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Test method

Influencing factors for static immersion tests of compatibility between elastomeric materials and lubricants

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ABSTRACT

Material compatibility is an important factor to consider during the development of new lubricants and sealing materials. Static immersion tests provide a first idea about the compatibility between elastomeric materials and lubricants. For the same material combination, significant deviations among the test results of different laboratories have been reported. In order to identify the relevant factors affecting the results, a systematic investigation was carried out. Reproducibility tests show that the estimation of the compatibility is compromised due to the deviations that appear for the change of the mechanical properties of the considered reference elastomers. The influences of the closure of the test apparatus and the volume ratio, as well as the development of the aging process, were investigated. The results showed that, in order to differentiate between elastomer-lubricant material combinations, standard test durations of approximately 1008 h are preferred. Volume ratios of 64 and 80 and small variations of the vessel closure did not lead to significant deviations of the results. In contrast, tests with open and closed vessels showed significant deviations for the hardness and mechanical properties of the elastomeric materials.

1. Introduction

In order to ensure the correct functioning of machine elements made of elastomeric materials and to reduce maintenance costs, it is important to consider the interactions that occur between elastomeric parts and the surrounding medium in service. More than 500 failure analyses for O-rings show, for example, that approximately 41% of the failure causes can be grouped under the classes Media and Temperature/Aging [1]. Elastomeric materials have a three-dimensional network structure composed of polymer chains, in which degrees of freedom are restricted by crosslinks and chain entanglements. This network structure grants elastomeric materials a high elongation capacity. The properties of elastomeric materials are strongly affected by UV radiation, heat, oxygen and chemicals, which can cause a progressive deterioration of the material with time. There is a distinction between forms of aging in the literature. Physical aging refers to a change of the physical structure of the material e.g. crystallization, stress relaxation, alignment, whereas during chemical aging there is a non-reversible change in the chemical structure e.g. molecule cleavage, increase of the crosslink density. In reality, both forms of aging take place at the same time with effects which are sometimes opposed to each other [8,9].

Regarding the compatibility of elastomeric materials with different fluids, several influencing factors are mentioned in the literature. The polarity is known to influence the swelling behavior. Polar fluids are likely to diffuse into polar elastomers and, similarly, non-polar fluids are likely to diffuse into non-polar elastomers. The molar volume of the fluid and the percentage of bound acrylonitrile in nitrile rubbers (NBR) are also known to have an effect on the swelling behavior. According to Starmer [10], the relation between the volume change of the elastomer after the immersion in a fluid and the percentage of bound acrylonitrile in the elastomer can be approximated with a Gaussian curve. The percentage of bound acrylonitrile for the maximum increase of volume depends on the polarity index of the fluid. Furthermore, acid–base interactions play also an important role [10]. NBR is considered to be a hard base and a soft acid, which is likely to interact with hard acids and soft bases. According to ASTM D 471, the aniline point of an oil is also a determining factor for the swelling behavior. In general it applies that, the lower the aniline point, the stronger the swelling effect of the oil.

Static immersion tests provide a first idea about the compatibility between elastomeric materials and lubricants. In the test, specimens of elastomeric materials are immersed in a recipient

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with lubricant. The specimens remain immersed under controlled conditions of temperature and for a determined duration. Although these screening tests do not reproduce the tribological conditions present in reality, they allow a first assessment of the material compatibility with simple laboratory equipment. The test procedure has been standardized in several specifications. The standard ASTM D 471 [2] has the highest acceptance in America, while in Europe the standard ISO 1817 [3]/ISO 6072 [4] is the most recognized.

Like many other products, lubricants must comply with several tests before they get the approval for a specific market, e.g. DIN 51524: minimum requirements for hydraulic oils [5] and DIN 51517: minimum requirements for lubricating oils [6]. The test of compatibility with elastomers is among the requirements that lubricants must fulfill. Unfortunately, the current standards do not define clearly the test apparatus and test conditions. The specification of a loose closure of the recipient has for example led to different interpretations. Some test laboratories use, for example, aluminum foils, other use ground glass, etc. This has led to the definition of several company-specific standards, which in the end affects lubricant and elastomer producers, who have to fulfill several tests of elastomer compatibility as a consequence. Additionally, the definition of reference elastomeric materials in different standards with the same name convention has led to confusion [7].

In order to identify the relevant influencing factors of the test apparatus and to determine the influence of the test conditions on the test results a systematic investigation was carried out.

2. Methods

In order to test the compatibility of elastomeric materials with different fluids different standards have been developed in the past. The present study was mainly carried out taking the standard ISO 1817/ISO 6072 as reference for the tests. ISO 6072 applies specifically for the test of hydraulic fluids and is based on the more general standard ISO 1817, in which the test procedure is described in detail. A few tests were performed according to ASTM D 471. For this reason, a brief comparison of the standards ISO 6072/1817 and ASTM D 471 is provided in Table 1.

2.1. Test materials

The standard ISO 6072 defines reference elastomeric materials for the tests with hydraulic fluids. It includes specifications for the production of NBR (acrylonitrile butadiene rubber) elastomers with different acrylonitrile content, with different types of crosslink (sulfur, peroxide), as well as for the production of FKM (Fluoroelastomer) elastomers, among other materials. Besides ISO 6072, the standard ISO 13326 [11] also defines reference elastomers, which are quite similar to those defined in ISO 6072. Table 2 shows the reference elastomers, which were tested in this study.

Similarly, reference oils were selected for the tests. Table 3 shows the FVA (Forschungsvereinigung Antriebstechnik e.V.) reference oils, which were tested.

2.2. Test apparatus

Fig. 1 shows the test apparatus used in this study to perform the immersion tests according to ISO 1817/6072. It consists of a glass vessel with ground lid. The glass has a height of 153 mm and a diameter of 85 mm. A support made of stainless steel wire was used to hold the test specimens in place. For a test with 430 ml oil and 5 specimens type 2 (ISO 37) results a volume ratio (Volume of oil relative to the volume of the specimens in the glass) of 64. The volume of air in the closed vessel is approximately 78.3 ml.

2.3. Determination of the elastomer compatibility index

The Elastomer Compatibility Index (ECI) is a measure of how strong the elastomeric material is affected after the immersion. It is defined in ISO 6072 as the change of the properties hardness, volume, tensile strength and elongation at break of the elastomeric test specimen after the immersion test. The change of hardness was measured with a micro-IRHD hardness tester. The change in volume was determined based on the mass of the specimen in air, distilled water and in fresh test oil. The measurements were done with an analytical balance with 1 mg readability. In order to determine the tensile strength and elongation at break, tensile tests according to ISO 37 were performed using a tensile testing machine MTS Criterion Model C42.503 with a 1 kN load cell (class 0.5 1–100%) equipped with an extensometer with a resolution of 0.004 mm.

3. Results and discussion

Before considering the variation of parameters of the test, the reproducibility of the results with the test apparatus described in 2.2 and the test equipment described in 2.3 was estimated. For this purpose, immersion tests were repeated up to six times with the same materials and test conditions.

The deviations for the change of hardness and change of volume shown in Fig. 2 are acceptable in comparison to common limit values of compatibility (+/− 8 IRHD, −4%–15% Volume change after 168 h [12]).

Considering again common limit values of compatibility as a reference (maximum tensile strength change of −20% and maximum elongation change of −20% after 168 h [12]), Fig. 3 shows a significant dispersion for the change of the tensile strength and for the change of elongation at break. In the case of the change of elongation at break, the deviation of the test results is comparable to the limit value of −20% according to the guideline provided in the earlier ISO 6072 2002 [12] (The guideline no longer appears in

Table 1
Comparison of the test standards ISO 6072/ISO 1817 and ASTM D 471.

	ASTM D 471	ISO 6072/ISO 1817
Reference elastomers	Not defined	NBR1, NBR 2, FKM 2, EPDM 1, HNBR 1
Reference oils	IRM 901, IRM 902, IRM 903, IRM 905	IRM 901, IRM 902, IRM 903
Test vessel	Glass test tube, L: 300 mm × D: 38 mm	Not defined
Volume of the test fluid	100 ml (Volume change), 150 ml (Hardness change, change of tensile strength and elongation at break)	The volume of the test fluid must be at least 15 times the volume of the test specimens in the test vessel
Form of the test specimens	Rectangular 25 × 50 × 2 mm (Volume change), Dumbbell specimens type C according to ASTM D 412	Dumbbell specimens type 2 according to ISO 37, circular specimens, rectangular specimens