Effect of Hydrogen Pressure on the Fretting Behavior of Rubber Materials

Géraldine Theiler, Natalia Cano Murillo and Andreas Hausberger

Abstract: Safety and reliability are the major challenges to face for the development and acceptance of hydrogen technology. It is therefore crucial to deeply study material compatibility, in particular for tribological components that are directly in contact with hydrogen. Some of the most critical parts are sealing materials that need increased safety requirements. In this study, the fretting behavior of several elastomer materials were evaluated against 316L stainless steel in an air and hydrogen environment up to 10 MPa. Several grades of cross-linked hydrogenated acrylonitrile butadiene (HNBR), acrylonitrile butadiene (NBR) and ethylene propylene diene monomer rubbers (EPDM) were investigated. Furthermore, aging experiments were conducted for 7 days under static conditions in 100 MPa of hydrogen followed by rapid gas decompression. Fretting tests revealed that the wear of these compounds is significantly affected by the hydrogen environment compared to air, especially with NBR grades. After the aging experiment, the friction response of the HNBR grades is characterized by increased adhesion due to elastic deformation, leading to partial slip.

Keywords: fretting wear; rubbers; hydrogen; high-pressure

1. Introduction

With the development of hydrogen infrastructure, a growing interest is devoted to research on material compatibility with hydrogen due to high safety requirements in distribution and dispensing infrastructure. Some of the most critical parts are sealing materials that need increased reliability specifications to avoid any leakage, which would lead to an imminent risk of serious damage but also mitigate the acceptance of this technology [1,2].

For static and dynamic seals, polymeric materials are used as sealing components in a wide range of conditions, such as O-rings and piston rings in high-pressure and/or cryogenic hydrogen. Rubber O-ring seals have been commonly used in high-pressure hydrogen storage systems for preventing the leakage of hydrogen gas. However, failure of a sealing component can occur due to swelling induced by dissolved hydrogen [3,4]. It has been reported that most damage to the sealing materials occurs during rapid gas decompression (RGD). Therefore, more and more studies are dedicated to the influence of high-pressure hydrogen and RGD on the properties of rubber materials [5–19]. Most of the works reported the effect of the fillers and additives on the physical properties of the elastomer compounds.

Volume change upon RGD inevitably induces motion of the sealing material, particularly during compression cycles. Further low amplitude oscillating motion can also occur in the reciprocating O-ring seal [15]. It is therefore crucial to characterize the friction and wear of the rubber materials in hydrogen as well as after RGD where more effects occur. Up to now, most literature refers to the effect of the hydrogen environment on the sliding friction and wear of polymers [20–26] or rubbers [27,28].

Works have been published on EPDM and NBR grades under 28 MPa hydrogen in a linear reciprocating custom-built apparatus [27,28]. Both rubber materials showed a higher...
coefficient of friction (COF) and drastically reduced wear behavior in all hydrogen exposed
tests compared with the test results in ambient air conditions. It was suggested that the
variations in the friction and wear behavior of EPDM and NBR are due to a combined effect
of high-pressure hydrogen and the plasticizer and fillers [28].

Choi et al. studied the friction and wear behavior of NBR rubber under continuous
sliding in air after high-pressure exposure in hydrogen [29]. They reported that the
hydrogen-induced damage was related to the tribological properties of filled NBR.

Reports on fretting experiments in hydrogen are rare [30]. Zhou et al. developed a
numerical model to investigate the fretting characteristic of O-ring seals under the action
of swelling behavior induced by dissolved hydrogen [30]. They concluded that the amplitude
of reciprocating motion affects the fretting state of rubber seals, and that a high-pressure
hydrogen atmosphere promotes a sticking regime because of swelling of the rubber.

The scope of this study is to characterize the fretting behavior of selected rubber
materials against 316L steel under hydrogen atmosphere. The focus lies here on the friction
behavior and the characterization of the wear scar of the rubber materials. The core of the
study deals with the influence of the hydrogen pressure on the friction performance com-
pared to air. Tests were performed in gaseous hydrogen at 0.1 MPa and 10 MPa. In addition,
aging experiments were conducted under static conditions in 100 MPa hydrogen, followed
by further fretting tests under hydrogen immediately and 24 h after decompression. This
work gives an inside view of the relevant mechanisms in seal contact under a hydrogen
atmosphere and thus make an important contribution to material and seal development.

2. Materials and Methods

2.1. Materials

This study includes two different types of cross-linked hydrogenated acrylonitrile
butadiene (HNBR). Both are filled with CB in different compositions and the acrylonitrile
(ACN) content is relatively low, at 21%, to keep a low glass transition temperature and to
have good low temperature properties. Furthermore, four grades of nitrile butadiene rubber
specially developed for this project were investigated. The effect of vulcanization, filler
and plasticizer have been considered in this study as indicated in Table 1. In addition, two
sulfur-cured EPDM rubber grades with different carbon black were tested. All materials
were provided as a 2 mm sheet by Arlanxeo Deutschland GmbH, Dormagen, Germany.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Curing</th>
<th>Fillers</th>
<th>Hardness (ShA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HNBR-CB</td>
<td>Peroxide</td>
<td>carbon black (75 phr)</td>
<td>79</td>
</tr>
<tr>
<td>HNBR-CB-PA</td>
<td>Peroxide</td>
<td>carbon black (67 phr) + PA (10 phr)</td>
<td>82</td>
</tr>
<tr>
<td>NBR-Sil</td>
<td>Sulfur</td>
<td>silica (60 phr) + SCA 2 phr</td>
<td>70</td>
</tr>
<tr>
<td>NBR-CB-perox</td>
<td>Peroxide</td>
<td>carbon black (70 phr) (MgO)</td>
<td>81</td>
</tr>
<tr>
<td>NBR-CB</td>
<td>Sulfur</td>
<td>carbon black (75 phr)</td>
<td>76</td>
</tr>
<tr>
<td>NBR-CB-plast</td>
<td>Sulfur</td>
<td>carbon black (95 phr) + plast. (10 phr)</td>
<td>78</td>
</tr>
<tr>
<td>EPDM1</td>
<td>Sulfur</td>
<td>carbon black (100 phr)</td>
<td>77</td>
</tr>
<tr>
<td>EPDM2</td>
<td>Sulfur</td>
<td>carbon black (120 phr)</td>
<td>81</td>
</tr>
</tbody>
</table>

The samples were cut to 15 mm × 10 mm, cleaned with isopropanol and exposed to
dry heat at 60 °C for 48 h. This step was undertaken to remove any moisture and outgassing
of the samples that might affect the friction properties.

2.2. Test Method

The test method is described in Figure 1. “As-received sample” means here the cleaned
and dried sample. As-received samples were tested at ambient temperature in air and in
hydrogen at 0.1 MPa and 10 MPa. Some samples were aged in high-pressure autoclaves
according to the methodology described in [13]. After aging experiments, further fretting tests were performed immediately after decompression and 24 h later.

**Figure 1.** Exposure and test methodology.

Friction tests were performed in a fretting tribometer developed at BAM for hydrogen environments up to 10 MPa (Figure 2). The tribometer is designed as an insert for a stainless-steel autoclave with a maximum gas pressure of 10 MPa. The normal and frictional forces are measured using strain gauges that are attached to deformation bodies in a bridge circuit. The normal force is generated by a spring using an electric motor stretched via a spindle drive. The tribometer is driven by a voice coil drive, which generates the reciprocating motion. Both the normal force and the tangential displacement are set via PID control loops.

**Figure 2.** PT1 tribometer and ball-on-disc configuration.

The upper specimen is a 316L stainless steel ball with a diameter of 6.0 mm (G300). The lower specimen is the rubber sample fixed on a steel flat.

The friction measurement of rubber materials is complex and often raises some issues with regards to the setup. Since most rubber samples are available as a plate, friction tests are mostly conducted with a flat rubber material sliding against a steel ball or cylinder in a fretting test [27,31–34] or against a flat counterpart in a linear motion [35]. Other configurations found in the literature are a hemispherical rubber pin or a rubber cylinder against a steel disc [36,37]. In this study, flat rubber samples were used.

The normal load was set to 5 N, the frequency to 10 Hz and the stroke to 2 mm, with a total of 20,000 cycles. Tests were performed at room temperature in air (moisture range 40–60%) and in gaseous hydrogen (H$_2$O < 5 ppm) at 0.1 MPa and 10 MPa. Three tests were performed for each condition.
2.3. Friction Measurement

During the test, friction force, $F_f$, values were recorded along with time and displacement. The hysteresis data contains 1000 values per second, leading to 100 points for each cycle. Therefore, after 10 min, the values were recorded every 20 s.

The average friction force ($F_f$ mean) was calculated as follows:

$$F_f \text{ mean} = \frac{\sum_{i=1}^{n} |F_f|}{n},$$

where $n$ is the number of points.

The average friction coefficient (COF mean) was calculated as follows:

$$\text{COF mean} = \frac{F_f \text{ mean}}{F_N},$$

where $F_N$ is the normal load.

2.4. Surface Analyses and Wear Measurement

Worn surfaces of the rubbers and transfer film formed on the discs were inspected by means of an optical microscope (VHX-500, Keyence Deutschland GmbH, Neu-Isenburg, Germany), a confocal 3D profilometer (µsurf expert, NanoFocus AG, Oberhausen, Germany) and a scanning electron microscope (SEM, Supra™40, Carl Zeiss AG, Oberkochen, Germany) equipped with an energy dispersive X-ray spectrometer. Chemical analyses were performed by micro-ATR-IR attenuated total reflection (Hyperion 3000, Bruker Optics, Ettlingen, Germany) with a germanium crystal. The wavelength range was set from 4000 cm$^{-1}$ to 500 cm$^{-1}$. An atmospheric compensation, an extended ATR correction and a baseline correction were carried out for all spectra. After the test, the wear scar on the rubber surface was characterized by a 3D profilometer and the wear volume was measured based on the 3D profile, considering the area below the reference line (Figure 3).

Figure 3. Profile of the wear scar: wear volume corresponds to the red area.

3. Results and Discussion

3.1. Friction Tests on as Received Samples: Effect of Hydrogen Environment

3.1.1. Friction Force Curves

Figure 4 depicts the variation of the friction force ($F_f$) versus relative displacement ($D$) as a function of time. The friction loops characterize friction and deformation behavior at the interface. The area of the loops corresponds to the energy dissipated during the cycles, but this was not calculated in this study. As described in [37–39], the fretting mechanism can be deduced from the shape of the fretting hysteresis, which can be linear, elliptical and parallelogram shapes. This corresponds to the three fretting regimes, namely partial slip, mixed fretting and gross slip regime [39].

As shown in Figure 4, the loops of the rubber materials described an ellipse at the beginning of the tests and move toward a quasi-parallelogram, whereby the slope corresponds to the static friction and the horizontal segment to the sliding regime. The fretting regime changes from partial to gross slip. The transitional phase takes place after several cycles for most materials.
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For the HNBR and NBR grades, the loop is rather uneven. The inclined slope reveals a large sticking phase on one hand, and the sliding regime is relatively unstable on the other hand.

For NBR-Sil, the frictional force is characterized by a first loop at the beginning of the test, which significantly increases with time. After that, the fretting regime reaches a stable state.

In comparison, EDPM materials have a relatively stable sliding friction, although the maximum friction force at the beginning of the test is higher than that at the end. This may be due to the increase of hardness within the first cycle, leading to the decrease of adhesion and hysteresis resistance between the rubber and the steel metal ball.

For each rubber grade, the shape of the loop is rather similar in air and hydrogen. The main differences are related to the maximal friction force and the calculated friction coefficient.

Figure 4. Friction force–D curves of HNBR-CB (a), HNBR-CB-PA (b), EPDM1 (c), EPDM2 (d), NBR-Sil (e), NBR-CB-perox (f), NBR-CB (g) and NBR-CB-plast (h) in air, and in hydrogen at 0.1 MPa and 10 MPa (dashed line: begin; solid line: end of the test).
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3.1.2. Average Friction Coefficient

Figure 5 shows the exemplary evolution of the friction coefficient as a function of time of several grades tested in ambient air and in hydrogen at 0.1 MPa and 10 MPa. As indicated in Figure 5a,b, the friction coefficient of HNBR-CB and EPDM2 are higher in a hydrogen environment compared to the values in air. For both rubber materials, the COF increases rapidly at first before decreasing to a stable value for EPDM2. The COF of HNBR-CB, however, gradually increases in hydrogen after this descent stage.

With regard to the NBR grades, the COF in air is higher than in the hydrogen environment (Figure 5c–f), with similar friction curves independent of the hydrogen pressure. It is noticeable that the COF of NBR-Sil increases gradually and slowly in all conditions, reaching a steady state friction only after 1000 s (10,000 cycles). This may be related to the lower hardness of NBR-Sil compared to the other grades, leading to a larger deformation.
and an increased contact area, as observed in [40]. As a comparison, the friction curves of CB-filled NBR grades reach a stable friction state after a short running in phase.

An overview of the mean friction coefficient of all rubbers measured at the end of the tests is presented in Figure 6a. The COF of HNBR and EPDM increase slightly with hydrogen pressure, while lower values were obtained in hydrogen with all NBR grades. Comparing the HNBR grades, the addition of a PA filler leads to a slight increase of the friction coefficient, independent of the environment, while increasing the CB content in the EPDM grades results in a minor decrease in the COF. Among the sulfur-cured rubber, both CB-filled grades, NBR-CB and NBR-CB-plast, have a lower COF compared to the SiO₂-filled NBR-Sil. This is consistent with another previous study performed in air [41]. Further, it is noticeable that the peroxide-cured NBR-CB-perox grade has a higher COF than the sulfur-cured grades under both air and hydrogen conditions. As mentioned in [42], the peroxide-cured CB-filled NBR grade shows the stiffest material behavior with the highest crosslink density among these NBR grades. Due to the restriction of its polymer chain movements, a higher shear strength is expected, which could lead to increased adhesion.

The influence of hydrogen on the friction coefficient of EPDM and HNBR grades are in accordance with the work done by Kuang et al. [28], who found that the COF of EPDM against steel is larger under high-pressure hydrogen than that measured in ambient air under reciprocating sliding. Concerning NBR materials, however, discrepancies in the results were found compared to [28]. This is possibly due to different formulations and testing conditions, namely sliding friction and hydrogen pressure. Wang [43] observed different trends between reciprocating and fretting behavior of several polymers in air, due to more energy generated at the surface during fretting. On the other hand, at very high-pressure hydrogen the sticking regime of rubber materials should be promoted because of the swelling effect [30]. In any case, as mentioned in [44], the fretting characteristic of polymers is strongly related to the frictional heat produced during fretting and the heat resistance of the materials. It is therefore reasonable to suggest that the cooling effect of the hydrogen environment, which has a higher thermal conductivity than air, affects the deformation and therefore the fretting behavior of these rubber materials. However, this should not be the only influencing parameter since different results were observed depending on the materials.
3.1.3. Effect of Hydrogen on the Wear Volume

Figure 6b gives an overview of the wear volume obtained by 3D profilometry. For most rubber materials, the wear values are similar or reduced in a hydrogen environment, which is in good agreement with [28]. The wear value of the rubbers is rather stable for HNBR-CB and EPDM2 but decreases for HNBR-CB-PA and EPDM1, which have respectively a lower amount of carbon black compared to the former grades.

Similarly, by comparing the results of the CB-filled sulfur-cured NBR grades, it is noticeable that increasing the CB content from 75 ppm to 95 ppm leads also to higher wear in hydrogen. This is possibly related to the restriction of the polymer chain with increased CB loading, although the addition of a plasticizer to the NBR-CB-plast compensates the stiffness of higher filler loading as reported in [42].

On the other hand, although the silica-filled rubber (NBR-Sil) has a higher friction coefficient compared to the CB-filled NBR, the wear values of both rubber materials were similar in air, as well as in hydrogen.

Therefore, other factors should be taken into consideration. HNBR-CB-PA contains polyamide fillers and as reported in [23], the tribological properties of polyamides are influenced by the environment. NBR-CB-plast contains plasticizers which may also have an effect on the wear behavior in air and in hydrogen.

Further, most significant effects were obtained with NBR-CB-perox, which is a peroxide-cured rubber with additives. It has the highest wear values among the NBR materials in air and the lowest in hydrogen. It is therefore appropriate to suggest that either the curing process and/or the additive have a major influence on the wear properties of these rubber materials in hydrogen. This will be discussed in the following section.

3D profile images of the wear scar were used to evaluate the wear damage of the rubbers. Figure 7 illustrates examples of some of them. Deeper wear scars were obtained after the experiment in air compared to those performed in hydrogen.

![3D profilometer images of the wear scar](image-url)

Figure 7. 3D profilometer images of the wear scar of (a) NBR-Sil, (b) NBR-CB-perox, (c) NBR-CB and (d) NBR-CB-plast after tests in air (left) and in 10 MPa H₂ (right).
3.1.4. Surface Morphology and Wear Mechanism

Optical microscopy of the wear scar of selected rubber materials and associated counterfaces after testing in air and hydrogen at 10 MPa are compared in Figure 8.

(a) air

(b) air

(c) air

(d) air

Figure 8. Optical microscope images of the wear scar (left and middle) and of the ball (right) after tests in air and in 10 MPa H₂: (a) HNBR-CB, (b) EPDM2, (c) NBR-CB and (d) NBR-CB-perox.
As shown in Figure 8a, the wear scar of HNBR-CB is perpendicular to the fretting direction and forms a wavy structure, which is associated with adhesive wear [33]. Cracks are observed on the surface and propagated perpendicularly to the sliding direction after tests in air and hydrogen. Wear debris is present outside the contact in both testing conditions. The main difference is observed on the counterface. A large amount of rubber stuck to the steel ball after the hydrogen experiment. The friction mechanism is therefore mainly adhesive in hydrogen. This may explain the higher coefficient of friction for HNBR grades in hydrogen.

In comparison, the wear damage of EPDM2 is visibly milder than for the HNBR grade. No visible cracks were detected at the surface of the rubber but a typical wave pattern perpendicular to the fretting direction was observed (Figure 8b). Wear particles are seen on the ball in both conditions.

Figure 8c displays the worn surface of the sulfur-cured NBR filled with CB (NBR-CB). While a wave pattern is detected after testing in air, the wear scar in hydrogen is covered with some wear debris. These fine particles are also present on the counterface and more abundant in hydrogen conditions. These particles may act as a solid lubricant in the contact area, reducing the friction and wear of the rubber [36].

Figure 8d presents the surface images of the CB-filled peroxide-cured NBR grade (NBR-CB-perox). Significant differences are observed compared to the previous sulfur-cured NBR. After testing in air, a torn tongue perpendicular to the sliding direction is observed, similar to HNBR. It is therefore likely that the curing process affects the material properties as mentioned in [41], and therefore the friction behavior. After the hydrogen test, however, ploughing marks parallel to the sliding direction are present, with no visible stick regions between the two contacting surfaces. This suggests that abrasive wear was more likely to occur. The formation of a thin transfer film on the counterface leads to reduced friction and wear in hydrogen.

To characterize further the fretting mechanisms and the influence of additives and fillers in the NBR grades, further EDX was performed on the wear scar of the rubber after testing in air and hydrogen at 10 MPa. Figure 9 collects the EDX maps of NBR-Sil, NBR-CB-perox and NBR-CB-plast.

From Figure 9a, a homogenous distribution of the silica fillers in the wear scar after testing in both conditions can be deduced. ZnO, however, appears more pronounced at the surface of the rubber after the hydrogen experiment. Similarly, more additives (ZnO and MgO) were detected on the surface of the wear scar of NBR-CB-perox after hydrogen exposure (Figure 9b). The EDX analyses of the NBR-CB-plast indicate a significant agglomeration of additives and plasticizers (identified by Zn and S, respectively) after the hydrogen experiment (Figure 9c). It seems that hydrogen promotes the migration and agglomeration of the additives towards the surface of the material.

Additional ATR-IR spectroscopy of selected rubber materials were performed to detect possible chemical reactions during the friction process.

Figure 10 depicts the ATR-IR spectra of the HNBR, EPDM and NBR grades. No significant chemical reactions could be observed but some increase in signal intensity was detected for several rubbers.

Wear scar analysis of HNBR-CB through FT-IR reveals a general broadening of the IR signal after testing in hydrogen in the region of 1521 and 860 cm$^{-1}$, possibly due to more exposed CB and higher absorption of the IR signal. As reported in [13], the exposure to hydrogen can cause a tendency of the CB fillers to come to the surface, absorbing in this range of IR. ATR-IR of the wear scar of EPDM1 indicates marginal changes; however, a small peak in 1735 cm$^{-1}$ corresponding to carbonyl group was detected on the sample tested in air, which could possibly be related to some oxidation on the worn surface.
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Figure 9. EDX maps of the wear scar after tests in air (top) and in 10 MPa H₂ (bottom): (a) NBR-Sil, (b) NBR-CB-perox and (c) NBR-CB-plast.
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Regarding the NBR-CB, testing in hydrogen brings small changes in the IR spectra; the 1517 cm$^{-1}$ band might be related to additives, such as antioxidants. The 969 cm$^{-1}$ band corresponds to the C-H out-of-plane bending band of the butadiene double bond. The cyano group in air and after testing in hydrogen, however, shows no significant change. While no chemical changes could be detected for NBR-Sil (the same for the broad peak of the silica filler at 1086 cm$^{-1}$ that is present in both conditions), some small changes are detected for the NBR-CB-perox materials; the peaks between 1690 and 1000 cm$^{-1}$ in NBR-CB-perox mostly related to CB, increasing slightly after testing in hydrogen, while 2358 cm$^{-1}$ is related to MgO. The 964 cm$^{-1}$ band corresponds to the C-H out-of-plane bending band of the butadiene double bond. The 910 cm$^{-1}$ band is related to the peroxide crosslinking additive. ATR-IR therefore confirms the EDX results, suggesting that the presence of a curing activator and an increase of CB at the surface. The dissolved H2 may act as plasticizer in the rubber materials, increasing the motion of the polymer and fillers. Upon friction, these particles move towards the surface, acting favorably at the friction contact.

Figure 10. ATR-IR of the wear scar after tests in air and in 10 MPa H$_2$: (a) HNBR-CB, (b) EPDM1, (c) NBR-CB, (d) NBR-Sil and (e) NBR-CB-perox.
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3.2. Friction Tests on Exposed Samples: Effect of High-Pressure Exposure Followed by Rapid Gas Decompression

Following the test method described in Figure 1, further samples were aged in high-pressure hydrogen at 100 MPa for 7 days at 120 °C for HNBR and at 85 °C for the EPDM grades, respectively. A day before the end of the exposure period, the temperature was switched off to cool down the specimens before taking them out and the pressure was adjusted to replenish the pressure loss due to the cooling effect. This step is necessary to allow the characterization of the cooled sample immediately after decompression, without a temperature effect.

After exposure at 100 MPa, friction tests were performed at 10 MPa immediately after decompression and one day after. Figure 11a shows the hysteresis of HNBR-CB-PA immediately after exposure. The loop has an elliptical shape corresponding to partial slip. The displacement occurs only on the edge part and is mainly due to elastic deformation of the elastomer. The effect of the high-pressure hydrogen on the physical and mechanical properties were studied and published in [13]. Accordingly, the swelling of the rubber immediately after the RGD significantly decreases the hardness of the rubber. This friction process is mainly ascribed to the deformation of the rubber. By repeating the friction tests 24 h later, the hysteresis almost retrieves a parallelogram shape as the volume of the rubber recovers (Figure 11b). The elastic deformation, however, is still predominant.

As a comparison, further tests were performed with the EPDM2 grades. The friction response in 10 MPa hydrogen was similar to before (Figure 4) and after aging in high-pressure hydrogen (Figure 11c,d). This can be related to a relatively stable behavior of the physical and mechanical properties of EPDM2, as reported in [45]. Other works revealed similar trends regarding the aging of elastomers in hydrogen [46], where EPDM material showed the least degradation compared to the HNBR grade.
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4. Conclusions

In this study, the fretting behavior of several elastomer materials against a 316L ball were evaluated in an air and hydrogen environment up to 10 MPa. Several grades of cross-linked hydrogenated acrylonitrile butadiene (HNBR), acrylonitrile butadiene (NBR) and ethylene propylene diene monomer rubbers (EPDM) were investigated.

The influence of the hydrogen environment was studied at first on as-received samples. Based on the friction results and wear characterization, the following conclusions can be drawn:

- Under the testing conditions (a normal load of 5 N, frequency of 10 Hz, a 2 mm stroke), the fretting behavior was in the gross slip regime for all rubber materials. The shape of the loop was similar in hydrogen compared to air, and the influence of hydrogen pressure was relatively small, although some effects were seen on the HNBR and EPDM grades. The average friction coefficient and the wear volume, however, were affected differently by the hydrogen conditions depending on the materials. This suggests that the cooling effect of the hydrogen environment is not the only influencing factor.

- The friction of the HNBR and EPDM grades increased with hydrogen pressure. Adhesive wear is predominant in hydrogen in CB filled HNBR, while the addition of PA fillers reduced wear.

- Concerning NBR grades, reduced friction and wear were measured for all grades in hydrogen. It was found that the curing process and the additive have a major influence on the wear properties of these rubber materials in hydrogen. Most significant effects were obtained with the peroxide-cured rubber, having the highest wear value among the NBR materials in air and the lowest one in hydrogen. Among the sulfur-cured NBR grades, lower friction was achieved with CB compared to SiO 2 fillers.

- No significant chemical reactions were detected by means of ATR-IR, apart from a possible oxidation on the worn EPDM surface tested in air. However, both EDX and ATR-IR analyses revealed migration and agglomeration of the additives at the friction contact of the NBR grades in hydrogen, acting favorably on the friction and wear resistance of the rubbers.

The influence of high-pressure exposure followed by rapid gas decompression was studied on aged samples. Significant effects were observed immediately after decompress-

Figure 11. Friction force versus displacement for HNBR-CB-PA immediately (a) and 24 h (b) after finishing the high-pressure exposure with corresponding optical microscopy images and 3D profile measurements of the wear scar; Friction force versus displacement for (c) EPDM2 immediately and 24 h (d) after finishing the high-pressure exposure.
sion for the HNBR material, which is related to the softening of the rubber after RGD. EPDM rubber, however, was less affected by the aging experiment and therefore might be more suitable for high-pressure applications than HNBR grades.

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