	95% confidence range [N]
1	Lengthwise	643	5,6	599 - 688
2	Lengthwise	743	4,0	706 - 780
3	Lengthwise	1020	3,3	980 - 1060

4.2 Bonding material

Table 4 summarizes the images of the examined areas of the bonding compound between the filter material and end caps of sample 1 to 3 and of the extensively applied adhesive mass in the closed end cap of samples 1 and 2. The microscopy images show that the adhesive mass for sample 1 is undamaged and uniform both at the outer edge of the filter between the metal and the filter material and on the area applied to the inside of the closed end cap over a large area. On the adhesive surface, no blisters and only few overlying particles are detected. At the edge, the conclusion by means of adhesive between the metal end cap and the filter material, is very even and without voids. When comparing samples 1 and 2, no differences could be observed in sample 2 that would make the state of the adhesive compound appear poor. Superficial particle build-up can be seen on the outer edge, most likely deposited there during operation in the hydrogen gas flow. On the inside of the filter, no particles can be seen on the large-area application in the end cap, the compound looks still even and uniform as if it was not used, conform sample 1. Only the part at the edge of both rings of sample 3 can be assessed, as this sample does not have a closed end cap and therefore no larger surface area of adhesive can be examined. Like with sample 1, in this area at the edge of the ring, the adhesive was also uniformly applied without any voids and showing only a few overlying particles.

On the SEM images of the adhesive mass in the end caps of samples 1 and 2, no differences can be observed between the adhesive mass for samples 1 and 2 in the areas examined. The adhesive mass is applied over a large area and no voids, fractures or other damage can be observed on sample 2 compared to sample 1. This indicates that the use in hydrogen gas does not cause any damage to the bonding material. The occasional particles that were visible on the surface of the inspected sample areas, were probably deposited there during sample preparation.

4.3 Filter material

The filter media, a high-quality polyester nonwoven

with PTFE membrane, is pleated to increase surface area. Despite the expectation that the heat treatment and mechanical action of pleating will alter the material, much attention is paid to this in the production process. This is verified with regular measurements, for which the material is tested for separation efficiency and for differential pressure. This is performed by means of standardized test dust which is injected into a closed test system. The efficiency is tested by means of an optical particle counter. For this current investigation, the tensile strength was tested again after the material was pleated and (sample 2 and 3) has been in operation in the gas stream. Tungsten dust is very abrasive, which, in addition to its use in pyrophoric hydrogen gas, presents an additional challenge.

Table 5 summarizes the images of the inside (downstream side) of the filter material. The microscope images show no significant differences between the samples when comparing the inside of the filter materials of samples 1 to 3. Sample 1 in new condition is cleanest on the inside, but samples 2 and 3 also show only minimal contamination that may have gotten there during transport. At the examined areas of samples 1 to 3, all surfaces are undamaged; no tears, holes or other defects can be observed. The fibres also show no differences in terms of structure or generally protruding fibre ends.

The SEM images of the inside of the filter material are summarized in the 3rd column. For all three samples, a network structure of fibres with a thickness of about 20 µm can be seen on the inside and only few to no particles are visible on the inside. This confirms the effectiveness of the filter material and no significant number of particles could get onto the clean inside of the filter. Individual visible particles could also have gotten there in the course of transport. Damage such as cracks or holes cannot be observed in any of the samples examined, so use in the gas stream did not cause any obvious damage.

Table 6 summarizes the images of the upstream side of the filter material. Microscopy images in columns 1 and 2: Comparison of the outside of the filter materials of samples 1 - 3 shows no significant differ-

Table 8: Maximum tensile strength - elongation of applied filtering material

SMP no.	material direction	max. tensile strength elongation [%]	variation coefficient [%]	95% confidence range [%]
1	Lengthwise	33,0	7,0	30,2 – 35,9
2	Lengthwise	34,5	4,4	32,6 – 36,4
3	Lengthwise	24,7	3,4	23,7 – 25,8

ences between the samples, with the exception of the filter cake formed for samples 2 and 3. Sample 1 is in new condition and accordingly unpolluted. Sample 2 shows a significant white / beige filter cake (tungsten). After careful removal of the filter cake, a white surface of the filter material is revealed, which is comparable to sample 1. No ruptures, holes or other imperfections can be observed on the material in the examined area and in the examined pleats (folds). Sample 3 has a darker overlay than sample 2 (very fine natural gas dust), but after careful removal of the overlay, the white base material is also very clearly visible here and it shows a comparable picture to sample 1. No tears, holes or other defects can be observed here either in the surface or at the crease in the pleat.

Sample 3 is from a different batch of material, which means that the base material has a different surface structure than samples 1 and 2, but both materials are 100% polyester with the same membrane. The only difference is that sample 3 shows a diamond pattern, while samples 1 and 2 have horizontal stripes. This is even more evident because of the fine, dark natural gas dust deposited in the matrix of the material.

SEM images on the outside of the filter material show a network structure of fibres for all samples, which is significantly denser than for the inside of the filter material. In addition, the fibres are significantly finer than on the inside and have a thickness well below 1 micron. With samples 2 and 3, the filtration effect is visible due to the deposit of many particles on the surface of the filter material during operation in respectively the hydrogen and natural gas flow. For sample 1, no particles are visible on the surface, which was to be expected due to the new condition. There were no tears, holes or other defects observed at the examined areas, so operation in the gas flow has caused no damage.

5. Tensile properties of textile fabrics

Since the influence of hydrogen and natural gas on the elasticity of polymer is considerable, it was important to have the maximum tensile strength of the filter material examined as part of the investigation of material durability. The test was carried out according to DIN

EN ISO 13934-1, determination of maximum tensile strength and maximum tensile elongation using the strip tensile test, issue date: 2013-08.

The maximum tensile strength of the filter material was determined in one test direction only, in order to have the material without the folds in the filter material transverse to the test direction. This was decided because these could possibly be noticed as a weak point in the tear tests and falsify the result. Thus, the samples were cut to a width of 5 cm and the creases in the material were kept parallel to the tensile direction. The maximum tensile strength of the filter material in its new state (sample 1) is about 650 N at an elongation of about 30 %. For the filter material used in hydrogen (sample 2), a maximum tensile strength of about 750 N at an elongation of also slightly more than 30 % has been measured. The value of the maximum tensile strength is thus even somewhat higher than in the new condition and the ductility is almost unchanged, so that it can be stated that the use of the filter material in hydrogen gas obviously has no negative influence on the material in this respect. In the data sheet for the filter material, values of 700 - 900 N or an extensibility of about 17 % are given. The measured 650 - 750 N thus fit perfectly with the value in the supplier's data sheet for the transverse direction (tensile strength CD) of 700 N. Since no tolerance range is given, practical experience is used at this point. Here, a scatter of about 10 % around the target value is considered very acceptable, especially since the value for the material used in hydrogen gas is even higher than for the virgin material. The ductility in the measurements shown here is higher than in the supplier's data sheet, but this should not be seen as negative. Since the extensibility in the new and used condition is comparable, no negative effect of use in hydrogen gas can be detected here either. These results confirm the impression of the microscopic examination as well as the SEM measurements, which did not reveal any damage to the material.

For sample 3, an even higher tensile strength of 1000 N was determined than for samples 1 and 2, while the ductility was somewhat lower. This also

speaks for an intact filter material after the application set in this case in natural gas. In addition, it confirms the results of the microscopic examination and SEM measurements, which did not reveal any damage to the material. The higher value compared to samples 1 and 2 could be due to a different material batch, as the basic structure of the material also differs slightly from the material in samples 1 and 2, as described earlier in the microscopic examination and SEM images.

Summary

The visual evaluation of the samples under the magnifying glass/microscope showed no overall damage to the individual components tested for the three samples. Overall, the filter materials are undamaged on the inlet and outlet side, indicating that the use of the filters in natural gas or hydrogen under used conditions, does not cause any damage. The filtering effect is maintained, since no difference was observed between sample 1 (new condition) as a reference and samples 2 (hydrogen) and 3 (natural gas). Discoloration and overlying filter cake are present due to use and cannot always be completely removed. Also, for the other components of samples 2 and 3, consisting of perforated sheet steel, metal parts of the end caps, bonding compound in, and at edge of the end caps, as well as at the gaskets, no damage could be detected when comparing these to sample 1.

In summary, the SEM images show very good and detailed images of the different components of the samples. No damage was observed on any of the components of samples 2 and 3, allowing them to be used in hydrogen and natural gas, respectively, under the previous conditions without damage.

The determination of the maximum tensile force

showed no significant differences between the sample in new condition (sample 1) and the sample already used in practice (sample 2). The values are readily comparable and range around the reference value of 700 N from the filter material supplier's works certificate. The material in new condition is somewhat lower at around 650 N, but a variation of 10% around the reference value is not unusual in practice. The filter material is thus resistant to hydrogen gas and natural gas. Even higher values were determined for sample 3, so that it can also be assumed here that the material was not damaged by use in the gas stream, as indicated by the microscopic examinations and the SEM images.

In summary, the tested filters were not negatively affected by use in hydrogen or natural gas with respect to the tested parameters; the use of the tested materials does neither lead to damage or reduced durability as was determined optically under the microscope and in the SEM, nor to the tear strength of the filtering material. The cartridges can therefore be used in hydrogen gas without restriction with regard to the tests / assessments presented under the previous conditions for the respective times.

Author

Stephanie van Ooijen
VoTech Filter GmbH
Heinsberg
Phone +49 2452 95900
info@votech.de

Acknowledgements:

We would like to thank the independent DTNW Öffentliche Prüfstelle GmbH in Krefeld for their factual and technical support.