

VDMA
Fluid Power Association

22nd

ISC

International Sealing Conference

Stuttgart, Germany
October 01 - 02, 2024

Sealing Technology –
Challenges accepted!



© 2024 VDMA Fluidtechnik

All rights reserved. No part of this publication may be reproduced, stored in retrieval systems or transmitted in any form by any means without the prior permission of the publisher.

ISBN 978-3-8163-0768-6

Fachverband Fluidtechnik im VDMA e. V. Lyoner Str. 18

50628 Frankfurt am Main

Germany

Phone +49 69 6603-1513

E-Mail maximilian.baxmann@vdma.org

Internet www.vdma.org/fluid

Friction and wear properties of several rubber materials for high pressure O-ring

Hiroyoshi Tanaka, Ayako Aoyagi, Hikaru Hashimoto, Yoshinori Sawae, Joichi Sugimura

Friction and wear characteristics of rubber materials were investigated to look for the suitable candidates for O-ring using in hydrogen energy system for renewable energy society. In this study, reciprocating sliding test of rubbers were conducted under hydrogen environment. To understand the sealing performance and durability of rubber O-rings, friction and wear characteristics of hemispherical rubber specimens sliding against stainless steel disk were evaluated. Several rubber materials including silicone rubber, EPDM rubber, fluorinated rubber and natural rubber based elastomer were selected to identify the best fit for the hydrogen facility. Each rubber material exhibited unique friction and wear performance depending on its nature (mechanical properties, chemical composition, type of filler), environment gases and operating conditions. A silicone rubber demonstrated that low wear and high and stable coefficient of friction in hydrogen compare with that in Air. A fluorinated rubber with carbon black filler showed low coefficient of friction in hydrogen. In order to understand the wear process of each rubber, topography measurement and surface analysis were conducted after the sliding test.

1 Introduction

Hydrogen energy system for renewable energy society that handles high pressure hydrogen gas requires high safety performance and high environmental compatibility. Rubber O-ring is usually used in many components as simple, reliable sealing element. Further efforts are requested to improve durability which directly connected to the lifecycle and/or maintenance period of component[1,2,3]. Friction and wear properties of rubber materials were investigated to look for the suitable candidates for O-ring in hydrogen energy system.

In this study, reciprocating rotational sliding test of rubbers are conducted under hydrogen and the other gas environments. Friction and wear properties of rubber specimens slide against stainless steel are evaluated to understand the seal performance and durability of rubber O-ring.

2 Tests and results

A reciprocating sliding tests were carried out using tribo tester with environment control chamber. Hemispherical rubber specimen and stainless steel disk specimens were used as a tribo-pair. Several rubber materials were selected to identify the most suitable materials for hydrogen facilities. In order to understand the progress of wear on each rubber specimen, surface observation and chemical analysis were performed after the sliding test.

2.1 Experimental

In order to obtain the useful information for hydrogen facilities, it is necessary to carefully design the experimental set up[4,5,6].

2.1.1 Test rig

Pin-on-disk sliding tests were conducted in a chamber equipped with a turbo molecular pump and gas impurity control system [7] because impurities such as oxygen and water must be avoided from hydrogen gas environment. Also reciprocating sliding was selected to evaluate the wear on elastomer surface in the compression decompression process of high pressure vessel in hydrogen facilities.

2.1.2 Specimens

The dimensions of the pin and disk specimens are shown in Fig. 1. The disk was made of JIS SUS316L stainless steel. The pin specimens were moulded with hemispherical shape with 5mm diameter as shown in Fig. 1. The pin specimens were wiped with ethanol just before the sliding tests.

2.1.3 Test conditions

The disk specimen was mounted on a rotating shaft, while the pin was pressed against the disk specimen by a loading lever in the sealed chamber. After the specimens were set up, the chamber was evacuated to a pressure level of 1×10^{-4} Pa, and then the test gas was introduced into the chamber at a flow rate of 1.0 L/min. Impurities in the supplied gas were measured at the exhaust line with a moisture sensor and an oxygen sensor. Experiments started after trace water and oxygen in hydrogen were stabilized at 3 ppm and 0.5 ppm, respectively. All the sliding tests were conducted at 296 K in dry contact condition. Test load was 5 N. Test conditions are summarized in Table 1.

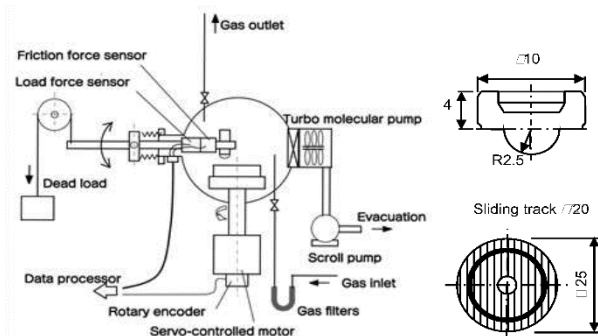


Figure 1: Test rig (a) and specimens (b, c) Roughness of disk surface: $R_z=0.045$

Table 1: Test conditions

Description	Values	Unit
Load	5	[N]
Stroke	1, 2, 3	[mm]
Sliding velocity	6	[mm/s]
Temperature	23 (R.T.)	[C]
Environmental gas	Hydrogen, Atmospheric air	[-]

2.2 Materials

In order to find a best candidate for hydrogen energy system, six elastomers were selected as shown below. NR means a natural rubber based material.

Table 2: Rubber test specimens

	A	B	C	D	E	F
	VMQ1	VMQ2	EPDM	FKM1	FKM2	NR
Hardness(IRHD)	81	81	82	93	82	74
Tensile strength(Mpa)	11	6.32	20.4	15.7	17.9	21.4
Elongation(%)	310	170	210	140	290	436

2.3 Surface analyses

During the sliding tests, friction force and normal force were measured. After the tests, the pin specimens were observed with an optical microscope and a confocal laser microscope, and analysed with Raman microscopy.

2.4 Test results

The typical results of reciprocating sliding test were summarized in Fig. 2 to Fig. 5. Basically, COF increased with increasing reciprocating stroke in all rubber samples. Rubber materials exhibited individual friction and wear performance depending on each environment and operating condition. VMQ rubbers showed lower COF than others in both hydrogen and air. VMQ rubbers showed a sharp drop in COF in air at stroke of 2mm and 3mm. In contrast, FKM showed a sharp drop in COF in hydrogen regardless of stroke distances. There were little differences in the changes in COF due to the difference in environmental gas with EPDM and Natural rubber. As the stroke distance increased, the COF also steadily increased. Natural rubber showed stable performance against friction and wear due to its soft and stable mechanical properties at room temperature. In summary, friction and wear properties strongly depended on the type of rubber base resin. On the other hand, rubber materials contained multiple elements included fillers besides of base resin, and the following surface analyses and detailed surface observation were carried out to know the effects of these functions.

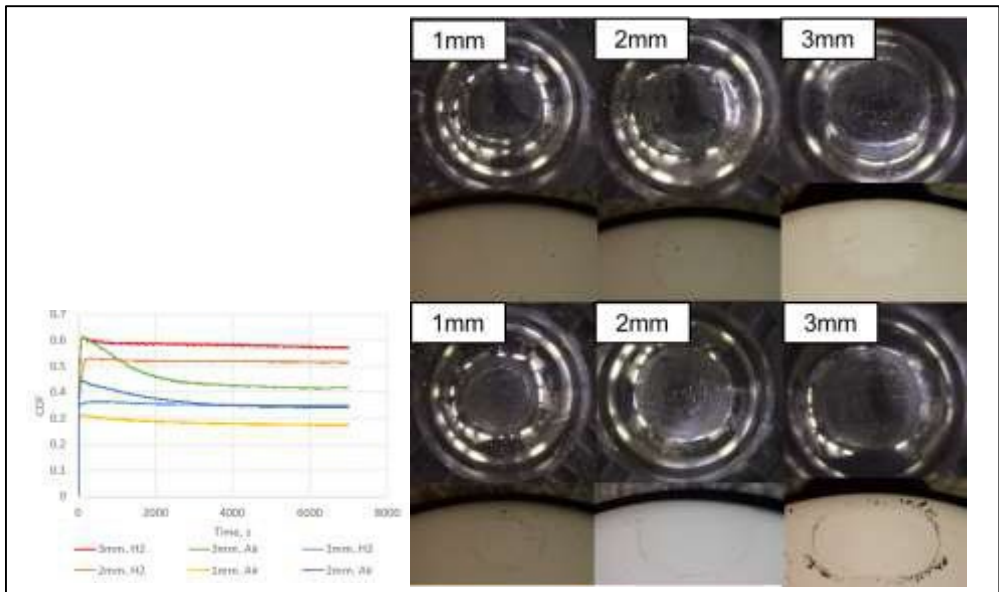


Figure 2: Changes in COF tested with VMQ1 and Photos after the sliding test (a) in hydrogen (b) in air

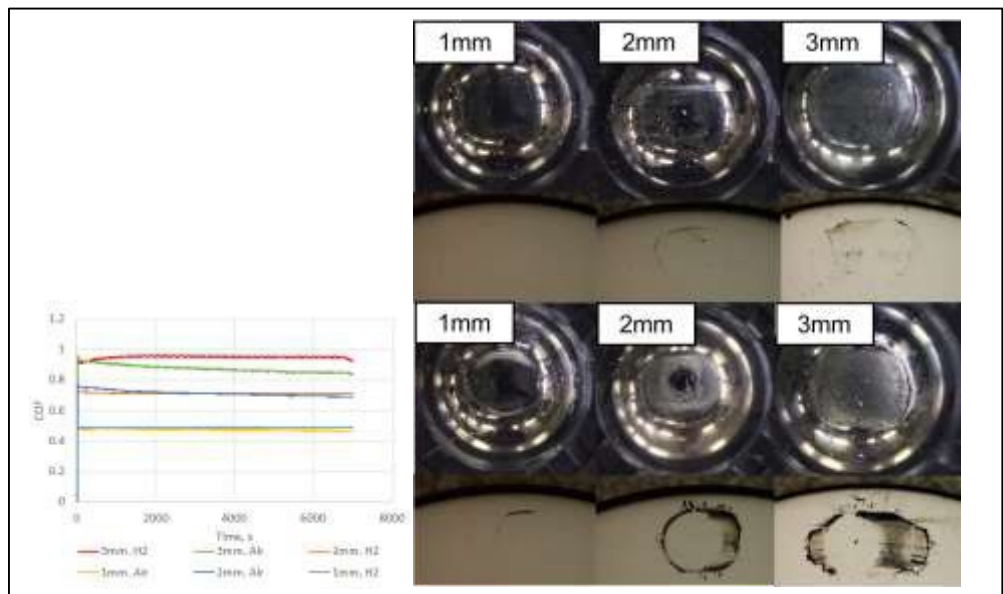


Figure 3: Changes in COF tested with EPDM and Photos after the sliding test (a) in hydrogen (b) in air

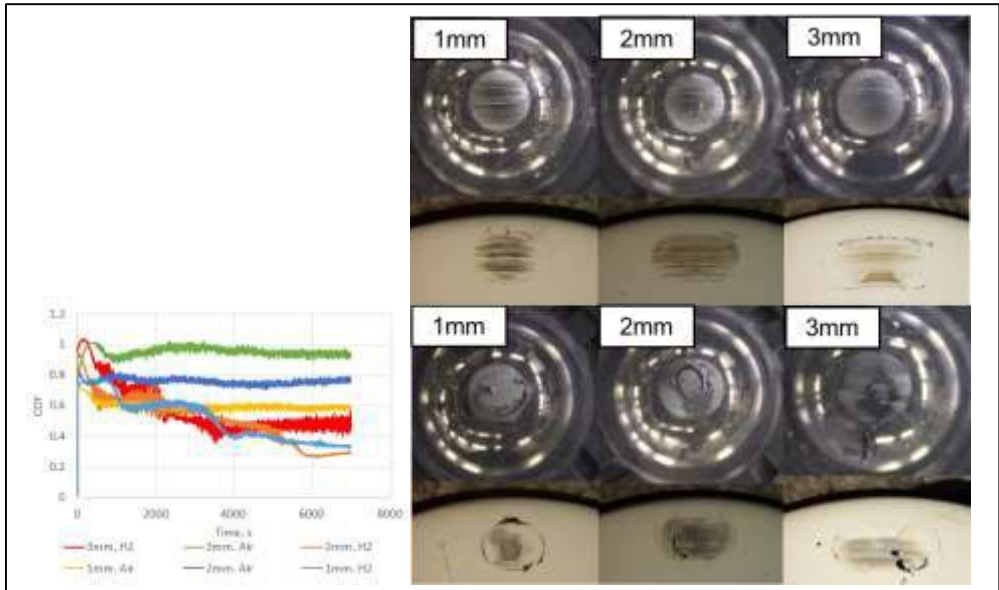


Figure 4: Changes in COF tested with FKM1 and Photos after the sliding test (a) in hydrogen (b) in air

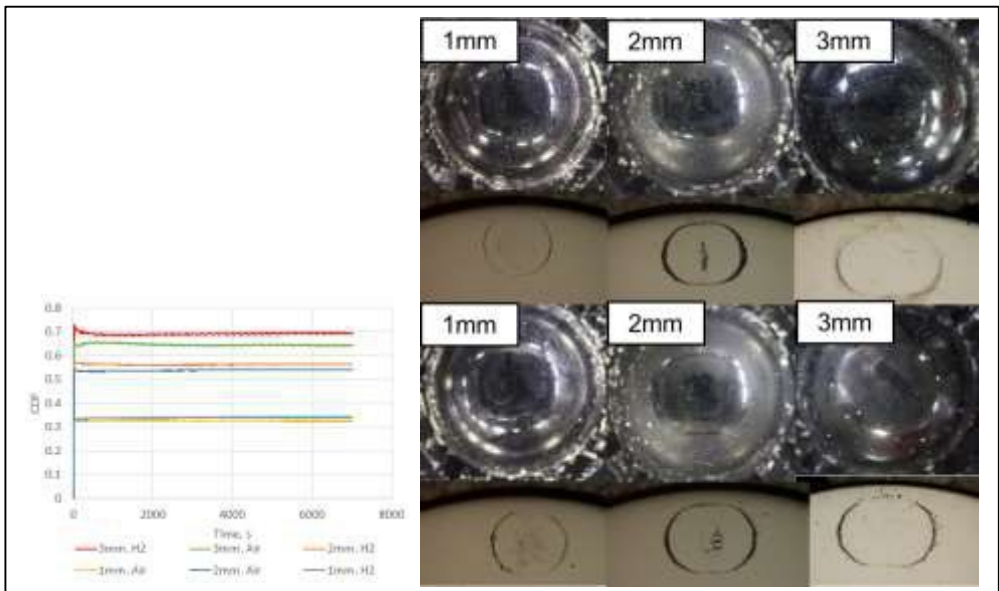


Figure 5: Changes in COF tested with NR and Photos after the sliding test (a) in hydrogen (b) in air

2.4.1 COF and mechanical properties

As the results above, friction and wear properties varied with type of rubbers. Figure 6 showed the changes in COF for each rubber material as a function of reciprocating stroke. Roughly the greater tensile strength exhibited higher COF in reciprocating sliding test. The greater tensile strength maintained the adhesion between the rubber and steel surface even at larger displacements, avoiding local slip and abrasion of rubber surface. Note that FKM1 had the highest hardness and NR the lowest in this series of tests. The softest NR showed elastic behaviour and hardly be worn even with a 3mm stroke. On the other hand, the hardest FKM1 rubber tended to slip and abraded easily in hydrogen. Except for these two cases, the COFs were in the order of tensile strength.

2.4.2 Running in process

When slip started, running-in process also started. Running in process included tearing of rubber, abrasion, transfer of substance and surface film formation. FKM1 had a harder matrix and slip at a small displacement. Slip, both local and entire slip, promoted local wear, which exposed carbon black filler particles. Figure 7 showed the Raman analysis results of rubber surfaces and disk surfaces after the sliding test in hydrogen and air respectively. The Raman results for FKM1 showed the surface film contains a large amount of carbon black in hydrogen, but in the atmosphere, the rubber base resin itself was the main component. The facts suggested that transfer of filler material tended to occur selectively in hydrogen, while transfer of rubber itself tended to occur in air. Initial transfer process as mentioned above drastically changed the following frictional process.

2.4.3 Effects of filler type and content

FKM1 rubber contains a larger amount of carbon black, which tends to transfer to the stainless steel surface and forms a carbon film on the stainless steel surface under hydrogen environment. Amorphous carbon film often reduces friction under hydrogen environment[8]. This may be the reason why FKM1 rubber had lower friction after running-in in hydrogen.

VMQ showed completely opposite trend. A large amount of silica filler was observed on the disk after the test in hydrogen especially with VMQ2, but COF remained high. Detached silica filler was pushed out from the contact area and didn't involve in friction. Raman analysis supported this hypothesis. Silica was hardly observed in any cases on the contact area of the disk surface slide against VMQ1 and 2, whereas silicone rubber base resin itself was observed in both rubber samples tested in air. This meant that silica couldn't absorbed effectively on the stainless surface under hydrogen environment. VMQ rubber also degraded in air, making VMQ's friction in air lower than hydrogen.

As mentioned above, it was found that the friction and wear characteristics were complexly and strongly influenced by the type of rubber, the type of filler, and the environmental conditions.

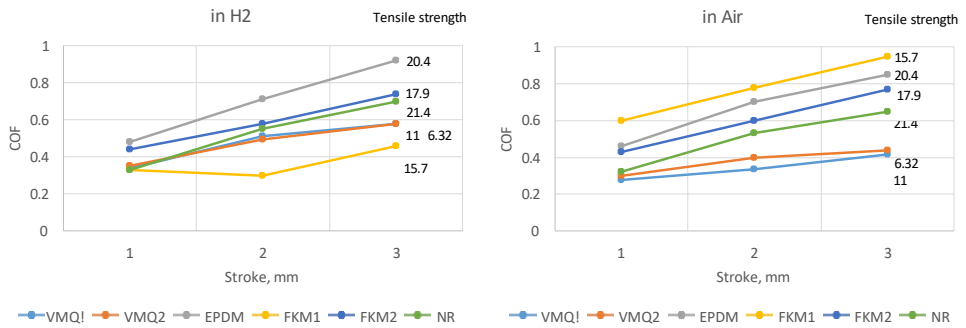


Figure 6: Changes in COF depending on rubber type under different environments

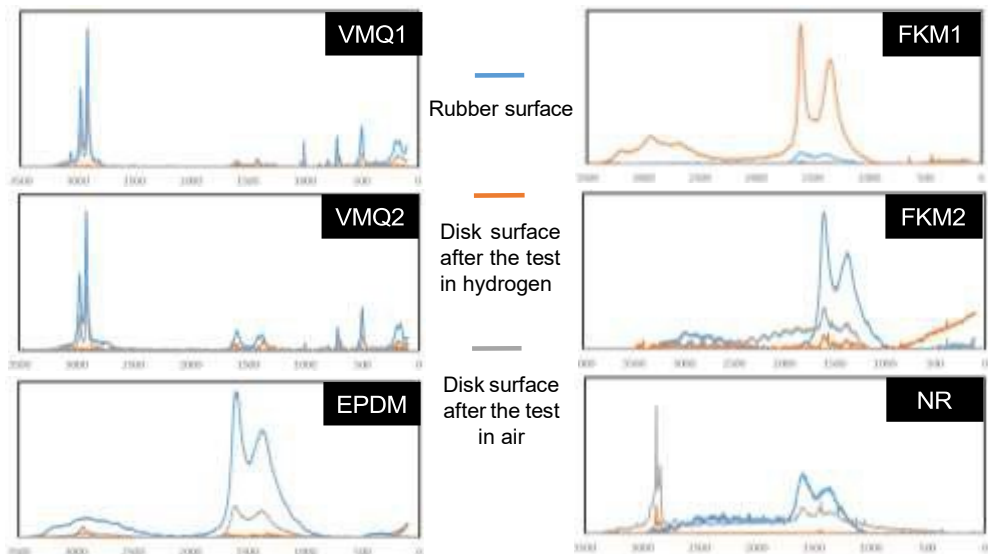


Figure 7: Variety of Raman spectrum for rubber and disk surface after the sliding test

3 Summary and Conclusion

A reciprocating sliding tests were carried out using hemispherical rubber specimen. Friction and wear characteristics were complexly and strongly influenced by the type of rubber, the type of filler, and the environmental conditions. Greater tensile strength of rubber exhibited higher COF in reciprocating sliding test if rubber sample had similar hardness. VMQ showed lower COF than others in hydrogen and air. VMQ showed a sharp drop of COF in air. In contrast, FKM showed a sharp drop of COF in hydrogen.

4 Acknowledgements

This research work was performed within the “Strategic International Collaborate Research Program(SICORP)” (grant number: JPMJSC2121), which was supported by Japan Science and Technology Agency (JST) and Federal Ministry of Education and Research (BMBF)

The authors are grateful to the Bundesanstalt für Materialforschung und -prüfung (BAM), namely Dr. Theiler and Dr. Murillo, to Freudenberg Technology Innovation SE & Co. KG corporate R&D, namely Dr. Dobbelaar, Dr. Schaubert and Mr. Christian for informative discussion.

5 References

- [1] Yamabe, J., Koga, A., Nishimura, S.; Failure behavior of rubber O-ring under cyclic exposure to high-pressure hydrogen gas, *Engineering Failure Analysis*, 2013, <https://doi.org/10.1016/j.engfailanal.2013.01.034>
- [2] Fujiwara, H., Ono, H., Nishimura, S.; Degradation behavior of acrylonitrile butadiene rubber after cyclic high-pressure hydrogen exposure, *International Journal of Hydrogen Energy*, 2015, <http://dx.doi.org/10.1016/j.ijhydene.2014.11.106>
- [3] Kuang, W., Bennett, W. D., Roosendaal, T. J., Arey, B. W., Dohnalkova, A., Petrossian, G., Simmons, K. L.: In situ friction and wear behavior of rubber materials incorporating various fillers and/or a plasticizer in high-pressure hydrogen, *Tribology international*, 2020, <https://doi.org/10.1016/j.triboint.2020.106627>
- [4] Johnson, K. L., Kendall, K. and Roberts, A. D., *Proc. R. Soc. London, Ser. A*, 324 (1971) 301
- [5] Barquins, M. (1985). Sliding friction of rubber and Schallamach waves - A review. *Materials Science and Engineering*, 73(C), 45–63.
- [6] Barquins M, Courtel R. Rubber friction and the rheology of viscoelastic contact. *Wear*, 1975, (32):133-150
- [7] Fukuda, K., Kurono, Y., Izumi, N., Sugimura, J., “Influence of Trace Water and Oxygen in a Hydrogen Environment on Pure Fe Friction and Wear,” *Tribology Online*, 5, 2010, 280-86.
- [8] Sawae, Y., Morita, T., Takeda, K., Onitsuka, S., Kaneuti, J., Yamaguchi, T., Sugimura, J.: Friction and wear of PTFE composites with different filler in high purity hydrogen gas, *Tribology international*, 2021, <https://doi.org/10.1016/j.triboint.2021.106884>

6 Authors

NOK corporation

4-3-1 Tsujidoshinmachi Fujisawa-shi, Kanagawa-ken, 251-0042 Japan:

Hikaru Hashimoto, M. Sc., hashimoto.18.hikaru@jp.nokgrp.com

Suguru Norikyo, M. Sc., norikyo.21.suguru@jp.nokgrp.com

Dr.-Eng. Ayako Aoyagi, aoyagi.07.ayako@jp.nokgrp.com

Kyushu University, Faculty of Engineering, Department of Mechanical Engineering

744 Motooka Nishi-ku, Fukuoka-ken, 819-0395 Japan:

Univ.-Asst. Prof. Dr.-Eng. Hiroyoshi Tanaka, ORCID 0000-0002-2392-4789,
tanaka.hiroyoshi.315@m.kyushu-u.ac.jp

Takehiro Morita, M. Sc.,
morita.takehiro.871@m.kyushu-u.ac.jp

Univ.-Prof. Dr.-Eng. Yoshinori Sawae, ORCID 0000-0001-9255-5297,
sawae.yoshinori.134@m.kyushu-u.ac.jp

Dr.-Eng. Joichi Sugimura, ORCID 0000-0001-0002-0003,
sugimura.joichi.666@m.kyushu-u.ac.jp

<https://doi.org/10.61319/BCWRTC88>
