Materials aspects associated with the addition of up to 20 mol% hydrogen into an existing natural gas distribution network

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ABSTRACT

The introduction of hydrogen into the UK natural gas main has been reviewed in terms of how materials within the gas distribution network may be affected by contact with up to 80% Natural Gas : 20 mol% hydrogen blend at up to 2 barg. A range of metallic, polymeric and elastomeric materials in the gas distribution network (GDN) were assessed via a combination of literature review and targeted practical test programmes.

The work considered:

- The effect of hydrogen on metallic materials identified in the network
- The effect of hydrogen on polymeric materials identified in the network
- The effect of hydrogen exposure on polyethylene pipeline techniques (squeeze off and collar electrofusion)

The trials concluded that the majority of metallic materials showed no significant deterioration in mechanical (tensile) properties when stored in hydrogen environments compared to those stored in analogous methane or blended gas atmospheres up to 2barg. Polymeric materials showed no deterioration to efficiency of squeeze-off nor collar electrofusion in socket or shoulder orientations following soaking in hydrogen, methane or hydrogen blends.

Introduction

Concerns relating to the production of carbon dioxide (CO_2) and its effects on global background temperatures have led to international efforts to reduce CO_2 emissions. One of the main contributors to CO_2 emissions is the burning of fossil fuels in domestic and commercial fuel supplies, especially that of burning natural gas (NG) comprised primarily of methane (CH_4). A consortium of xxx, yyy and zzz has undertaken a feasibility study of supplementing NG supplies with hydrogen (H_2) up to a maximum injection level of 20% mol/mol Hydrogen (hereafter referenced as 20%H_2).

Asset Survey

To identify target materials representative of the UK gas network, a preliminary asset survey was carried out on an isolated, small scale (~ 100 properties) gas distribution network (GDN).

The asset survey identified that there were five distinct applications of materials on the GDN, these being :

- Mains pipeline and risers
- Valves
- Governors
- Meter connections to appliances, and
- Pipeline repair materials

Table 1 details the materials identified in these applications and their locations on the test site GDN.

Table 1: Materials identified on test site GDN from asset survey

Material	Area of use
Medium Density Polyethylene (MDPE) PE80	Pipelines
Low strength steel (eg API5L grade B)	Pipeline, risers
Cast Iron	Valves, governor valves, appliances
Copper	In-building pipelines
Yellow Brass	Pipe joints
Aluminium	Valves, appliances
Tin / Copper Based Solders	Pipe joints
Lead Based Solders	Pipe joints
Elastomers / Polymers	O-ring seals, diaphragms
Rubber Hoses	In-home connections
Nylon	Meters
Epoxy Cure Based Polymers	Meters, regulators, pipeline repairs
Stainless Steel	Hydrogen injection and mixing unit

This group was further expanded to better identify the elastomeric materials present as shown in Table 2.

Table 2 : Polymeric elastomer materials used at the test site GDN

Elastomer / Polymer name	Components
Nitrile (NBR) or Buna-N	Seats and seals in gate valves, non-return valves, slam shut valves and safety check valves.
Fluorocarbon rubber (Viton)	Seals, O-rings
Compressed asbestos fibre (CAF) gaskets	Gaskets (gate valves)
Ceramic loaded Polytetrafluoroethylene (PTFE)	Spindle bearings (safety check valves)
Nylon 581	Safety check valves
Polyurethane	Sealant (encapsulant repairs)
Polyamide	Turbine meters
Polyacetyl	Turbine impellers (turbine meters)
Polycarbonate	Glass panels (turbine meters)

Finally, the network operating conditions were determined as shown in Table 3.

Table 3: Test site GDN operating conditions

Operating condition	Values and units	Comments
Maximum operating pressure	1.5 barg (2.5 bara)	Upstream of the governor
Minimum operating pressure	20 mbarg (1.02 bara)	Connections to domestic properties

Those material types identified in the asset survey were further investigated via literature review and, where knowledge gaps were identified, practical experimental assessment.

THEORETICAL ASSESSMENT: LITERATURE REVIEW

Metallic Materials

A preliminary literature review identified that the majority of prior data related to the interaction of hydrogen with the target metallic materials was conducted under environments designed to promote the potential gas / solid interaction mechanisms (e.g. high hydrogen partial pressures or cathodic charging (1) to encourage hydrogen uptake) and did not directly reflect the GDN conditions detailed in Table 3. The general aim of previous research was to encourage hydrogen embrittlement in order that the phenomenon could be investigated as opposed to providing data on a specific GDN design. Selection of the extreme test conditions were taken to suggest that the thermodynamics of hydrogen absorption into metallic substrates was generally unfavourable.

Polymeric Materials

For polymeric and elastomeric materials, data generally suggested that there was no equivalent to hydrogen embrittlement within polymeric materials but gas permeation rates were generally higher than for metallic materials.

Summary

It was observed that there was also very little data related to the possible effects of blended gases on materials (e.g. preferential uptake of hydrogen from the blend or diminished embrittlement relative to pure hydrogen environments). The review indicated that a practical test program would be required to reflect the specific conditions of the chosen GDN. This would include soaking of materials at network pressures in a range of test gases and subsequent analysis. The literature review is not detailed here but the reader is directed to references 1 to 18 for further reading as required.

PRACTICAL PROGRAMME OUTLINE

To address the knowledge gaps identified in relation to the interaction of target materials with low pressure blended Natural Gas (NG) : Hydrogen (H_2) gases, a practical test programme was devised as shown in Table 4. Due to the inherent variability of the chemical composition of NG, a 100% methane (CH_4) test gas was used as a test analogue for NG to provide a sound basis for data comparison. Test sample types were selected based on the output of the asset review, the preliminary output from the literature review and the material propensity for being on the GDN. The blended gas chosen for assessment was UK standard G222 composed of 23%Vol H_2 in CH_4 as this approximated to the trial range of 20%Vol H_2 whilst also being an easily obtainable, recognized standard UK composition. Similarly, storage conditions were set to 2Barg as this value provided a safety factor relative to the maximum 1.5Barg found on the test GDN.

Figure 1 shows the test samples within a gas soaking facility prior to sealing. Polycarbonate racking as shown was placed within the soaking facility to allow gas contact on all sides of the metallic test samples. Due to the perceived high permeability of gas into polymeric materials, racking was not thought necessary for the pipe samples.

(1) Cathodic charging subjects a sample to a potential across its thickness whilst within a hydrogen environment to encourage ingress of hydrogen into the bulk material.

Figure 1 : Samples Within the Gas Soaking Facility Prior to Sealing

Table 4 gives details of the experimental test programme.

Table 4 : Practical Test Programme

Study Parameter	Material for Assessment	Storage Conditions	Post Storage Analysis
Effect of 100% H_2, G222 NG : H_2 blend, or CH_4 on metallic sample post-storage mechanical properties	304 austenitic stainless steel	6 week storage at 2 barg, 20 Deg.C	Tensile testing to BS EN ISO 6892-1: 2016 method A2 at the slowest acceptable crosshead speed of 0.01 mms^{-1} (equating to a corresponding strain rate of 0.000253 s^{-1}). Determination of • Total elongation at failure • Ultimate tensile strength • Proof strength *(See Notes 1 and 2 below)*
	API 5L Grade B steel		
	Copper		
	Yellow Brass		
	Copper specimens soldered with lead free solder		
	Copper specimens soldered with leaded solder		
	Cast iron		
Effect of 100% H_2, G222 NG : H_2 blend or CH_4 on polyethylene pipe PE80 handling and jointing properties	63 mm diameter Polyethylene PE80 Pipe	6 week storage at 2 barg, 20 Deg.C	Conditioning at 0 Deg.C followed UK gas standards including post squeeze-off hydrostatic assessment (water in water) at 80 ^{o}C for 165 hours with an applied pressure of 9.7 bar (actual pressure) (equating to 4.5 MPa hoop stress). Post conditioning assessment by dye penetrant inspection (DPI) / X-ray radiography.
			Electrofusion collar joint trials in both saddle (90 Deg) and socket (end-to-end) orientations.

Note 1 : Solder samples were produced from two "half" tensile test pieces with a 5mm overlap of solder joint and tested as closely as possible to the conditions of the BS EN ISO 6892-1:2016 procedure.

Note 2 : Literature review indicated that lower crosshead speeds were best suited to studies on hydrogen embrittlement of metals.

A pipe squeeze-off trial detailed above was undertaken to assess whether hydrogen ingress into Polyethylene (PE) would have a negative effect on the compression and reforming of PE pipe during squeeze-off operations. Pipe squeeze-off is used in the gas industry as an emergency gas control procedure where the two sides of a PE pipe are compressed until the sides of the pipe meet, stopping any gas flow past the area. As part of standard gas industry procedures, repetitive squeeze off of any location is a pipeline is not undertaken and a distance of ~ 1m is maintained between any subsequent squeeze-off locations so as to not overly weaken the pipeline.

To mirror this procedure, a once only squeeze-off of hydrogen soaked, 63mm diameter test pipe specimens was undertaken. Industry experience indicated that squeeze off would be more likely to damage pipes at low temperature and where thicker wall pipes are closed : to represent this "worst case" scenario, the pipe was chilled in an ice bath for 45 minutes prior to testing.

The electrofusion pipe collar jointing trial was conducted to inform whether hydrogen absorbed within the polymer matrix would be liberated during the high temperature jointing process, potentially leading to blistering or other failure of the joint. Electrofusion collar jointing of the PE80 pipework was selected as the jointing process for assessment over the alternative technique of butt fusion jointing as the use of butt fusion jointing is limited to jointing of new, over-ground pipes and therefore would not have been exposed to contaminant gases prior to joint formation.

TEST RESULTS

Tensile Test Results

On completion of the 6 week soaking period, samples were removed from the facility and immediately transfered for tensile testing (so as to minimise the potential for hydrogen outgassing). Typically, up to seven specimens were tested for each parent material/soaking condition combination. Data and commentary are shown for each test sample below, with the proof strength values quoted being the 0.2% values.

304 Stainless steel

Tensile test data for Stainless Steel 304 following the soaking period in the various test gases are shown in Table 5.

Table 5: Tensile test data for SS304

Material (as- manufactured)	Proof strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation at Break (mm)
S Steel 304 Control	361 SD 4	639 SD 30	28 SD 6
S Steel 304 in H_2	370 SD 3	635 SD 30	23 SD 6
S Steel 304 in CH_4	370 SD 20	627 SD 26	20 SD 5
S Steel 304 in G222	364 SD 8	641 SD 23	24 SD 6

Although there is some scatter in the tensile test data, overall, the data suggest that there has been no change in the mechanical properties of the material caused by soaking with the various test gases.

Grade B Steel

Tensile test data for Grade B steel after the soaking period is shown in Table 6.

Table 6 : Tensile test data for Grade B steel

Material (as- manufactured)	Proof strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation at Break (mm)
Grade B Steel Control	489 SD 19	501 SD 17	6.8 SD 0.9
Grade B Steel in H_2	488 SD 27	503 SD 23	6.0 SD 1.0
Grade B Steel in CH_4	479 SD 14	495 SD 14	6.8 SD 0.6
Grade B Steel in G222	470 SD 4	487 SD 4	6.6 SD 0.7

The tensile test results for Grade B steel indicate no significant change in mechanical properties after the soaking period within the various test gases.

Copper

Tensile data for copper following the soaking period in the various test gases are shown in Table 7.

Table 7 : Tensile test data for copper

Material (as- manufactured)	Proof strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation at Break (mm)
Copper Control	159 SD 13	191 SD 4	10.9 SD 0.9
Copper in H_2	173 SD 4	199 SD 3	10.3 SD 0.6
Copper in CH_4	175 SD 3	197 SD 4	10.6 SD 1.4
Copper in G222	173 SD 5	199 SD 5	11.4 SD 1.0

Although some difference was observed between the proof stress values for the control sample compared to the gas-soaked samples, overall, the data suggest that gas soaking had no detrimental effect on the mechanical properties of the copper.

Brass

Tensile test data for 70:30 yellow brass samples consistent with that used in brass fittings at the test GDN, following the soaking period in the various test gases are shown in Table 8.

Table 8 : Tensile test data for brass

Material (as- manufactured)	Proof strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation at Break (mm)
Brass Control	146 SD 12	398 SD 8	19.8 SD 1.4

Brass in H_2	165 SD 8	401 SD 6	20.4 SD 2.3
Brass in CH_4	161 SD 2	403 SD 5	19.7 SD 0.4
Brass in G222	173 SD 4	398 SD 4	21.0 SD 1.1

Some differences were observed between the proof strength values for the control sample compared to the gas-soaked samples (146 against ~ 168 MPa). However, all gas-soaked samples exhibited similar changes in mechanical properties relative to each other. This suggested that the use of blended NG : H_2 gases was not markedly more detrimental in response to brass jointing than pure NG.

Overall, the data indicated that gas soaking had no detrimental effect on the mechanical properties of the brass between gas types.

Lead Free Solder

Table 9 shows the tensile test data for the lead free solder samples.

Table 9 : Tensile test data for lead free solder samples

Material (as- manufactured)	**Lap Shear Strength (MPa)**	**Ultimate Tensile Strength (MPa)**	**Elongation at Break (mm)**
Lead Free Solder Control	------	240 SD 1	13 SD 0.7
Lead Free Solder in H_2	19.4 SD 0.6	238 SD 0.9	8 SD 5.1
Lead Free Solder in CH_4	19.8 SD 0.4	237 SD 3	10 SD 4.2
Lead Free Solder in G222	15.9 SD 3.9	237 SD 4	8 SD 5.6

As detailed in Table 4, the lead free solder samples were produced as 5mm lap shear joints between copper plates as opposed to the standard “dog bone” of the ISO standard. This provided opportunity for the samples to fail in two different modes:

- Failure in the overlap (lap shear failure), and
- Failure in the copper base metal

Table 9 shows a mix of sample test data including lap shear data (relating to failure in the joint) and ultimate tensile strength (UTS) data (relating to failure in the copper support). This mixture of data are present as different specimens showed differing failure modes during testing and is summarised as:

- All control specimens failed in copper support
- For Hydrogen stored specimens, 4/7 failed in the copper support, and 3/7 in the solder lap joint
- For Methane stored specimens, 5/7 failed in the copper support, 2/7 in the solder joint
- For G222 stored specimens, 3/7 failed in the copper support and 4/7 in solder joint

Although the failure mode for the test samples differed between the soaked sample and control sample, the data appears to suggest that all soaked samples fail at approximately the same lap shear strength and in similar modes (i.e. near equal number of failures in the joint and the support for each sample type). This suggested that, although some change in failure mode may be encountered due to gas contact with the soldered joint, the magnitude of that failure is the same for each gas types. This in turn suggests that the use of blended gases within the test site will perform similarly to that of NG

Lead Solder

Table 10 shows the tensile test data for the lead-based solder samples.

Table 10 : Tensile test data for lead based solder samples

Material (as- manufactured)	Lap Shear Strength (MPa)*	Elongation at Break (mm)*
Lead Solder Control	10.4 SD 3.0	1.1 SD 0.2
Lead Solder in H_2	8.4 SD 3.8	0.9 SD 0.5
Lead Solder in CH_4	8.7 SD 6.1	1.2 SD 0.5
Lead Solder in G222	12.9 SD 4.7	1.2 SD 0.5

No data is provided for Ultimate Tensile Strength within Table 10 as all samples failed in the solder joint (by lap shear failure).

Although there is some minor variation within the test data, the data indicates that there is no significant difference between the control sample and those samples soaked under methane and the 20 % hydrogen blend. Failure in the joint may be due to bonding defects rather than the effect of hydrogen.

Cast Iron

Table 11 shows the tensile test data for the cast iron samples.

Table 11: Tensile test data for cast iron samples

Material Cast Iron EN 1561-GJL250	Strain at break (%)	Ultimate Tensile Strength (MPa)	Elongation at Failure (mm)
As manufactured	1.53 SD 0.5	268 SD 80	0.6 SD 0.2
Hydrogen soaked	1.15 SD 0.3	193 SD 25	0.5 SD 0.1
Methane soaked	1.80 SD 0.3	239 SD 45	0.7 SD 0.1
G222 soaked	1.52 SD 0.6	222 SD 81	0.6 SD 0.3

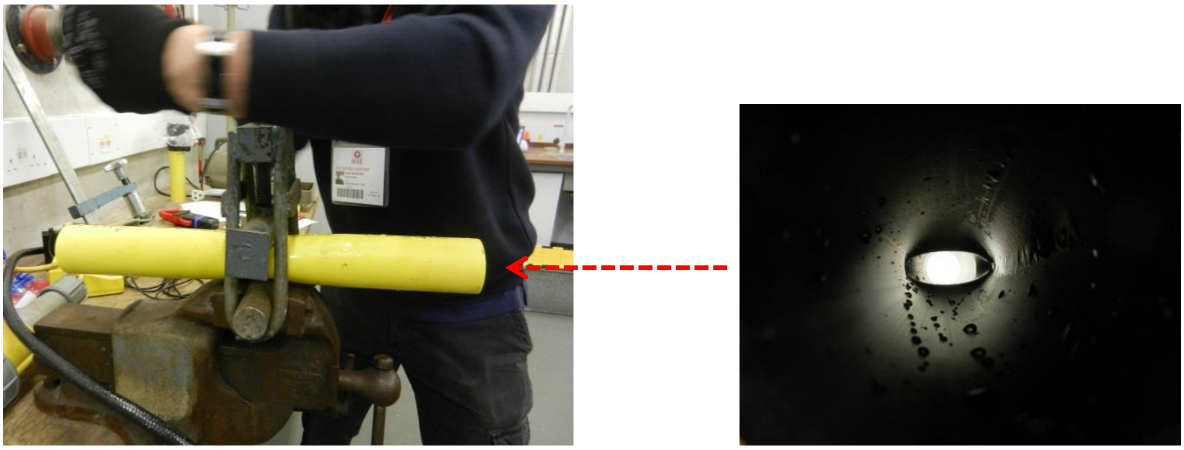
Although there is some variation within the test data, the data suggest that there is no significant difference between the control sample and those samples soaked under the various test gases. This was an unexpected result as literature studies suggested that cast iron was particularly susceptible to hydrogen ingress.

Due to these additional concerns, metallographic examination of the specimens was undertaken by optical microscopy and showed that microstructural influences such as variation in specimen to specimen grain size, graphite flake size and shrinkage had likely contributed to the tensile test results. As these contributors were independent of the soaking trials and gas composition, it was concluded that the presence of 20 % hydrogen blend had not adversely affected the tensile properties.

Pipe Performance Tests

Squeeze off Studies

Following pipe chilling to 0 Deg.C, the PE sample was squeezed-off, in accordance with industry standards, until both inner walls (top and bottom) were in contact as shown in Figure 3.



External view of pipe with squeeze-off tool

Internal view (inner walls) of pipe midway during squeeze off

Figure 3 : Squeeze-off of hydrogen soaked, 63mm PE80 pipe

On completion of the squeeze-off, the sample was placed back into the ice bath at 0 ºC for 1 hour with the squeeze off tool in place.

Once the hour-long cold soak had been completed, the squeeze off tool was removed within the 1 minute time specified in the industry standard and the pipe manually re-rounded using the approved tool (as shown in Figure 4).

Figure 4 : Re-rounding PE80 Pipe After Completion of Squeeze-off

To assess whether the squeeze off procedure had had a detrimental effect on the gas retention efficiency of the pipe, a single point hydrostatic test (water in water) was performed at 80 ºC for 165 hours with an applied pressure of 9.7 bar (actual pressure) equating to a Hoop Stress of 4.5 MPa: the assembled sample is shown in Figure 5 and a second control (un-squeezed pipe) was also similarly prepared.

Figure 5 : PE80 pipe prepared for hydrostatic testing

Post Squeeze-off Analysis

Once all tests were completed, samples were inspected using dye penetrant inspection (DPI) for evidence of stress crack onset from the pipe bore. X-ray radiography was also used to inspect for mid-wall voids. Figure 6 shows the sectioned squeeze off tube from the hydrogen soaking trial after hydrostatic testing.

Figure 6 : Sectioned view of hydrogen PE pipe 16413 following hydrostatic testing

The figure shows an even colour over the surface of the sectioned area indicating that there was no cracking to the body of the PE during the squeeze off nor hydrostatic testing. X-ray analysis also indicated no void formation. A similar result was observed for the control sample with both the hydrogen soaked and un-soaked control sample passed the test as defined in the controlling standard.

Electrofusion Jointing

Electrofusion jointing was achieved by electrical heating of the ends of two 63mm hydrogen soaked pipes to achieve softening and melting within an external collar. Controlled cooling resulted in formation of a homogeneous pipe-to-pipe weld between the two ends. Post weld assessment for weld integrity was undertaken to ensure weld quality.

Electrofusion jointing was carried out in both the socket (end-to-end) orientation and as a saddle joint (90 Degree orientation). After bond formation, water-in-water hydrostatic testing was undertaken with each sample held at 80 ^{o}C.

Six samples of the socket joint type and two samples of the saddle joint type were produced for testing. One set of three socket joint samples was tested for 165 hours at a higher hoop stress value (4.5 MPa) than that specified in the industry standard to provide rapid indications of any potential detrimental effects of hydrogen soaking. The other set of 3 socket joints was tested for the required 1000 hours storage time at the industry standard hoop stress of 4.0 MPa. After testing was completed, all the samples were assessed by X-ray and dye penetrant inspection (DPI): no evidence of stress cracking was found by DPI nor X-ray on any of the samples tested.

In addition to the X-ray and DPI analyses, some samples were crash tested, during which the quality of the joint was evaluated by compressing either side of the pipe that has been bonded to the fitting. If defective, the region between the pipe and the joint would be expected to de-bond or become detached as the compression is applied. Post-crash test, the samples were sectioned and assessed by DPI. Figure 7 shows a representative photographic record from testing indicating that the region between the pipe and the fitting (i.e. the formed joint) with the joint remaining intact after compression.

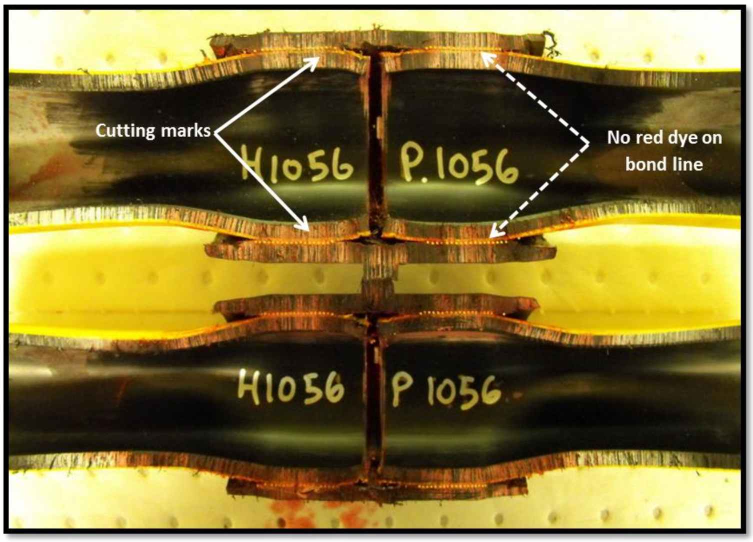


Figure 7 : Sectioned view of crash tested electrofusion welded hydrogen PE pipe following hydrostatic testing

CONCLUSIONS

A series of materials identified as being present on the test site GDN have been investigated to assess their possible reactions with blended NG: 20 % H_2 gases. The conclusions are summarised in Table 11.

Material	Summary
Medium Density Polyethylene (MDPE) PE80	Hydrogen absorption does not affect subsequent squeeze-off or electrofusion welding of pipework.
Low strength steel such as API5L grade B	Although literature suggests the interaction of hydrogen with Grade B steel at high pressure may lead to increased fatigue, practical testing indicated that no change to tensile properties is observed at gas pressures and compositions typical of the test site GDN.
Cast Iron	No significant change in tensile properties for the 20 % hydrogen blend.
Copper	No change in tensile properties was observed following hydrogen soaking.
Yellow Brass	Tensile testing of brass test specimens indicated that there was no change in the tensile properties of the metal after storage in a hydrogen, methane or blended gas environment up to 20 % hydrogen.

Material	Summary
Lead Free Solders	Although there was a change in failure mode for lap shear test specimens after storage in various gaseous environments, the failure did not vary with gas composition: this indicated that the integrity of tin / copper solders in blended gases would not differ from their integrity in natural gas.
Lead Based Solders	During lap shear testing there was no change in tensile properties between soldered joints soaked in a range of control gases and that of an un-soaked control.

Overall, all the materials examined were found to be acceptable for use within a blended NG : H_2 gas feed within the range of 0 – 23% mol% H_2 and 0-2 barg operating pressure.

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