

How to Evaluate the Compatibility of Thermoplastic Materials with Lubricants

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ABSTRACT

Over the past few years the use of thermoplastic materials has been broadened considerably in many different parts of automotive components, such as door handles, dashboards, interior trim, seat covers, gearshift linkages and gearboxes. This paper highlights the importance of compatibility issues of polymers coming into contact with lubricants. In order to enable a proper function of car components, advanced elaborated testing should be conducted. Using the example of compatibility tests, this paper illustrates how the “lubricant” design element contributes to improving the function of polymers. Moreover, relevant test rigs and results are described and discussed.

Thermoplastic materials and lubricants are essential for the construction of many technical applications. Therefore, it is very important to consider the compatibility of both components. This however raises the question of how to evaluate the compatibility. Contrary to rubber materials, there is no general standard like the ISO 1817 for thermoplastic materials [1]. Furthermore, it is difficult to transfer test results from lab tests to technical applications. Currently some OEM and tier 1 suppliers for the automotive industry are formulating standards to define acceptable tests and give certain ranges for the testing. These standards are generally focusing on tensile tests and sometimes on environmental stress cracking (ESC). This article aims to show some different methods for the immersion procedure and their limits. Materials like PA 66, POM, PP and PC will be discussed. Influences caused by additives and solvents will also be considered.

INTRODUCTION

Is my polymer compatible with your lubricant? In recent years this question has gained a very high priority. But what

is “compatible”? There is no easy answer. Information about certain conditions such as temperature, mechanical stress or humidity is needed in order to give more qualified answers. Furthermore, it is important to use an appropriate test rig in order to evaluate proper values and to obtain reliable results. As far as rubber materials are concerned, the ISO 1817 standard gives a very good basis for the testing procedure simplifying the comparison of the test results. For thermoplastic materials, a comparable standard is not available. Standards like ISO 527, ASTM D 638, ISO 22088, ASTM D 543 and ISO 175 only describe several techniques, but they give no general guidance for the complete procedure of testing polymers with lubricants [1,2,3,4,5,6]. The manner in which the immersion is performed has a significant influence on the test results. This situation was basically the motivation for this investigation. In this paper, the main focus was put on the influence of different testing conditions, on the mechanical properties after exposure of thermoplastic materials to the grease. At the focus of interest were tensile tests with polyacetal (POM), polyamide (PA), and environmental stress cracking (ESC) tests with polyacetal and polycarbonate (PC). A short discussion of swelling experiments was given for polypropylene (PP).

EXPERIMENTAL

The tensile experiments were performed on a Zwick Roell Z 020 material testing machine with a range up to 20 kN. The load sensor has a sensitivity class of 0,5 starting from 200 N. The elongation sensor has a measurement range of 50 mm with a resolution of 0,2 µm. The deviation was evaluated with a minimum of 3 tensile experiments. Tensile experiments were performed with polyacetal Delrin 100 by DuPont and polyamide PA 66 Ultramid A3K by BASF. The tensile bars were obtained from the polymer manufacturer, like DuPont and BASF, and were produced by injection molding. The test patterns for the environmental stress cracking (ESC) test according to the bent strip method (ISO

22088-3) were manufactured in-house from stainless steel tubes with different defined radiuses. ESC tests were carried out with Ultraform W 2320 003 (BASF), Hostaform C 52021 and Hostaform C 9021 (both Ticona), Delrin 100 and Makrolon 2805 (Bayer Materialsience). The base oils and additives were obtained from different suppliers in a technical grade. The swelling experiments, discussing the PAO mixtures, were conducted with plates of Celstran PP GF 3005 (Ticona) with a shape of about 40 mm × 40 mm × 3 mm. Most of the experiments were conducted at 100°C over a time of 1000 h and more. Many applications based on the thermoplastic materials, mentioned in this article have a service temperature between 60 and 120°C. Thus 100°C is a well balanced compromise between strong and moderate aging.

METHODS AND RESULTS

TENSILE EXPERIMENTS

For thermoplastic materials, the tensile experiment itself is well defined in the standards ISO 527 and ASTM D 638. The immersion process itself is not considered. Of course it is possible to orient oneself by the standards ISO 1817 for rubber or ISO 175, but this approach is not harmonized at all. In order to consider different aspects, two methods for the immersion experiments were tested. Method A puts the focus on the direct interaction of grease with polymer under exclusion of atmospheric oxygen, due to a thick grease protecting layer of about 30 mm to 60 mm. Method B includes also a thermo-oxidative aspect, due to a thin grease layer of about 3 mm to 6 mm ([figure 1](#)).

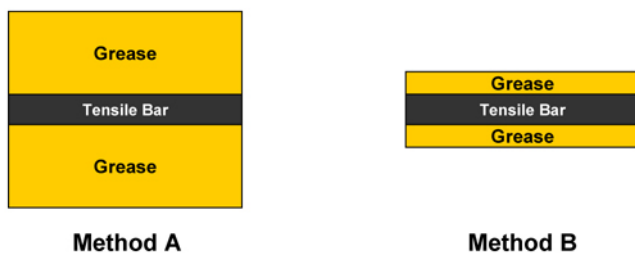


Figure 1. In method A, the tensile bars are completely covered with a large amount of lubricant. The surface in method A is covered with approximately 30 mm to 60 mm of grease, while in method B the tensile bars are only covered with about 3 mm to 6 mm of grease.

The grease is applied to the tensile bars, and after the immersion at 100°C and 1000h the tensile tests were performed with a speed of 50 mm/min, which is a common velocity for unreinforced thermoplastic materials. The focus was put on two different parameters. The nominal tensile strain at break (ϵ_B) and the tensile strength (σ_M) were mainly considered for the evaluation of the influence of the different immersion methods. The [Figures 2](#) and [3](#) give an overview of

the results obtained with nine different grease formulations, considering the nominal tensile strain at break (ϵ_B) for Ultramid A3K and Delrin 100. [Table 1](#) presents the basic information regarding the grease compositions. [Figure 4](#) and [5](#) present the results, taking into account the tensile strength (σ_M).

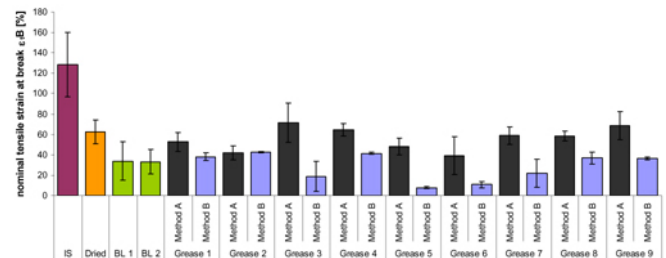


Figure 2. Results of the nominal tensile strain at break (ϵ_B) after immersion according to method A and B with nine different lubricants and Ultramid A3K (PA 66) at 100°C for 1000h. (IS: Initial Sample, Dried, BL 1-2: Blank Test)

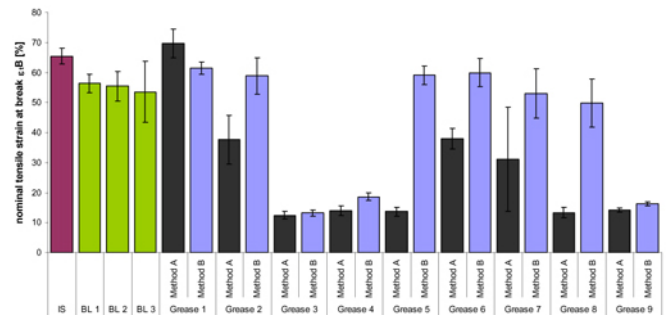


Figure 3. Results of the nominal tensile strain at break (ϵ_B) after immersion according to method A and B with nine different lubricants and Delrin 100 (H-POM) at 100°C for 1000h. (IS: Initial Sample, BL 1-3: Blank Test)

Table 1. Formulation of lubricants. (Li: Lithium, Ba: Barium, HC: Hydrocarbon)

Grease 1	Synth. HC Oil Li-Thickener A	Grease 4	Polyglycol Oil Li-Thickener B	Grease 7	Polyglycol Oil Li-Thickener B
Grease 2	Synth. HC Oil Ba-Thickener	Grease 5	Synth. HC Oil Li-Thickener C	Grease 8	Synth. HC Oil Mineraloil Urea-Thickener
Grease 3	Synth. HC Oil Mineraloil Li-Thickener B	Grease 6	Synth. HC Oil Li-Thickener C	Grease 9	Synth. HC Oil Li-Thickener C

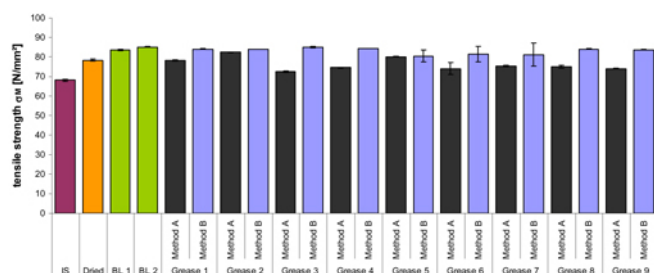


Figure 4. Results of the tensile strength (σ_M) after immersion according to method A and B with nine different lubricants and Ultramid A3K (PA 66) at 100°C for 1000h. (IS: Initial Sample, Dried, BL 1-2: Blank Test)

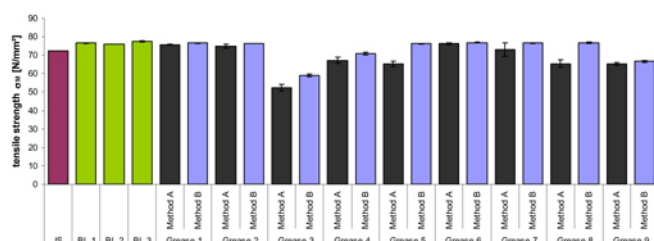


Figure 5. Results of the tensile strength (σ_M) after immersion according to method A and B with nine different lubricants and Delrin 100 (H-POM) at 100°C for 1000h. (IS: Initial Sample, BL 1-3: Blank Test)

<table 1 here>

Discussion of the results with Ultramid A3K

The high value in the initial sample for the nominal tensile strain at break (ϵ_B) can easily be explained by the remaining water content. Some polyamides can incorporate up to 9 % of water. The water softens the material significantly, so that high values are obtained. The dried sample was stored for 3 h at 120°C. During this procedure incorporated water evaporates. A reduction of the nominal tensile strain at break is the result. Two blank tests have also been investigated and were compared to the grease exposed samples. The increase of the tensile strength and the strong reduction of the nominal tensile strain at break indicates that, besides a further

reduction of humidity, a thermo-oxidative ageing occurred. This effect is visible in the color change from white to dark brown, Figure 6. Figure 7 gives a rough approach to the side reactions related to the ageing of polyamides [7, 8]. These side reactions may give an idea of how the embrittlement of the PA happens. To what degree this cross linking mechanism is influenced by lubricants is at the focus of further investigations. Some first indicators have been found how additives of lubricants can accelerate this embrittlement. The embrittlement can only be observed on the surface of the tensile bars. The inner part of the material is usually not harmed very much [9, 10]. In case of the tensile test, the force applied to the test bar is continuously increasing, but the test bar is not able to elongate, due to the cross linked surface. When the force is so strong that the surface suddenly cracks, the faultless inner part also cracks immediately, because of the sudden force peak. Comprising the results of the tensile strength (σ_M), for method B, a slight increase for most of the values was observed. This is an additional indicator that the cross linking of the polyamide surface leads to stiffer performance. For deeper investigation of the embrittlement, of course a test specimen of less than 4 mm should be favored. The standard test specimen for tensile tests according to ISO 527 has a thickness of 4 mm. Corresponding results with aged polyamide 66 has been reported by Eriksson et al [10]. A re-crystallization of the materials also leads to a higher stiffness, but generally spoken, this process is of minor priority. It is remarkable, that among all the PA tests, primarily method B is more aggressive to the PA. Thermo-oxidative ageing is the dominating effect. The greases 3, 5, 6 and 7, may also have additional effects under the conditions prevalent at method B. The other greases show almost equal values for B as seen in the blank tests. When using the method A, the results of the nominal tensile strain at break are almost in the same region as for the blank tests. Thus indicating that only thermal ageing occurs and that the grease prevents the polyamide from oxidization. This can also be observed when considering the tensile strength. All the tests according to method B have almost the same values or even higher as do the blank tests. The values for method A are slightly lower. The PA was not particularly harmed by the grease environment. Figure 8 gives a sketched assumption for the different test methods. Kinetic considerations would be