DEVELOPMENT OF A NEW GENERATION OF EMBEDDED SENSORS FOR MONITORING STRUCTURAL COMPOSITES

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Abstract
The demand of smart composite materials is increasing in all sectors. With it, the need of new sensor technologies for structural health monitoring purposes is also increasing. The requirements for the next generation of sensing devices are quite high. Ideally, they should be able to sense different physical parameters, in-situ, on a (sub-)micrometer scale and without affecting the mechanical performance of the host component. For this purpose, multiple technological solutions can be employed. This paper presents three sensing solutions investigated by our research groups. The first solution consists of polymer photonic sensors with micrometer thickness capable to measure both uni- and multi-axial strain using Bragg reflection laws. The second solution focuses on mechanolumiscent materials, which are able to emit visible light when subjected to mechanical load. The third solution regards thin film sensors obtained by direct DC magnetron sputter deposition. Preliminary experimental results obtained with the three sensor solutions are presented and discussed.

1. Introduction

Damage mechanisms in fiber reinforced composites differ from the typical damage mechanisms occurring in isotropic materials. In composites, the damage mechanisms are numerous, show complex interactions and have many associated variables, such as temperature and strain rate. The detection and monitoring of these damage mechanisms is crucial for reliable life-time prediction. Therefore, over the years, many damage monitoring approaches have been proposed. However, there is currently no universal technique that allows predicting the health of composite materials. Current standard inspection techniques, such as ultrasonic inspection [1] and radiography [1,2], do not answer the need for continuous in-service and in-situ monitoring. For this purpose, other techniques based on extrinsic sensors, such as strain gauges, accelerometers, and fiber optics can be used. Surface bonded strain gauges are often the obvious choice, but they require challenging wiring and calibration. Moreover, they are not able to detect important internal damage mechanisms, such as delaminations or fiber debonding, because such damages are internal [3] and quite often can only be sensed at micro-scale levels. Fiber optic sensors can partially solve these issues, since they can be embedded in composites

[4,5], but they equally suffer from critical limitations. For instance, they are sensitive mainly in the axial direction, they can be affected by high signal distortion in presence of non-uniform strain fields [6], they do not allow full field measurements and they need special adjustments for temperature compensation.
In order to tackle these problems in a more effective way, new sensor technologies need to be developed and investigated. Our research groups are currently working on the conception of novel smart composites which can achieve self-sensing capabilities by exploiting innovative optical and electrical micro/nano sensing principles. This paper presents three of the sensing approaches which are currently under analysis.
The first approach is based on optical measurements and consists of ultrathin polymeric photonic foils for multi-axial strain sensing [7]. These sensing foils, can be manufactured with a thickness of few tens of micrometers and can be embedded in composites components.
The second approach described in this paper is also based on optical measurements and exploits the inherent properties of so called mechanoluminescent (ML) materials, which are able to emit light when subjected to external load [8]. Micro or nano particles of ML materials can be produced and used to be directly embedded in the composite matrix or to realize thin ML foils that can be successively attached on the composite surface.
The third approach is focused on electrical-based sensors, which are manufactures via DC magnetron sputter deposition [9]. Preliminary results on thin-film thermocouples (thickness below 300 nm) embedded in fiber reinforced polymeric coupons are reported and discussed.
The paper is structured as follows. Section 2 describes the ultrathin optical sensors based on gratings inscribed in waveguides created in polymeric substrates. It also presents some of the results obtained in both static and dynamic tests. Section 3 is dedicated to mechanoluminescent materials while Section 4 focuses on the electrical-based thin-film sensors. Section 5 contains the conclusive remarks and ideas for future works.

2. Polymer photonic sensor foils

The polymer photonic sensor foils presented in this research have been developed in order to improve the state-of-the-art in structural health monitoring of composites. These sensors, in fact, offer the same advantage of conventional fiber optics, such as immunity to electromagnetic interference, resistance to corrosion, dense sensor multiplexing in space, reduced weight and size. In addition to this, our sensors can withstand very large elongation and allow very accurate placement and alignment, which is extremely desirable when multi-axial strain in anisotropic materials has to be measured.
The sensors are obtained by inscribing Bragg gratings in waveguides obtained in polymeric substrates. For more details on the manufacturing process, refer to [7]. The working principle of our photonic sensor foils is similar to that of a conventional fiber Bragg grating sensor [10]. If broadband light is injected in the photonic sensor, the Bragg gratings act as mirrors and (in accordance with the Bragg law [10]) reflect back only the wavelengths associated with their grating pitches. Figure 1 shows one of the tri-axial photonic sensors used for this research. The sensor was made using OrmoClad® and OrmoCore® material and its final dimensions were 40 mm × 40 mm × 190 µm (although sensors of smaller size are feasible).

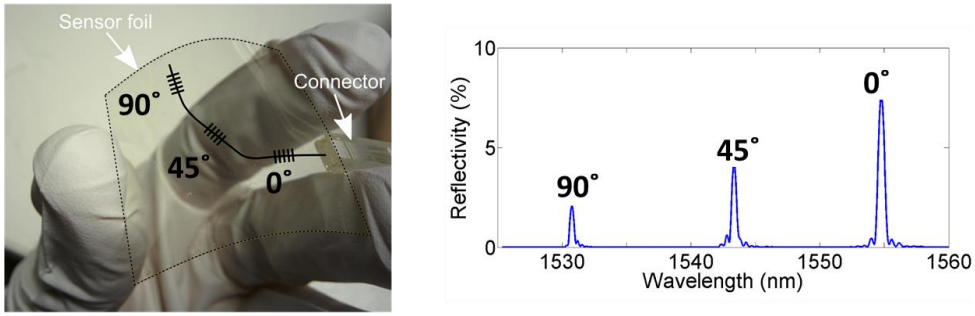


Figure 1. Tri-axial polymer sensor. (left) Sensor foil and connector; (right) reflected spectrum.

Three Bragg gratings were inscribed in the non-straight polymer waveguide in a 0˚, 45˚, 90˚ configuration. The grating pitches were selected to be slightly different in order to achieve wavelength multiplexing. The reflected wavelengths are shown on the right side of Figure 1.
To investigate the sensing capabilities of the developed photonic sensor foils, both quasi-static and dynamic tests were performed. For the quasi-static tests, one tri-axial sensor foil was glued on a glass fiber reinforced (GFRP) laminate (stacking sequence 0_2) and subjected to 4-point bending test. For the dynamic test, one tri-axial sensor was glued on top of a carbon fiber reinforced (CFRP) laminate (stacking sequence 0_8) whose modal characteristics were obtained using the roving hammer approach [11]. For both type of tests, the optical signal reflected by the photonic sensor foil was acquired using an FBGS 804 interrogator. The acquired signal was successively demodulated in Matlab®. Figure 2 shows the results for both type of tests.

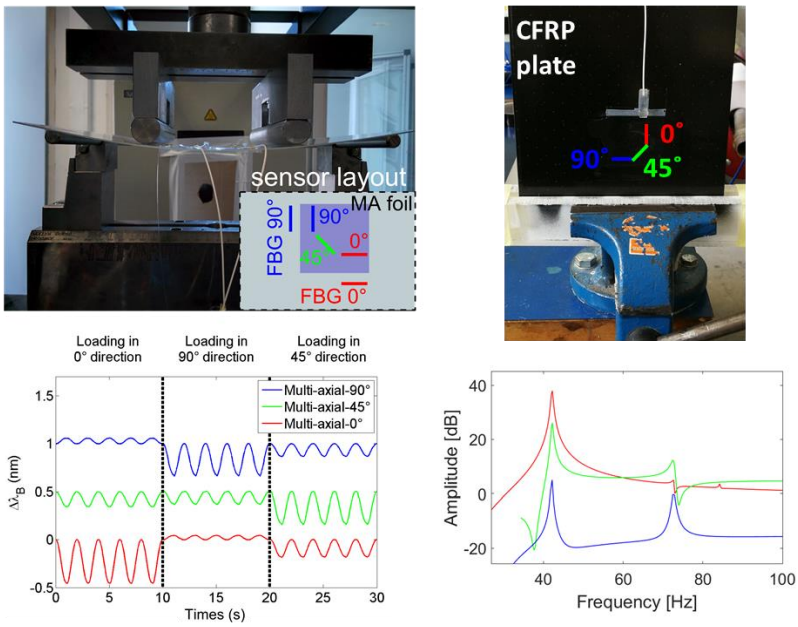


Figure 2. Multi-axial strains measured with polymer photonic sensor foils under 4-point bending (left) and roving impact hammer (right) test.

The 4-point bending test was carried out in order to reproduce three loading conditions: 1) loading acting in the direction of the reinforcement fibers (0˚); 2) loading acting in the transverse direction (90˚); 3) loading acting in the 45˚ direction. The strain amplitude obtained by the different gratings of the photonic foil was in agreement with the direction of the applied load (Fig. 2 bottom left). The same multi-axial sensing capabilities were investigated during the dynamic tests. The frequency response functions calculated from the three gratings showed different resonating amplitudes for different impact locations. For impacts exciting longitudinal (i.e. along 0˚ direction) mode shapes, the grating at 0˚ gave better results. Conversely, for impacts exciting more torsional modes, the 45˚ grating resulted more sensitive.

3. Mechanoluminescent materials

Mechanoluminescence (ML) materials represent a valid alternative route to sense stress distribution in composites. ML materials are special phosphors which emit visible light if loaded. The emitted light has an intensity that can be directly linked to the applied stress level. The latter can therefore be measured by detecting the ML emission and by knowing the relation between emission and stress. Nowadays, phosphors can be readily obtained by putting luminescent ions in transparent crystals.

However, up to now, the number of nondestructive ML phosphors is still lower than 100. Moreover, for many ML phosphors, the load induced emission of light is still quite limited, requiring then sophisticated and expensive test setups and measurement devices. This means that, for many applications, such as for monitoring composites, efforts in engineering and tailoring of new ML phosphors with better emission capabilities are still essential. In this paper we report about the anorthite $(Ca_{0.4}Sr_{0.6})Al_2Si_2O_8:Eu^{2+},Ho^{3+}$ ML material, which we used to visualize stress distribution in an epoxy disc subjected to compression. $(Ca_{0.4}Sr_{0.6})Al_2Si_2O_8$:1%$Eu^{2+}$, 1%$Ho^{3+}$ was prepared by solid state method, i.e. sintering start materials under high temperature. Starting raw materials of total mass ~1.4 g were grounded in an agate mortar and pressed into disc of 16 mm diameter. The disc was placed in a corundum crucible, inserted in a horizontal tube furnace and heated at 1350 °C for 6 hours under 95%N_2-5%H_2. Then, the disc was allowed to cool down till room temperature, before being crashed and grounded into fine power for further characterization. The $(Ca_{0.4}Sr_{0.6})Al_2Si_2O_8:Eu^{2+},Ho^{3+}$ powder was then embedded in a disc (diameter ϕ=25 mm and thickness t=10 mm) made of epoxy resin (Struers® /Epofix®). The weight ratio between the $(Ca_{0.4}Sr_{0.6})Al_2Si_2O_8:Eu^{2+},Ho^{3+}$ powder and the epoxy resin was 3%. The obtained disc was then compressed using an 8801 hydraulic Instron machine. Figure 3 shows the actual ML intensity distribution detected during the test with CMOS camera (Ximea® MC031MG), together with the corresponding Von Mises stress distribution calculated according to the following model

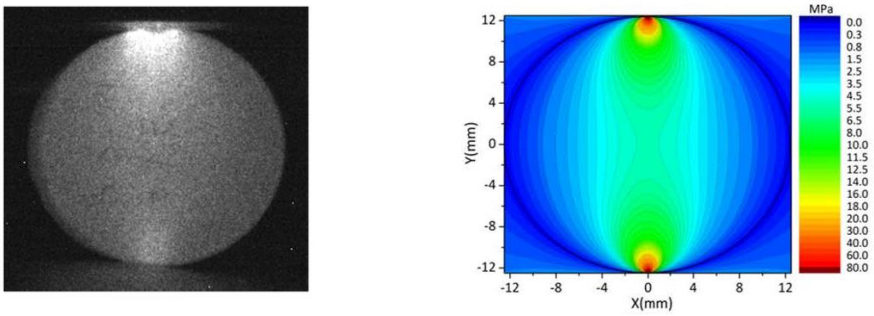
$$\sigma_x = -2P/(\pi l)\{(R-y)x^2/[(R-y)^2+x^2]^2 + (R+y)x^2/[(R+y)^2+x^2]^2 - 0.5/R\}, \quad (1)$$

$$\sigma_y = -2P/(\pi l)\{(R-y)^3/[(R-y)^2+x^2]^2 + (R+y)^3/[(R+y)^2+x^2]^2 - 0.5/R\}, \quad (2)$$

$$\tau_{xy} = 2P/(\pi l)\{(R-y)x^2/[(R-y)^2+x^2]^2 - (R+y)x^2/[(R+y)^2+x^2]^2\} \quad (3)$$

$$\sigma_{VM} = [\sigma_x^2 + \sigma_y^2 - \sigma_x\sigma_y + 3\tau_{xy}^2]^{0.5} \quad (4)$$

where R indicates the disc radius, P the diametric compression load acting along y-direction, x-y the Cartesian coordinate system and the subscript $_{VM}$ stands for Von Mises.

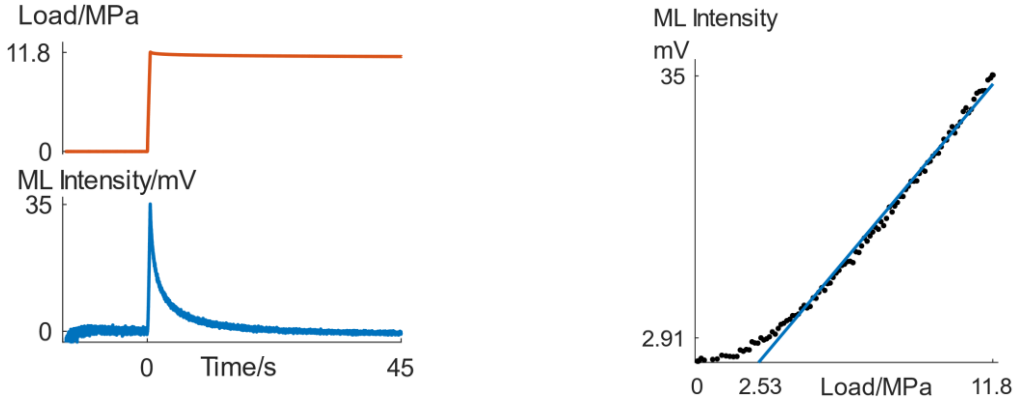


(a) (b)

Figure 3. The ML pattern displayed by $(Ca_{0.4}Sr_{0.6})Al_2Si_2O_8$:1%$Eu^{2+}$, 1%$Ho^{3+}$ under compression (a) agrees with the Von Mises stress from calculation (b).

For quantification purposes, the visualization of the stress field through ML intensity relies on the dependence existing between ML intensity and applied load. Such dependence was obtained by performing tensile tests on dog-bone coupons manufactured vacuum assisted resin transfer molding with the same amount of $(Ca_{0.4}Sr_{0.6})Al_2Si_2O_8:Eu^{2+},Ho^{3+}$ powder used for the disc in Fig.3. Figure 4 reports the evolution of the ML intensity and applied tensile load P as function of time (Fig. 4a) together with the relation between the ML intensity and the applied load (Fig.4b). ML was detected by a photomultiplier tube (Hamamatsu® H10722-21) and synchronized with displacement and force signals via a National Instrument® data acquisition unit (NI 9234) whose sampling rate was 2048 Hz.

Clearly, during the application of the load, the ML-P relation can be considered linear. When the load P reaches the maximum value, the ML intensity is also at its maximum. From this point on, although the load is maintained constant, the ML intensity decreases, which is explained by the relaxation of electrons liberated from the defects of the ML phosphors induced by the stress.



(a) (b)

Figure 4. $(Ca_{0.4}Sr_{0.6})Al_2Si_2O_8$:1%$Eu^{2+}$, 1%$Ho^{3+}$ ML intensity during tensile tests: (a) ML and Load vs time; (b) ML vs Load experimental data (dots) and linear fit (blue line).

4. Thin film thermocouples

Thin film thermocouples (TFTCs) were deposited on uncured prepreg substrates by means of DC magnetron sputtering. Magnetron sputtering is a vacuum assisted deposition technique where ions – from a magnetically confined glow discharge in the vicinity of the target – are electrically accelerated towards the target after which they collide and initiate a collision cascade inside the target material. The emitted clusters and particles from the target consequently, after transport through the gas phase, condense on the substrate and form a thin film. The thin films used to grow TFTCs in this work are in the range from 100-350 nm which is small compared to the dimensions of other conventional sensor types. Therefore, the embedding of TFTCs inside composite materials is expected to only have a minor impact on mechanical properties and structural integrity of the material. Furthermore, thin film thermocouples can be deposited in arbitrary shapes and have a very fast response time because of their low thermal mass. The integration of a TFTC inside a composite laminate is pictured in Figure 5.

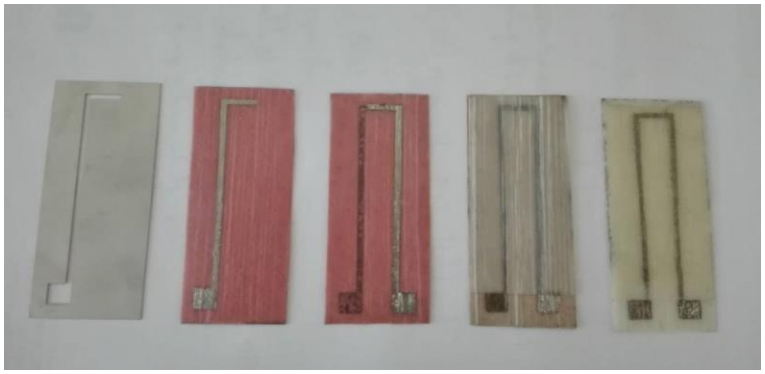


Figure 5. The different steps in embedding a thin film thermocouple in a composite pre-preg lay-up. From left to right: the sputter mask, a first leg of constantan, a second leg of chromel, embedding the TFTC in the lay-up and finally a cured composite. The release foil has a pink color.

A sputter mask has been used for patterning the depositions. A first leg of constantan ($Cu_{55}Ni_{45}$) was deposited on a layer of MTC275 GFRP pre-impregnated material from CASTRO Composites, followed by the deposition of the second leg of chromel ($Ni_{90}Cr_{10}$). The sputter mask was protected from the sticky resin by a release foil. After the deposition was finished, the TFTC was finally embedded by integrating it in a lay-up of GFRP material which was then cured in the oven. As stated above, the thermocouples consist of a bimetallic junction of constantan and chromel thin films. This material combination results in an E-type thermocouple. The output voltage of the TFTCs was measured by an electrometer (Keithley) as function of a continuously increasing temperature of the hot junction while the cold junction was kept constant by a water-cooled copper block at a temperature of 10 °C. The Seebeck coefficient was derived based on the slope of a linear fit. An example of a measurement is presented in Figure 6. The Seebeck coefficient of the presented measurement was derived to be 67.6 µV/˚C, in good agreement with reported bulk values in the range of 68 µV/˚C. Of course, the sensitivity of the TFTC will depend on the deposition conditions. The major benefit of the use of magnetron sputtering over evaporation is the additional energy that is delivered to the adatoms diffusing over the substrate surface improving the overall adhesion on the substrate and film quality. Where the higher energy contained in the material flux is beneficial for several thin film properties, controlling the heat load towards the temperature-sensitive polymeric substrate is one of the major challenges. In general, the heat load towards the substrate will scale as $P_{th} \sim d^{-2}$, with d the distance between the target and the substrate. On the other hand, the sensitivity of the thermocouple, i.e. the Seebeck coefficient S (µV/˚C), decreases as function of increasing target-to-substrate distance. Therefore, a compromise has to be made between the heat load towards the substrate and sensitivity of the thermocouple.

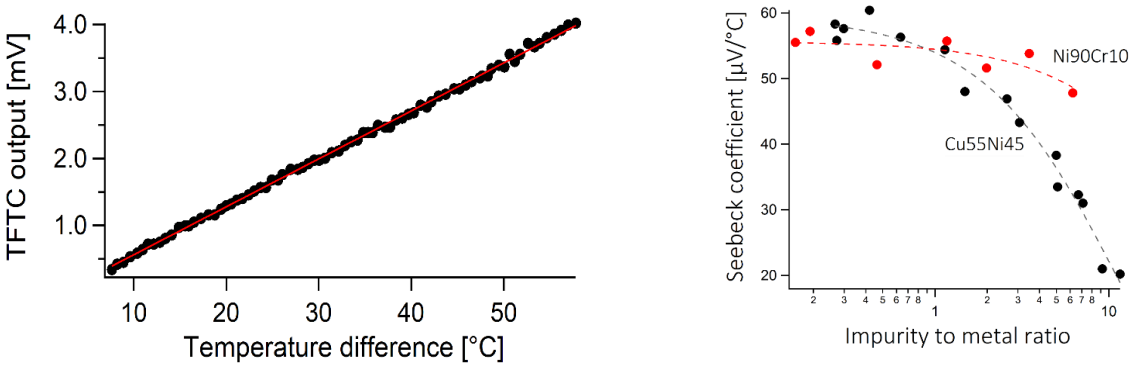


Figure 6. (left) An example of the TFTC output voltage as measured by the electrometer as a function of an increasing temperature difference over the hot and cold junction. The cold junction was kept at a constant temperature of 10 °C. The red line represent the best linear fit. (right) The Seebeck coefficient of a constantan thin film with respect to a chromel wire and the vice versa as function of the impurity-to-metal impingement flux ratio on the substrate surface.

Another aspect related to thin film growth that was under investigation is the degree of contamination present in the vacuum system prior to deposition. It is generally accepted in thin film growth that contaminants and impurities can have a detrimental impact on many thin film properties. Indeed, as presented in Figure 6, we measured the Seebeck coefficient of several constantan and chromel thin films as function of an increasingly contaminated background with atmospheric air. We conclude that the Seebeck coefficient (mainly for constantan) was greatly affect by the presence of impurities during growth. As such, the degree of vacuum, i.e. the background pressure, achieved in the system during growth is therefore a great importance. For a fixed volume and pumping speed, the background pressure in a system decreases as function of time. However, for many industrial applications, large volume coaters are desired at high coating throughput. Polymeric substrates furthermore suffer more outgassing in vacuum than for example cleaned glass or silicon wafers. Therefore, with regard to the

deposition of TFTCs on composite materials, one must again compromise between production time and sensor sensitivity. In conclusion, the profound understanding of the deposition process offers the possibility to tune the deposition conditions to find a proper balance between the optimal sensor properties, and the temperature sensitivity of the used polymers.

5. Conclusions

In the coming future, composite materials will become smarter and smarter and will require more advanced sensors technologies. In this scenario, this paper presents three of the sensing technologies developed at Ghent University which might be implemented in the composite of the future. Ultrathin polymer photonic sensor foils are the first of these technologies. We have demonstrated that this technology is mature to be used for monitoring composites both in static and dynamic loading conditions and in presence of multi-axial loads. Future developments regarding this technology are already undergoing and aim to further miniaturize the sensor size and to speed up the production process. The second sensor technology investigated in this paper is represented by mechanoluminescent ML materials. In particular, we have shown data advanced and novel ML materials can be engineered and embedded in the resin used for composite manufacturing in order to be able to visualize stress. Additionally, we have shown how the ML intensity can be used for stress quantification via the linear dependence existing between ML and applied load. Future developments in this field will focus on the development of new more sensitive ML materials. Activities regarding the controlled synthesis of manganese doped zinc sulfide colloidal nanocrystals and their evaluation as a solution-processable material for mechanoluminescent sensor materials are already ongoing. The third technology presented is based on DC magnetron sputtering. Our study shows that thin film thermocouples are a promising candidate for in-situ temperature measurements inside the bulk of the composite material. Moreover, the results of this study opens a whole new domain of sensors capable of retrieving environmental and operational information such as temperature and strain, in-situ from the bulk of the material under investigation without compromising on its mechanical properties.

Acknowledgments

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