How Tightly Sealed are Connectors Really?

Measurement Method for the Exact Leakage Determination of Polymer-Metal Hybrid Parts

The interfacial tightness of hybrid polymer-metal parts such as injection-molded connectors represents a decisive quality characteristic. Until now, however, there has been a lack of suitable measurement technology with which to quantify the media tightness of such molded parts. A new method delivers significantly more accurate results than previous test methods.

The DDP method enables more accurate determination of the tightness of polymer-metal hybrids

Every day, high volumes of hybrid polymer-metal parts, e.g. in the form of connectors, are produced worldwide using injection molding. In this process, inserts of electrically conductive metal are overmolded with polymeric material which, after solidification, acts on the one hand as electrical insulation and on the other as protection against environmental influences.

The contact area between polymer and metal represents an interface that must exhibit good adhesion between the joining partners in order to be able to ensure interfacial tightness. A critical problem with hybrid polymer-metal parts is their possible leakage at the interface, which can cause migration of media or gases through the interface. For this reason, many applications of polymer-metal hybrids require a high degree of media tightness at the interface.

However, a metrological verification of the interfacial tightness is difficult to achieve. Up to now, a hybrid system has been described as „tight" if the leakage rate measured on the test specimen is below a previously defined, permissible limit value. The leakage rate describes the possible media transfer, i.e. the flow of gaseous or liquid media, through a hybrid part.

Limits of the Differential Pressure Leakage Method

However, the definitive permissible leakage rate is often not known for many applications. This is partly due to the lack of knowledge of a minimum achievable leakage rate for the hybrid system in question. Consequently, a suitable measuring method is needed to determine the possible media leakage rate of various material combinations and corresponding manufacturing conditions during the injection molding of hybrid parts. A corresponding measurement method was therefore developed at Aalen University, Germany.

The differential pressure leakage method is usually used to detect leaks in technical molded parts, whereby the disadvantage of this method is the limited resolution in the range of 10^{-3} mbar · l · s^{-1} under standard conditions (1013.25 mbar at 0 °C). Therefore, for the investigations presented in the following, a test gas method with a metrologically higher resolution was used instead to determine the interfacial leakage. The helium vacuum leakage measurement was used. The company Dr. Wiesner Steuerungstechnik GmbH in Remshalden, Germany, designed and manufactured a special helium leakage measuring station for the tests, which allows extremely low leakage rates of up to 10^{-10} mbar · l · s^{-1} to be detected.

One problem with measuring possible interfacial leakage with a test gas method is that the measured value can be falsified due to simultaneous permeation of the test gas through the measuring chamber seal and the test specimen itself, as well as existing leakage flow along the contact area between the measuring chamber seal and the test specimen. Potential weak points for unanticipated gas permeation include the polymeric part of the hybrid specimen to be measured, as well as any seals and sealing contact areas necessary to create a pressurized chamber filled with the »

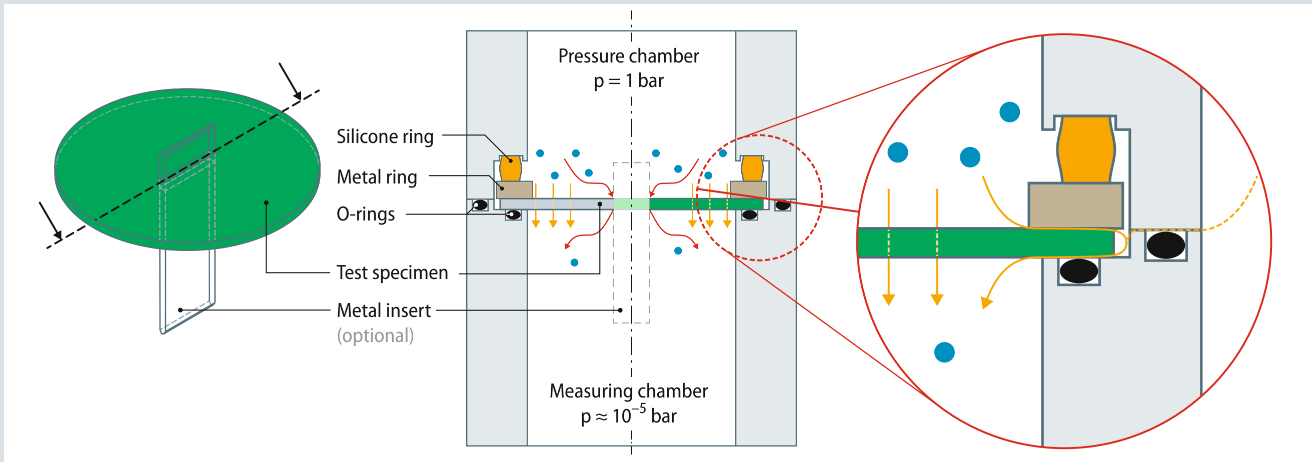

Fig 1. Left: three-dimensional view of a test specimen with metal insert; right: test specimen located between pressure and measuring chamber, arrows show possible permeation paths (orange) and interfacial leakage paths (red) Source: Aalen University; graphic: © Hanser

test gas and a vacuumed measurement chamber during this procedure. When comparing the performance of polymer-metal combinations, for example, this can lead to a material combination being declared unsuitable in terms of its interfacial tightness, although it is only the higher permeability of the polymeric material that is responsible for the comparatively higher leakage rate.

The New DTP Method

For this reason, an approach was developed and tested that allows the permeation effects and the interfacial leakage rate to be considered separately: the so-called Differential Tightness and Permeation measurement (DTP) method. The test method requires a hybrid circular disc specimen with an overmolded passing through metal insert as well as a monolithic polymeric specimen with same shape and dimensions. Both test specimens were examined for their leakage rate using the test gas method.

The actual interfacial leakage rate is equal to that of the hybrid polymer-metal specimen minus that of the monolithic polymeric specimen. The following equation is therefore obtained:

$$\dot{q}_{interface} = \dot{q}_{hybrid} - \dot{q}_{permeation}$$

The requirements for a low measurement uncertainty are equal measurement parameters (pressure and temperature) as well as the same measuring chamber seals throughout a measurement series.

For the present studies, disc-shaped test specimens with a diameter of 30 mm and a wall thickness of 1.2 mm, both with and without metal inserts, were manufactured and analyzed. The outer dimensions of the metal inserts were 25 mm x 8 mm x 0.8 mm.

An O-ring made of nitrile butadiene rubber (NBR) with a Shore A hardness of 72 was used as the measuring chamber seal, with an inner diameter of 22 mm and a cord thickness of 2.5 mm.

Another O-ring made of the same material with an inner diameter of 39 mm and a cord thickness of 3 mm was used to seal the chambers against the environment.

Setup for the Comparison of the Measurement Methods

For helium vacuum leakage measurements, the test specimen is placed between the measuring and pressure chambers and pressed against the measuring chamber seal (**Fig. 1**). To ensure complete filling with the test gas, the pressure chamber is initially evacuated and subsequently filled with helium at a pressure of 1 bar (±50 mbar). The measurement chamber is connected to an ASM340 mass spectrometer from Pfeiffer Vacuum, which measures the helium leakage rate of the tested specimen. All measurements were carried out at room temperature (23 °C ± 2 °C).

A measuring device of the type Integra DD6 from Dr. Wiesner Steuerungstech-

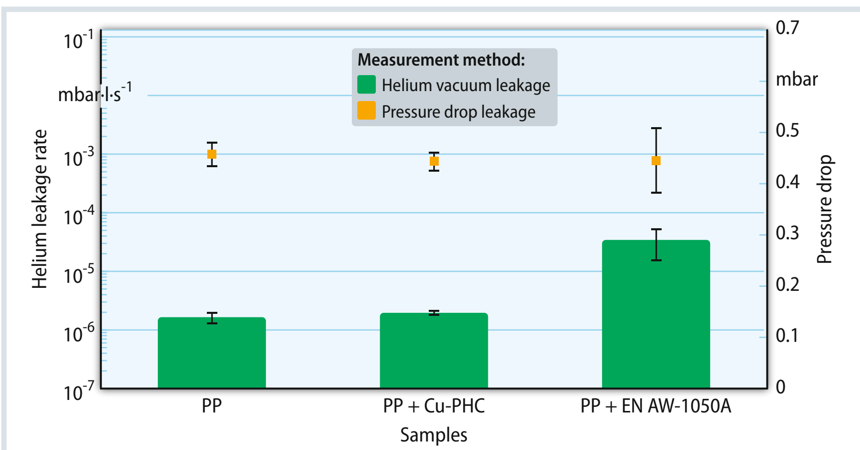

Fig. 2. Leakage measurement results using different measuring methods on the same samples at 23 °C (averages from five measurements each): in contrast to the differential pressure method, clear differences in the leakage rate can be seen with the helium vacuum test

Source: Aalen University; graphic: © Hanser

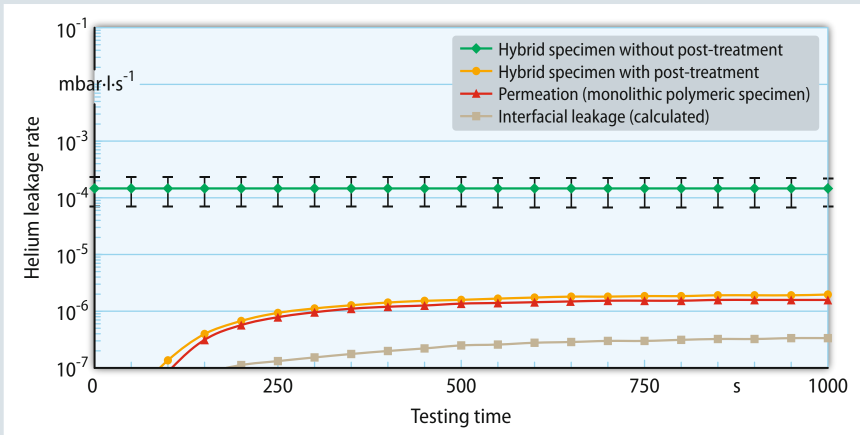

Fig. 3. Differentiation between permeation and interfacial leakage determined by using DTPM on thermally post-treated, adhesion-modified polymer-metal hybrids at 23 °C: thermal post-treatment of the hybrid parts clearly influences the leakage rate as well as its development over time
Source: Aalen University; graphic: © Hanser

nik was used for the differential pressure measurements at room temperature. In the differential pressure method, the test specimen is as well placed between the measuring chamber and the pressure chamber, whereby the latter, together with a sealed reference volume, is subjected to an overpressure of 1 bar for a period of 10 s. After a settling time of 10 s, the relative pressure drop with respect to the reference volume is measured.

For the present investigations, a commercially available, natural-colored homopolymer polypropylene (PP) with a melt flow rate of 12 g/10 min was used to produce both the test specimens with and without metal inserts.

In order to improve the adhesion between the polymeric material and metal, approximately 0.2 wt. % maleic anhydride (MAH) was added to the neat PP in the form of a PP grafted with MAH as an adhesion promoter (PP + PP-g-MAH). The metal inserts were punched from rolled sheets and then cleaned and degreased with propan-2-ol in an ultrasonic bath. The inlay materials were pure aluminum (EN AW-1050A) and deoxidized copper with low residual phosphorus content (Cu-PHC). Both metals are characterized by very good electrical conductivity and are therefore preferred as conductor materials. The test specimens were injection-molded in a single-cavity mold on an Arburg 220 S Allrounder 150-30 injection molding machine with a screw diameter of 15 mm (manufacturing conditions: **Table 1**).

The injection mold was made by Gindele GmbH, Neuhausen, Germany, and, via rotatable mold inserts, enables the production of disk-shaped test specimens with single- or double-sided gates as well as with and without metal inserts. Some of the test specimens with metal inserts were subjected to a targeted post-treatment with heat to potentially improve the interfacial adhesion between polymer and metal.

Parameter	Unit	Value
Melt temperature	°C	245
Mold temperature	°C	100
Injection velocity	cm^3/s	30
Holding pressure	bar	400

Table 1. Injection molding conditions for the production of the investigated test specimens Source: Aalen University

Only One Method Shows Clear Differences

Considering the standard deviation of the measurement results, the differential pressure method does not show any differences between the test specimens investigated with regard to their media tightness. The results of the helium vacuum test, on the other hand, show clear differences between the PP-Cu and PP-Al material combinations (**Fig. 2**). The injection-molded combination of PP with aluminum (EN AW-1050A) exhibits a leakage rate that is more than a decade higher than the comparative sample of copper and PP. Accordingly, the differential pressure method with its limited resolution is not suitable for a precise determination of the media tightness of hybrid parts.

The injection-molded PP-Cu hybrids exhibit a relatively high leakage rate, which is practically constant over the measurement period (**Fig. 3**). Already at the beginning of the measurement, a high leakage value of $1.5 \cdot 10^{-4}$ mbar · l · s^{-1} is measured. Since the leakage value remains constant over the measurement time, the gas passage of the helium occurs without a prior dissolution and diffusion process of the test gas. This indicates leakage at the interface of the hybrid part. In contrast, after conducting the post-heat treatment on the hybrid sample, the helium leakage rate curve starts at a much lower level and now resembles a permeation curve. A typical permeation curve shows a vanishingly small leakage rate at the beginning of the measurement, which increases sharply after some time as a result of gas breakthrough and shows a constant level over time after the gas concentration equilibrium has been reached. The post-treatment obviously creates a material barrier in the interface between copper and the modified PP, which prevents spontaneous gas breakthrough at the beginning of the leakage measurement.

The permeation curve (measurement curve) of the pure PP sample is slightly lower than that of the post- »

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- Read the German version of the article in our magazine ***Kunststoffe*** or at ***www.kunststoffe.de*** treated hybrid specimen and indicates the permeation behavior of the examined PP for helium gas at a polymer part wall thickness of 1.2 mm. The interfacial leakage rate curve is determined according to the above equation. Post-treatment of the injection-molded hybrid specimens reduces its interfacial leakage by almost three decades to the range of 10^{-7} mbar·l·s^{-1}.

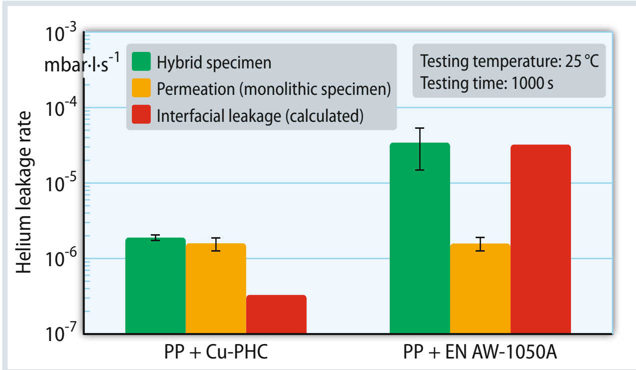

Fig. 4. Interfacial leakage rates minus permeation values of different polymer-metal hybrids: the PP-copper specimens show significantly lower leakage than specimens of PP and aluminum
Source: Aalen University; graphic: © Hanser

Post-Treatment of Hybrid Parts Improves the Interfacial Tightness

Figure 4 shows the values of the interfacial leakage of PP-Cu and a PP-Al hybrid specimens measured by using the DTP method. Both hybrid combinations were thermally post-treated and subsequently exhibited permeation characteristics during the leakage measurement. The measured interfacial leakage rate of the PP-Cu samples is $3 \cdot 10^{-7}$ mbar·l·s^{-1}, which corresponds to a leak size of about 3 µm in diameter. Leaks of this size are declared to be „virus-tight." In comparison, the measured interfacial leakage of the PP-Al hybrid specimens is higher by about two decades with more than 10^{-5} mbar·l·s^{-1}.

The presented test results show that the media tightness of polymer-metal hybrids can be measured much more sensitively using helium vacuum leakage measurements compared to applying differential pressure testing. Using the DTP method, interfacial leakage between polymeric material and metal can be precisely determined. With the disc-shaped test specimen presented and by using the DTP method, it is possible for the first time to quantitatively test various polymer-metal material combinations specifically for their adhesion properties at the interface.

Thereby, the injection molding manufacturing conditions, any pretreatments carried out on the metal insert, and the effect of a possible post-treatment on a manufactured hybrid part can all be considered.

The developed method also provides information on the permeation behavior of a particularly investigated polymer for a given, process-related morphology. ■

PPA with Carbon Fiber-Reinforcement

Lightweight Metal Replacement

BASF expanded its polyphthalamide (PPA) portfolio of Ultramid Advanced with carbon fiber-reinforced grades with fillings of 20, 30 and 40 % . They make for extremely lightweight parts, can safely replace aluminum and magnesium without loss in stiffness and strength and are electrically conductive.

The new grades show high dimensional stability due to low water uptake, excellent chemical and hydrolysis resistance, high strength and modulus. They can be used to manufacture automotive structural parts for body, chassis and powertrain, for pumps, fans, gears and compressors in industrial applications as well as for stable and ultra-lightweight components in consumer electronics.

The mechanical performance of the new PPA grades can be tuned by the choice and the content of the carbon fiber as well as by the additive technology. Ultramid Advanced N3HC8 with 40 % carbon fiber filling shows a better strength and modulus at 80 °C (conditioned) than magnesium or aluminum.

The materials reportedly can contribute to functional integration and weight reduction in different industries: the range of cars with e-drive or fuel cell engines can be increased by weight reduction of structural or powertrain parts; lightweight, thin precision structures in consumer electronics benefit from the high stiffness and strength, the excellent dimensional stability as well as the extremely low weight and the good processability of the new PPA materials; heavy, highly loaded and long-lasting industrial equipment like pumps and compressors can be easily produced because of the good dimensional stability as well as the high chemical, heat and abrasion resistance of the new CF grades.

The carbon fiber-reinforced PPA compounds also show a lower weight and higher tensile modulus than glass fiber-reinforced polyamides (PA) with similar reinforcements. PPA grades reinforced with 20 wt.% carbon fibers are about 20 wt.% lighter than PA6 or PA66 filled with 50 % glass fibers. According to the company, the tensile strength of a 20 % carbon fiber-reinforced Ultramid Advanced compound is either better or equivalent to a glass fiber-reinforced polyamide filled with 50 % while showing better processability.

The carbon fiber- reinforced PPA e.g. can replace aluminum and magnesium © BASF

To the product presentation:
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