

Methods for Polymer Diagnostics for the Automotive Industry*

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The further development from polymer testing to polymer diagnostics leads to test methods with higher amount of information per test procedure as conventional tests. These changes contribute to fulfill the increased requirements in polymer testing and diagnostics. Selected examples of practice of industrial polymer testing show that these new methods can be applied appropriately to solve the issues of the automotive industry.

The automotive industry is one of the most important forces of the international economic system. The usage of materials is both diverse and innovative. A good example is the Opel RAKe which was presented at the 65th International Motor Show "IAA" in Frankfurt/Main in 2011. It is a tandem-two-seater with a 36.5 kW/49 hp electric engine that provides acceleration to 100 km/h (62 mph) in 13 seconds. One reason for the moderate price of 12,000 € (\approx \$ 15,700) is a comeback of cheaper materials: the floor assembly is made of steel and the chassis is made of polymer material [1]. Contrary to the trend of the recent years which contributed to the reduction of CO₂-emissions by using expensive materials like carbon fiber and aluminum components, nowadays the new price consciousness of the customers is met. This example shows how fast-paced the construction solutions in the automotive industry are and how flexible and agile the suppliers, that are established in the service sector around them, have to be. Belonging to this service sector in the broader sense is the "material testing trade" which accompanies the usage of new, substituted and modified materials conventional as well as further developed with cost effective methods of testing and diagnosing. The following examples show methods of polymer testing and polymer diagnostics with its application on characterizing polymer molding materials, semi-finished products as well as car body parts.

Methods of Polymer Diagnostics

The traditional idea of polymer testing includes methods that are used to determine material parameters, the differentiation of polymer material and also to provide a simple pre-selection and quality assurance which all are based on counting and measuring operations. A new generation of polymer testing procedures that are characterized by its high sensitivity and accuracy and/or physical active principles, that are new or differentiated, are summarized under the term of polymer diagnostics. These methods of diagnostics enable the identification of structure related parameters which are used for the analysis of components and materials, the defect analysis and the structure oriented material selection. Coupled mechanical structure sensitive and non-destructing methods for the analysis of micro-mechanical deformation and damaging processes particularly apply [2].

If conventional methods of mechanical polymer testing like the tensile test, the flexural test or the ball indentation test are extended with the help of electronic sensors that register and regulate the testing process and thus provide a larger amount of material information, then the transition to polymer diagnostics has been fulfilled.

Table 1 shows examples of already established methods of analytical and mechanical polymer diagnostics [3-13].

The headline "Analytical Polymer Diagnostics" abstracts the methods of thermo-analytical, spectroscopic, diffraction, and

microscopic methods. These techniques allow an analytical view of the structure and the morphology of polymeric material.

The section "Mechanical Polymer Diagnostics" consists of two parts: instrumented methods and hybrid methods. The instrumented methods, i. e. instrumented (registered) hardness test [3], instrumented Charpy impact test [5], instrumented tensile test [8], and instrumented impact test, contain testing technologies which realized the (outline above) larger amount of material information per testing procedure, because of the electronic registering of the testing event. The hybrid methods allow a multitude of combination possibilities of interlink non-destructive and conventional methods like tensile or flexure test. An exceptional position takes the micro test technology for example the in-situ peel test in the ESEM [13].

These technologies enable an online observation of mechanical experiments based on miniaturization of specimens. Especially by a small quantity of specimen, as in the rule of material development, the application of this method leads to desired results [13, 14].

Instrumented Hardness Testing

The hardness measurement is one of the most used material testing technique, because it is easily practicable, nearly non-destructive and there is a relationship between hardness values and other mechanical values for example the strength [6]. The

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Analytical polymer diagnostics	
Thermoanalytical methods	Dilatometric Analysis
	Differential Scanning Calorimetry (DSC)
	Thermo Gravimetric Analysis (TGA)
	Thermo Mechanical Analysis (TMA)
	Dynamic Mechanical Thermal Analysis (DMTA)
Thermo-optical Analysis (TOA)	
Spectroscopic methods	Infrared Spectroscopy
	Nuclear Magnetic Resonance
Diffraction methods	X-ray Diffraction Analysis
	Electron Diffraction
	Neutron Diffraction
	Light Scattering
Microscopic methods	Scanning Electron Microscopy (SEM)
	Environmental Scanning Electron Microscopy (ESEM)
	Transmission Electron Microscopy (TEM)
	Microanalysis of SEM and TEM
	Atomic Force Microscopy (AFM)
Instrumented methods	<p>Mechanical polymer testing</p> <ul style="list-style-type: none"> instrumented (registered) hardness test [3] nano, micro and macro hardness testing with temperature [4] instrumented Charpy impact test (ICIT) [5, 6, 9, 10] instrumented tensile test (ITT) [8, 11, 12] instrumented free falling dart test (IFDT) [8, 12]
Hybrid methods [6, 7] (in-situ methods)	<p>Mechanical testing/non-destructive testing (NDT)</p> <ul style="list-style-type: none"> tensile and bending laser extensometry [3, 16] video thermography Ultrasound (US)/Acoustic Emission Analysis (AEA) mechano-dielectrometry SEM/TEM <p>Fracture mechanics testing/non-destructive testing (NDT)</p> <ul style="list-style-type: none"> tensile and bending static fracture mechanics + AEA + video thermography static fracture mechanics (R-curve) + video thermography (e.g. for elastomers) static fracture mechanics (in-situ R-curve) + microscopy (REM) ICIT + AEA (laser extensometry) <p>Micro test technology</p> <ul style="list-style-type: none"> micro-tensile test + field measurement techniques (grey value correlation techniques, moiré techniques, laser interferometry) static fracture mechanics + AFM in-situ tensile testing in ESEM + AEA in-situ peel test in ESEM [13]

Table 1. Methods of polymer diagnostics (selection) [3-13, 16]

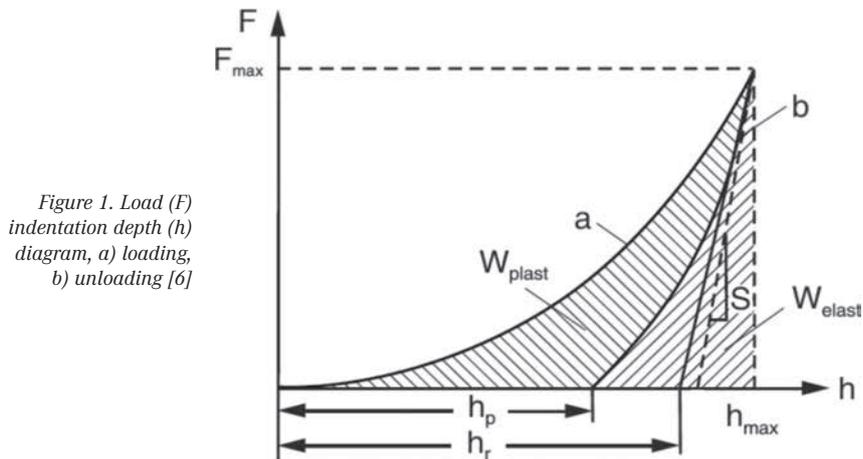


Figure 1. Load (F) indentation depth (h) diagram, a) loading, b) unloading [6]

different conventional hardness testing techniques for the different material groups are almost all based on the principle to estimate the ratio between force and amount of impression. The use of the instrumented hardness test as a method of polymer diagnostic leads to increase the sum of information per testing procedure [3, 4].

In the instrumented hardness test the registration of the depth of penetration and the force is performed during the penetration process (cf. Figure 1). This experimental extension allows statements about the amount of elastic deformation W_{elast} and the amount of plastic deformation W_{plast} of the investigated materials. The diagram shows also the maximal indentation depth h_{max} under the maximum load F_{max} , the elastic reset indentation depth h_r , and the plastic indentation depth h_p . The slope of the tangent to the discharge line makes it possible to identify the indentation modulus S .

In the present example the use of instrumented hardness test was performed to optimize the parameters for the welding of polymer compounds of polyethylene [3]. Such welds are mainly used in pipes, but another example for the application of this technology is car rear lights [10].

Figure 2 shows the elastic indentation modulus which was determined at different technological conditions across a weld joint. It can be seen that the heat affected zone and the actual weld seam are very sensitive to a variation of welding parameters such as welding temperature and welding pressure. Only at optimal parameters the entire area of the weld seam is characterized by a high indentation modulus S . In case of high weld pressure or low weld temperature the result of welding process is not satisfying. The indentation modulus S is too low or it reaches non-constant level at the heat affected zone. So the use of the method of instrumented hardness testing leads to improve the quality of welded polyethylene automotive construction components.

Instrumented Impact Test

Construction components are frequently loaded by dynamic forces, therefore accords the toughness characterization with the Charpy impact test prime technical importance. However, there are a few drawbacks, which can lead to misinterpretation: at the Charpy impact test, the toughness will be evaluated as an integral value. There is no information about the compo-

nents of strength and displacement, and there is no information about the crack propagation behaviour. In cases of “non break” for example of high impact modified polymer blends there are no possibilities to differentiate the materials respective to their toughness behaviour [5].

During the instrumented Charpy impact test (ICIT), the process is recorded by a force and a displacement sensor (Figure 3) and then plotted in a impact-force-deflection diagram. As a result this test has a significantly higher information content compared to the conventional Charpy Impact Test.

The registered force-deflection diagrams can be analyzed and fracture mechanics values can be determined [15]. The fracture mechanics values often show a sensitive reaction to morphological changes and so these values are predestined for application in material development and improvement of formulations.

The use of impact modified polypropylene/ethylene propylene rubber (PP/EPR) blends, for example as bumper compounds, is beneficial especially at low operating temperatures. The implementation of conventional Charpy impact tests in this group of material frequently results in the finding of “non break”, so that a differentiation is only possible by the application of ICIT. Figure 4 show the fracture mechanics toughness values J_{I_d} as a function of the modifier content of the three different modifiers [9]. The value J_{I_d} represents an energy determined parameter and describes the resistance to unstable crack growth. The modifier was added to ethylene propylene rubber (EPR) and two different types of polyethylene (PE 1 and PE 2). It can be observed that the modifier EPR shows the strongest toughness enhancing effect. The J_{I_d} values of the PP/EPR material with additional 20 wt.-% of modifier increase more than the double value. The TEM images show the characteristic phase morphology for PP/EPR without and with 20 wt.-% EPR modifier

system. The material with the additional EPR modifier system exhibits smaller and fine distributed particles.

Coupling of Tensile Test with Laser Extensometry

As shown in Table 1, the hybrid methods which couple mechanical testing with non destructive testing to get in-situ answers of the tested material, is an important part of the polymer diagnostic.

The application of such hybrid techniques is a good possibility obtaining structurally and physically well-based quantitative morphology property correlations. For application of non destructive defectoscopy, component monitoring, effective material optimization as well as damage analysis, these structure property correlations allow an adequate interpretation and evaluation of the material behaviour in a more essential way.

The present example shown in Figures 5 and 6 demonstrate the coupling of me-

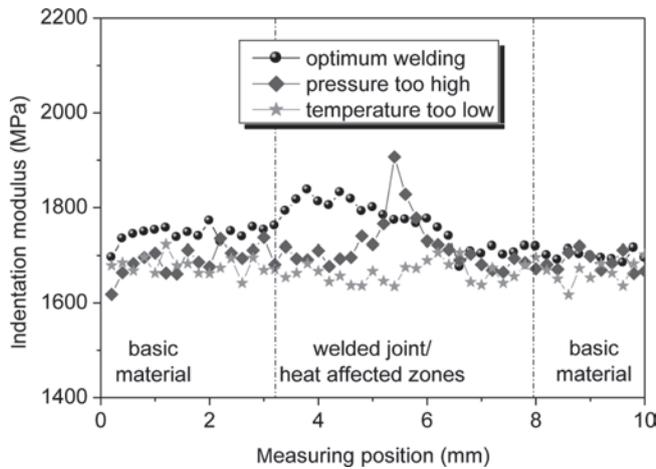


Figure 2. Indentation modulus of different welded joints [18]

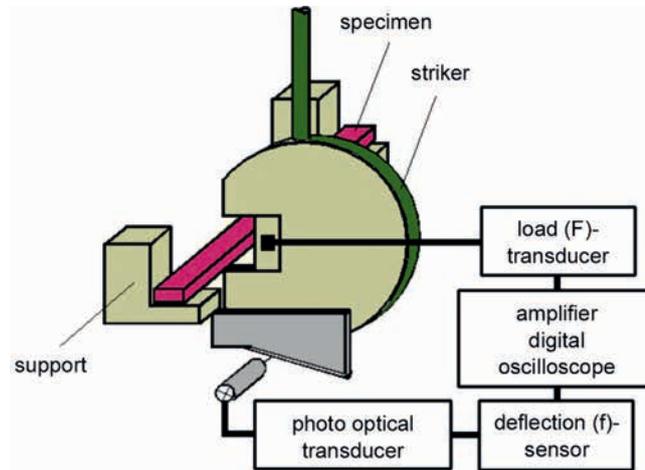


Figure 3. Schematic representation of the instrumented Charpy impact test (ICIT) with load transducer and deflection sensor to record load-deflection diagrams

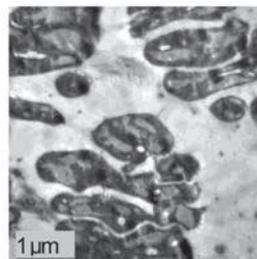
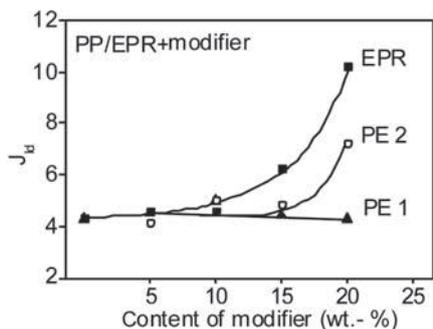
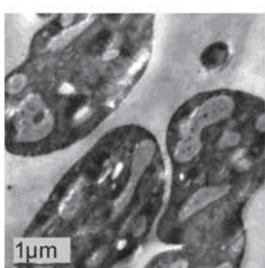


Figure 4. J_{I_d} values of bumper compounds and TEM pictures of PP/EPR without modifier (left) and PP/EPR with 20 wt.-% EPR-modifier (right) [9]

Figure 5. Schematic illustration of laser extensometry and a recorded diagram to characterize the local deformation behaviour

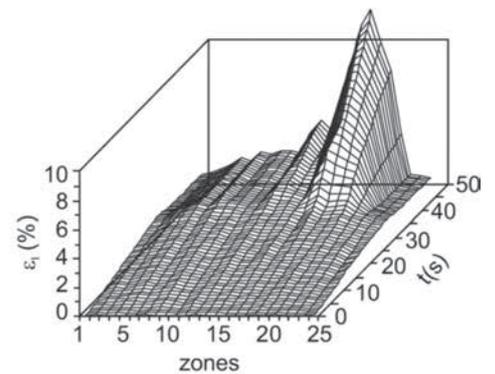
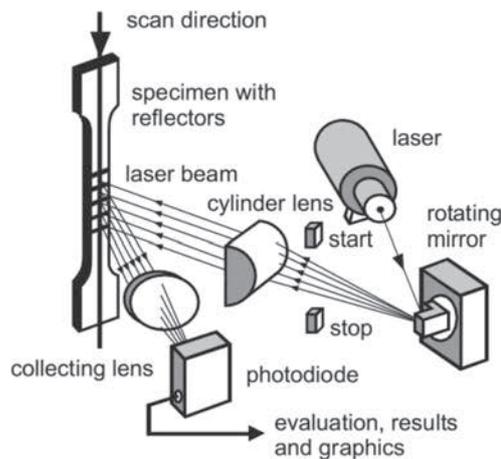
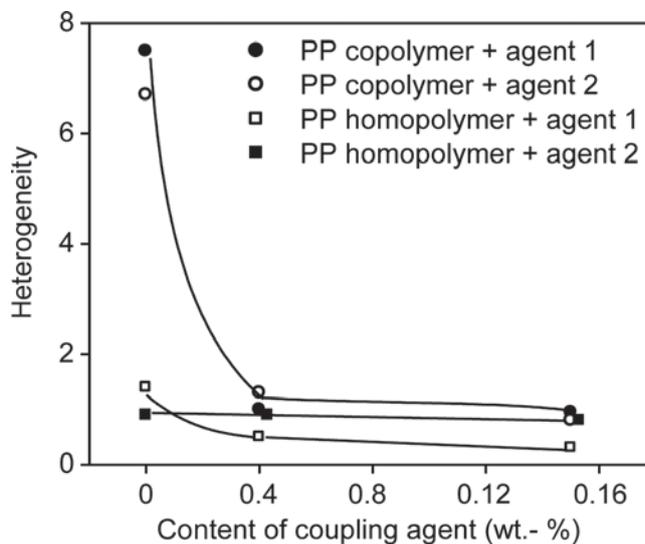


Figure 6. Influence of coupling agent on the heterogeneity of different polypropylene materials



chanical tensile test with laser extensometry [6, 7, 16]. During the tensile test the application of the position sensitive stretch measuring technology of the laser extensometry enables the registration of local deformation. The determination of characteristic values of the tensile test, for example the parameter of integral strain, the elongation at break ϵ_B , is also possible.

The presentation of the local elongation at the recorded diagram in Figure 5 shows that the area around the 20th reflector is characterized by raising elongation by local yielding. This heterogeneous material behaviour can be described by a material parameter, the heterogeneity H [17].

Figure 6 shows the results of the application of this hybrid method of polymer diagnostics on various Polypropylene glass fiber (PP/GF) composites. The heterogeneity is representing the dependence on the amount of two different cou-

pling agents. The PP/GF composites with an homopolymer matrix show a constant level of heterogeneity. Especially for PP/GF composites the use of coupling agents with copolymer matrix leads to a substantial reduction in heterogeneity. This way, stretching peaks through local flow (cf. Figure 5) can be excluded and the performance of the material can be improved. Those materials are used for instance in the automotive industry as compounds for instrument panels.

Coupling of Static Fracture Toughness Tests with Videography

The coupling of static fracture toughness tests with videography allows the evaluation of fracture toughness behaviour as resistance to stable crack initiation and crack propagation. The improvement of the resistance to crack initiation is one of the

aims of tire material development. The magic triangle of this intension consists of the components "wet slip behaviour", "roll resistance" and "abrasive wear/crack initiation". While increasing the resistance of tire materials to crack initiation and propagation, the life time of these rubber products can be enhanced.

The static fracture mechanic test was carrying out on single edge notched tension (SENT) specimen, notched with a razor blade. The notch tip was especially prepared with white powder. This was the requirement of application of the videography.

The loading parameter of the crack resistance curve (R-curve) J is enabled by the recording of force-extension process at the fracture mechanics experiment. The deformation and crack opening process is monitored by videography. Through the observation of the prepared notch tip, physical crack initiation values of the crack initiation can be determined. Figure 7 shows some photographic pictures which show the crack opening process in dependence of time. At instant of time zero, the crack is already closed. Until 75 seconds the prepared notch tip is white, no new surface was generated. The following photographic pictures exhibit the crack opening with a growing black area at the notch tip.

Figure 7 shows already the R-curves as the results of the investigations of different carbon black filled elastomeric materials. The amount of carbon black filler was between 0-50 parts per hundred rubbers (phr). With increasing carbon black content there is an increase on resistance to stable crack propagation. Knowledge of these mechanisms is mainly used for the evaluation of the abrasion behaviour of tire materials [4, 11]. It has to be noticed that

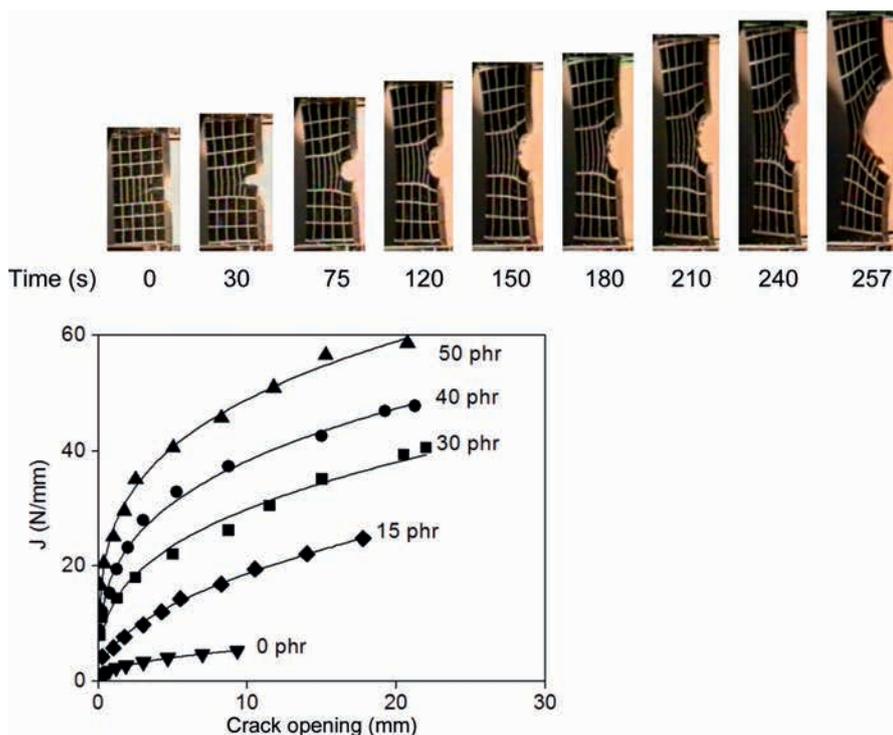


Figure 7. Results of coupling quasi-static fracture mechanic test with videography, above: videographic pictures of the crack opening process, below: crack resistance curve [6, 11]

the diminishment of the abrasion is not only a target of optimization of tire materials, it is also a contribution of environmental protection.

Summary

The multifaceted applications of polymer components in automotive engineering require a new generation of testing techniques and methods. The polymer diagnostics are modern methods and characterize by its high sensitivity and accuracy and/or physical active principles. They lead to a higher amount of information per test procedure. The presented examples are applications of the methods of mechanical and fracture mechanical polymer diagnostics to characterize polymer molding materials, semi-finished products as well as car body parts for the automotive industry.

Registering hardness testing, instrumented Charpy impact test and the hybrid methods of coupling the tensile test with laser extensometry as well as coupling the static fracture toughness test with videography are methods of mechanical polymer diagnostics which are used to investigate mechanisms of fracturing and deformation. These new techniques show how the fur-

ther development of polymer testing to polymer diagnostics is of purposeful use for handling key issues in the automotive industry.

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Abstract

Verfahren zur Polymerdiagnose für die Automobilindustrie. Die weitere Entwicklung der Polymerprüfung zur Polymerdiagnose führt zu Prüfverfahren mit einem größeren Informationsgehalt pro Prüfung als bei konventionellen Versuchen. Diese Veränderungen tragen dazu bei, den steigenden Anforderungen an die Polymerprüfung und -diagnose Rechnung zu tragen. Ausgewählte Praxisbeispiele aus der industriellen Polymerprüfung zeigen, dass diese neuen Verfahren geeignet sind und angewendet werden können, um Fragen aus der Automobilindustrie klären zu können.

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