

Damage detection and monitoring of glued elements in civil structures using adhesive layer integrated markers

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Today adhesive technology is of particular importance in different industrial sectors, for example in automotive industry or in aerospace technology. In building industry adhesive technology is often used to avoid punctual transmissions of power. Glued glass elements in constructional engineering are only one example of application. However, the adhesive joint is subject to an ageing process affecting the quality and the durability of the adhesive joint. Therefore an appropriate monitoring is essential in respect of adhesive joints. For these reasons this paper describes first results of an adhesive based joining technology enabling an early detection of failure of the adhesive joint which is under development. This method is based upon the fact that the adhesive used is formulated with additives indicating any situation of the defined load limit being exceeded. By now two different additives can be used for this method: olfactory additives, which can be detected by scenting on the one hand and dyestuffs, which can be optically detected on the other hand. These additives are inserted into the adhesive by encapsulation. In case of a damaged adhesive joint the capsule will be destroyed and the additives will be released. The released additives can be detected with adequate measurement systems. The sensors for the detection of olfactory additives consist of a metal doped metal oxide. Different scents of the additives produce a change in resistance at the sensor. Experiments with such a sensor system and the mentioned adhesives have shown that damages of the adhesive joint result in significant decrease in resistance. Therefore the change in resistance is used as a measure for the damage of adhesive joints and makes early detection possible.

1 INTRODUCTION AND STATE OF THE ART

1.1 *Adhesive technology in the building industry*

The use of adhesive technology in the building industry has become increasingly popular over the last years. For example, adhesive joints are particularly used in bridge constructions and buildings with glass. There has been a high demand for glass-constructions in the last years as they make clear and light-flooded constructions possible. For joining glass elements the adhesive technology is used ever more often due to its many advantages. Unlike other joining technologies as staking and screwing, the adhesive technology allows for a laminar transmission of power and avoids notch sensitivity. By reason of the high brittleness of glass this is of great benefit, Luhn et al. (2008).

There are different manners of using adhesives for joining glass elements. In the first case, the adhesive is only used to hold the self-weight of the glass element. In this case silicon adhesives will mostly be used, Hagl, 2008. Secondly, various applications are mentioned in the literature, where glass is used as a construction material. In this case glass elements serve as a reinforcement of the entire building. High-tensile adhesives such as acrylates are used in these applications. Furthermore constructions exclusively consisting of glass elements are conceivable enabling an even higher transparency of the buildings, Tasche (2008), Weller et al. (2006), Werner (2004), Blandini, L. (2007).

These applications prove that glass as a material as well as gluing as a joining technology for glass have gained great importance in the building and construction industry. The further development of the adhesive technology in the building industry is inhibited by the absence of a technology for monitoring the conditions of adhesive bondings. On these grounds, only certain classes of adhesives are accredited in structural engineering due to stricter safety regulations, Weller et al. (2007). With the relevance of safety and the building regulations gaining more and more importance, an elementary, fast and cost-efficient monitoring technology will be required.

1.2 Non-destructive testing of adhesive joints

In adhesive technology, destructive as well as non-destructive testing methods are in use. The drawbacks of destructive testing include the facts that the sample is destroyed and that no time-dependent behavior of ageing is recognizable. Established groups of non-destructive testing methods include ultrasonic based testing methods and thermography. Examples for ultrasonic testing are the fokker-bond-tester and the pulse resonance method. Great importance is given to the ultrasonic based thermography where the component is charged by ultrasound, generating a different oscillation behaviour for porosities, air pockets and delaminations. These oscillations at the defective locations produce heat, which can be detected by a thermography camera at the surface of the tested parts, Hasenberg et al. (2005).

All these testing methods are cost-intensive and not appropriate for mobile applications. Moreover the use of these methods is not too appealing because of the complex evaluation processes, Hasenberg et al. (2005). Another drawback is that these methods are only able to identify defects already evolved. An early detection before a defect becomes visible is not possible.

1.3 Industrial applications of microencapsulation

In the literature several applications of microencapsulation technology can be found. One of the oldest and most established operational areas is the screw locking with microencapsulated adhesives. The thread of the screw is coated with a microencapsulated adhesive. When screwing in, the microcapsules will be damaged and the adhesive or one component of the adhesive will be released, Endlich (1987), Bauer (1985). Moreover microencapsulation is used in the manufacturing of adhesives. One component of the two-component-adhesive is formulated microencapsulated in the other. When the adhesive is applied, the microcapsules will be destroyed and the adhesive will cure. Benefits are an easier storage and no failures regarding the mixing ratio, Endlich (1985).

Microencapsulated dyestuffs and scents have already been mentioned in the literature. For example microencapsulated scents are being developed that can be sprayed onto car seats and that will smell like leather. When sitting down, the microcapsules will be destroyed, simulating a continuous smell of a new car, N.N. (2003).

Furthermore microencapsulation is already used to detect defects in elements. For the visualization of defects by means of an impact load fiber, reinforced plastics are applied with a coating including a microencapsulated dyestuff showing certain strengths of impact in terms of color, Light et al. (1988). Moreover a gaseous medium can be used to detect failures, hence some applications cannot be controlled visually, Ummenhofer (2005).

Further applications for microencapsulation can be found in the food and pharmaceutical industries to decelerate the release of aroma or active substances, N.N. (1999), N.N. (2002).

1.4 Manufacturing processes of microcapsules

Microcapsules can be manufactured by means of various processes. They can be divided into chemical and mechanical-physical methods. In case of chemical methods the microencapsulation is carried out during the liquid phase, whereas with mechanical-physical methods the microencapsulation takes place in the gaseous state. Basic chemical methods are coacervation and interfacial polymerization; important mechanical processes are spray drying and fluidized bed process, Sliwka (1975).

In the coacervation process the substance to be microencapsulated is dispersed into a colloidal solution. A change of temperature, ph-value or ionic strength results in a precipitation of the capsule material which accumulates at the substance to be encapsulated and forms a membranous coating, Sliwka (1975), Kunz et al. (2003).

By interfacial polymerization two polymer solutions are mixed by reacting with each other, leading to the inclusion of the material to be encapsulated into one solution. The polymerization reaction takes place at the interface of the two immiscible phases, thus generating the capsule wall, Kunz et al. (2003), Kirk-Othmer, Mollet et al. (2000).

With spray drying an emulsion is gasified in a hot, inert gas flow. The hydrophilic capsule material is dissolved in an adequate solvent. The material to be encapsulated is typically hydrophobic and is emulsified in the aqueous phase. Evaporation of the volatile parts leads to the development of a polymer covering, Sliwka (1975), Kunz et al. (2003), Römpf (2006).

The main part of the fluidized bed process is to bring the material to be encapsulated into a fluidized condition by a hot or cold air flow. The capsule material is dissolved and sprayed onto the fluidized bed so that the material gets encapsulated, Kunz et al. (2003), Brötz (1952).

1.5 Sensor systems for the detection of olfactory additives

An adequate electronic measurement system is needed to detect the scents released due to the formation of cracks. This system should operate the way the human nose does. The main elements of the human nose are receptors in large numbers located at the olfactory cells. These receptors interact with the gas molecules in a specific way activating several biochemical reactions and generating a nerves signal, Daniels (2002).

In electronic measurement systems sensors similar to the receptors of the human nose are used. In many cases metal oxide sensors will be applied, but oscillating crystal sensors and sensors based on conductive polymers are also used, Heinig (1998).

The metal oxide sensors are chemo sensor arrays with a layer of conductive, semiconductor metal oxide. These metal oxides reversibly change their conductivity and their resistance subject to the configuration of the gas sample. The signals of the sensors generate a specific model, making it possible to detect changes, for example the release of scents. In many cases stannic oxide is used as metal oxide, but also zinc oxide or tungsten oxide are conceivable. In contrast

to the human olfactory detection the electronic measurement is regulated by a number of sensors. The kind of metal oxide and its doping, the geometry of the sensors and the temperature affect the sensitivity in relation to different gases. The electronic nose consists of significantly less receptors than the human nose. They form a summation signal consisting of all smells existing in the ambient air. Therefore it can only detect a change in smelling, which results in a change in resistance. A special training is necessary to calibrate the sensors on the desired gases, Heinig (1998), Koroncz (2009).

With sensors based on conductive polymers a change in conductivity of the polymer can be measured by a change of configuration of the gas. This kind of sensor reacts very sensitively to changes in air humidity which is a big disadvantage, Daniels (2002), Heinig (1998).

Oscillating crystal sensors are usually integrated into an electric oscillating circuit. A polymer coating adsorbs and absorbs molecules of the gas and thereby changes its mass. This leads to a measurable change in self-resonant frequency, Daniels (2002), Heinig (1998), Röck et al. (2006).

2 EXPERIMENTS

First of all experiments have been carried out to ensure that microcapsules are capable of detecting defects in adhesive bonds. For these experiments dyestuffs and scents were used. The dyestuff microcapsules used comprise the dyestuff xanthophyll. It has a yellow-orange color and is often used in the food and pharmaceutical industries. The capsules have a diameter of about 900 – 1120 μm . Differential scanning calorimetry (DSC) can demonstrate that the dyestuff capsules are resistant against UV radiation and thermal influences (figure 1).

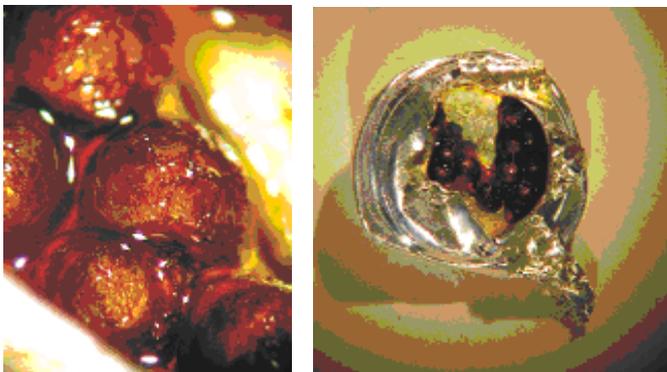


Figure 1. Microscopical image of dyestuff capsules and opened DSC-cup

Using these dyestuff capsules different experiments have been carried out. The dyestuff capsules were mixed with various adhesive systems. A polyurethane adhesive, an UV cross linking acrylate and a two-component acrylate were applied. With the mixture of adhesive and capsules pure substance samples according to DIN EN ISO 527-2 were formed. Yellow dyestuffs coming out of the capsules already become visible during the mixing of the polyurethane with the capsules. It has probably been the amines of the polyurethane that dissolved the capsule material.

The pure substance samples have been investigated with tensile tests. In samples using the highly brittle two component acrylate, a release of the dyestuff can already be noticed at low loadings. Samples consisting of the UV cross linking acrylate have shown an escaping of the

dyestuff with slightly higher loadings due to which this process is easy to follow, as this acrylate is colorless (figure 2).

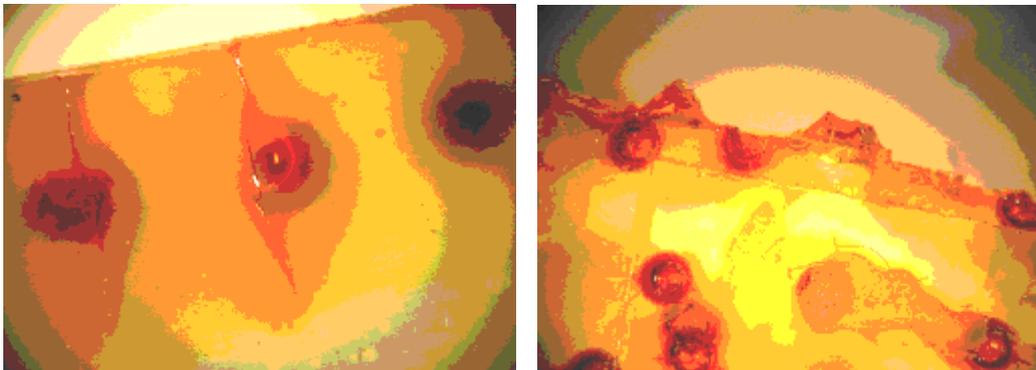


Figure 2. Dyestuff release using 2 component acrylate and UV-acrylate

The experiments have shown that the dyestuff capsules have been destroyed as expected as soon as cracks were formed in the sample.

Further experiments have been carried out with commercialized olfactory capsules with a diameter of about 70 μm . They include a scent which smells like spring flowers. Fourier transform infrared experiments have shown that the molecule consists of a nonpolar hydrocarbon rest and a polar part which include carbonyl and hydroxyl groups. These measurements were also done in connection with a change of temperature to detect a change in chemical composition. No alteration was found.

In the same way as in the experiments with dyestuff capsules the olfactory capsules were mixed with various adhesives and pure substance samples were formed. The samples were tested in a tensile testing machine. During the experiment no smelling could be detected with the human nose. It was supposed that the concentration of released scent had been too low. However, after the fracture of the sample an intensive smelling could be noticed.

In the next step experiments have been carried out using an electronic measurement system to detect released scents. For these experiments tensile shear samples according to DIN EN 1496 with different parameters were prepared. A brittle two-component epoxy resin adhesive and an elastic one-component polyurethane adhesive were used. They were mixed with the olfactory capsules in different concentrations within the range of zero to five percent. A contact-free mixing system was used for mixing. The adhesives were applied in two different adhesive layer thicknesses. On the one hand 250-300 μm is set with glass beads and on the other hand 700 μm with shims. All samples were cured at room temperature. The epoxy resin adhesive was cured 24 hours, the polyurethane 60 hours.

For the experiments with the electronic nose it is important to minimize influences of the ambient air during the tensile shear testing. For this reason the tensile shear samples are protected by a synthetic flexible tube. Figure 3 shows the experimental setup and the measurement system used.

For measurements the intake hose of the measurement system is positioned directly on the adhesive layer. A change of the ambient air, in this case the release of scents can be verified by

measuring the resistance. When applying strengths in the tensile test machine an explicit decrease in resistance at the sensor can be measured.

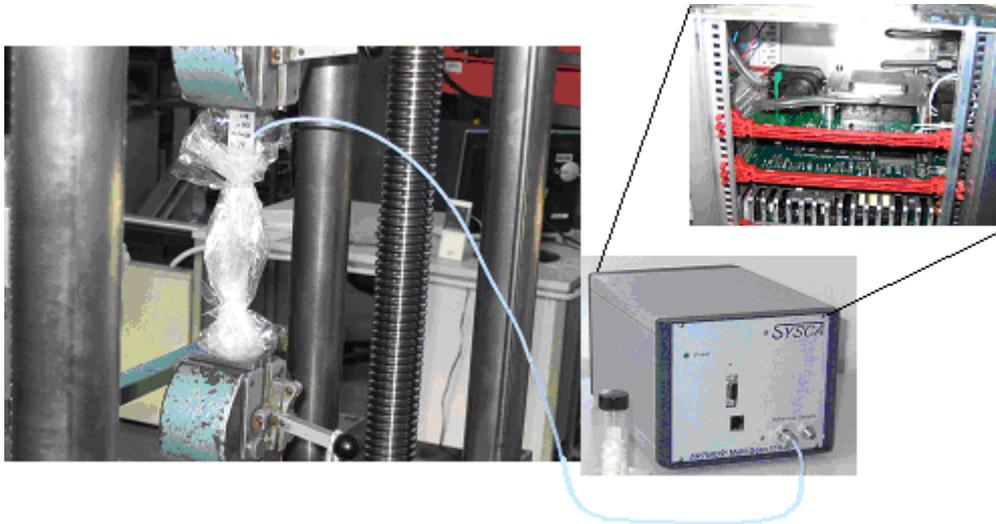


Figure 3. Experimental setup

To improve sensitivity the pump is not actuated continuously but the intake was done in cycles of 15-seconds. Therefore the rarefaction of air decreased and a stronger change in resistance could be measured. Moreover the sensor temperature was raised from 250 degree Celsius to 300 degree Celsius resulting in an acceleration of the oxidation reaction. This in turn results in signal amplification.

A problem is to test whether a change in resistance is initiated by the released scents or by the smell of the adhesive. Therefore one sample with zero percent scent and one with five percent were tested in comparison. Both samples showed a decrease in resistance, but in case of the five percent scent the decrease was more significant as shown in figure 4, where the average of the 38 sensor elements is shown.

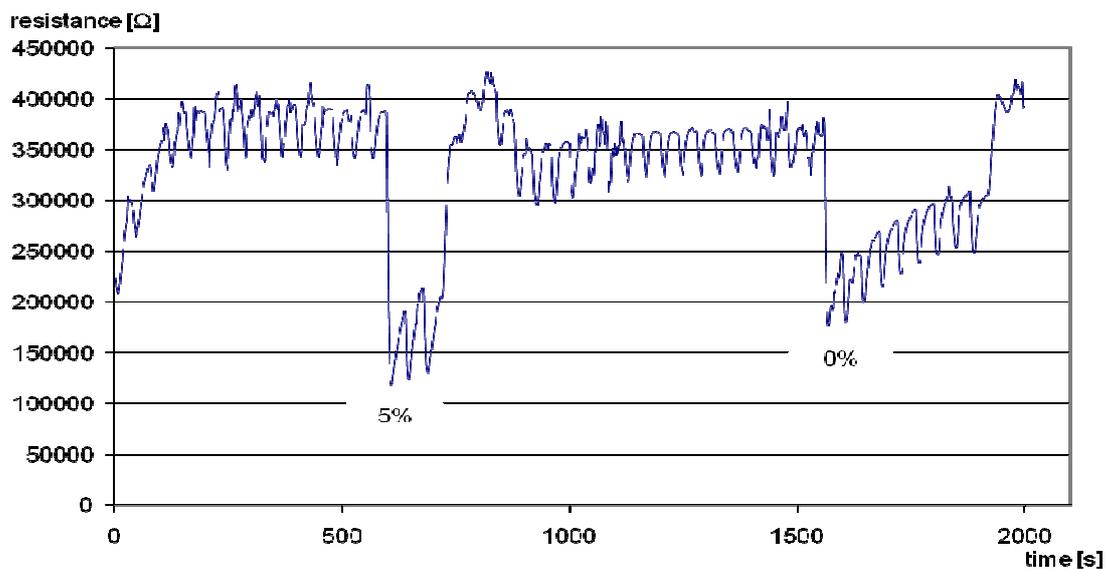


Figure 4. Average of change in resistance from the 38 sensors

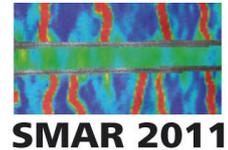
3 CONCLUSION

The experiments show that the idea of a new technology using dyestuffs and scents for the early detection of defects has excellent chances of success. Regarding the dyestuffs it can be concluded that early detection is possible, especially by using transparent adhesives. Particularly for joining transparent glass elements in combination with a colorless adhesive it seems to be a good method to monitor the ageing of the adhesive bond. In further proceedings it will be necessary to investigate the interaction between adhesives and the capsule material, as this is a problem that occurred in the experiments.

Good results can also be achieved with olfactory capsules. The release of scents from the capsules directly results in a change in resistance making immediate detection of defects possible. One problem of this method is to get a significant result. Therefore the next step will be to train the electronic nose to exclude influences of the ambient air and of the adhesive itself.

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