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## Highlights

- First time that the difference between intact, cracked and self-healed TRC laminates is reported in literature.
- Simple acoustic emission parameters are indicative of the fracture mechanisms and show the transition from cracking to mixed mode.
- The differences are evident by the early activity before the mechanical load induces serious damage.
- Self-healed specimens exhibit acoustic behavior closer to the sound rather than the damaged.

# Use of early acoustic emission to evaluate the structural condition and self-healing performance of textile reinforced cements

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## Abstract

Textile reinforced cements (TRC) are innovative materials that are used for repair of existing structures or recently as stand-alone lightweight structural members. Fracture and thermal behavior of these materials are very complicated due to multiple failure modes. The undergoing processes cannot be described by the simple constitutive equation. In this perspective the contribution of monitoring techniques is crucial. Acoustic emission (AE) is used to check the behavior of TRC beams and plates in different states of structural health: intact, thermally pre-cracked and self-healed by polymer powder. This is the first time that the AE behaviors of intact, cracked and self-healed TRC laminates are compared in literature.

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*Keywords:* matrix crack, fibre, delamination, self-healing, bending, attenuation, TRC.

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## 1. Introduction

The acoustic emission technique is used widely for structural health monitoring purposes. Piezoelectric sensors are attached on the material or structure in order to record the response under the excitation of the cracking sources [1]. The parameters of the recorded signals are related to the fracture processes and enable the monitoring of fracture from the start until final failure [2-6]. Fracture mechanisms like matrix cracking and debonding of external patches, show different AE signatures enabling the evaluation of the fracture stage as the load changes [7,8].

In this study textile reinforced cement (TRC) specimens were made of layers of glass fibre mat impregnated by inorganic phosphate cement (IPC) [9,10]. The TRC will show a strain hardening behavior due to the fiber volume fraction of 20%. This makes it useable as an external reinforcement of concrete structures or to be used as lightweight construction element.

The prediction of TRC response is troublesome, considering its laminated and fibrous nature. Interlaminar shear complicates the modeling of the stress fields. In an effort to gain some insight in the fracture process, three-point bending test is performed. Apart from the loading of sound beams, a number of specimens were thermally degraded before the bending test, leading to extensive cracking due to restrained shrinkage of the matrix. Finally, the effect of a self healing polymer agent was also investigated. This work is a continuation of a series of numerous tests where so far the effect of the bending span and the curvature of intact beams has been examined [11,12]. The results were consistent for each different type of specimen (straight and curved beams [11], as well as different loading scenarios [12]. In all of them, the change in load conditions was translated to changes in stress field and the received AE was studied in relation to

the applied stress components (shear/normal). It was shown that the gradual change in the shear over normal stress ( $\sigma_{xy}/\sigma_{xx}$ ) factor between 3% and 15% (caused by a decrease in the bending bottom span) resulted in strong shift of AE parameters and specifically as an example a decrease of frequency content by 50% even in the early AE activity. Therefore, it was demonstrated that the characterization is valid not only for extreme cases of loading (e.g. uniaxial loading) but for mixed mode conditions as well. Since the initial tests on beams were very consistent, the effect of thermal cracking and self healing was examined in this study in order to check the potential of AE parameters to characterize the effect.

In general the behavior of strain hardening TRC in tension can be described by the Aveston Kelly Cooper (ACK) theory [13]. Three stages are distinguished with increasing load. The first stage occurs when the specimen is still undamaged and both fibres and matrix are bearing the applied load. The second stage occurs when cracks start to appear in the matrix material until the point when the matrix is fully cracked. In the third and final stage only the fibres are responsible for the load bearing capacity until specimen fails due to fibre pull-out or delaminations [13]. Therefore, this material offers the possibility to monitor failure mechanisms related to tension like matrix cracking, as well as those related to shear that will eventually lead to the final termination of the structural capacity of the material. The material that was exposed to 300 °C will be cracked due to the restrained shrinkage. It is interesting to examine how the material behaves from the mechanical but especially from the acoustic point of view since one of the main failure mechanisms (cracking) has already been fully or almost fully completed before the start of mechanical loading. In addition, the effect of the self-healing agent is also examined, as the cracks created by the restrained shrinkage are filled by the polymer powder melted

at 300 °C and the mechanical properties are partially restored. The term self-healing is used in the sense that no extra repair material is being applied to seal the cracks, but the material heals by itself at the elevated temperature. The focus in this paper is not given on the mechanical response (and how to increase e.g. the strength) but on the interpretation of the AE activity which offers valuable insight as to the processes that occur in the material highlighting the part of AE recorded early during low load before serious damage is inflicted in the material.

Figure 1 shows the relation of the fracture mechanisms due to bending and the obtained AE waveforms. Initially, fracture is presented by cracks of the matrix due to the stresses of the tensile zone.

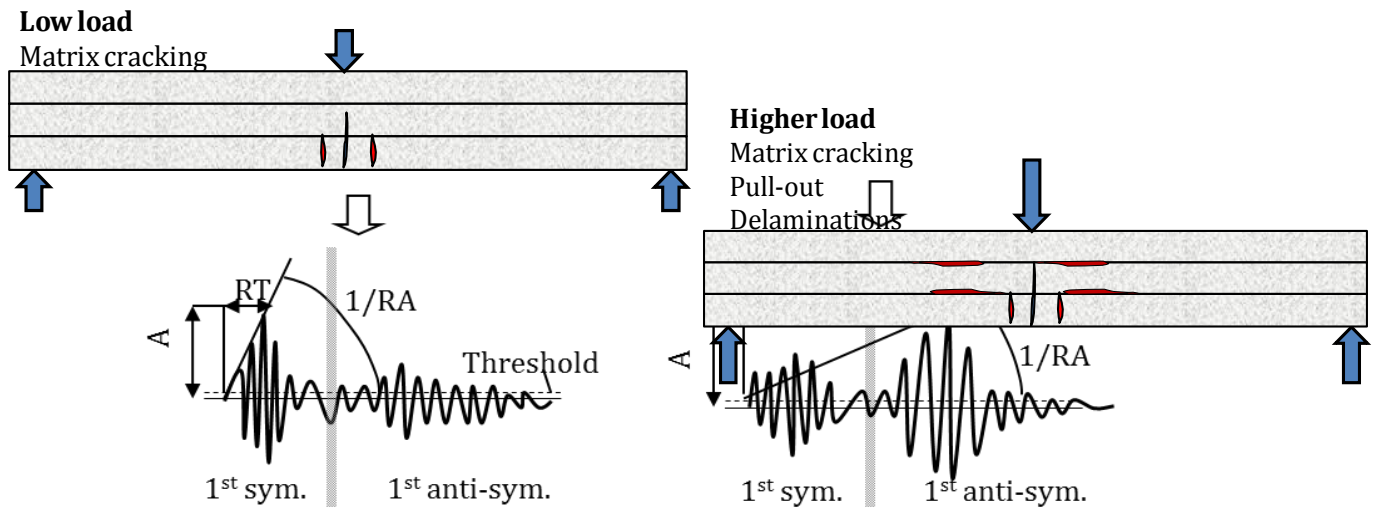


Fig. 1. Typical damage modes and their corresponding AE waveforms.

As load and deflection increase, the fibres that bridge the cracks are pulled out while at the crack tips, delamination events start to develop. Therefore, this shift between fracture mechanisms reasonably triggers different AE signals. As shown in literature, the elastic energy after transverse matrix cracks excite mostly the first symmetric plate wave mode ( $S_0$ ) rather than the antisymmetric ( $A_0$ ) while delaminations increase the proportion of energy of  $A_0$  [14,15]. The relative power of the different modes affects the shape of the received waveforms and consequently the AE parameters, as seen in Fig. 1. The most significant waveform parameters are the amplitude,  $A$ , the rise time,  $RT$ , the  $RA$ -value which is  $RT$  over  $A$ .  $RT$  and  $RA$  strongly depend on the cracking mode, obtaining lower values for cracking fracture and higher for shearing either in the form of delaminations or cracking due to shear stresses [3,5,7,8,11,12,16,17]. A typical frequency indicator is the so-called “average frequency”,  $AF$ , which is the number of threshold crossings divided by the duration while frequency domain features like central and peak frequency are also used.

## 2. Experimental details

### 2.1. Materials and testing

The TRC beams and plates were manufactured by hand layup. Eight randomly chopped glass fibre mats were impregnated with an inorganic phosphate cement (IPC) resulting in a fibre average volume fraction of 20%. The IPC consists of two main components that, if desired, can be supplemented by a third “filler” component. The two main components of IPC are the Wollastonite powder ( $CaSiO_3$ ) and a proprietary metal ion modified phosphoric acid. The powder to liquid mixing weight ratio is 8 to 10.

As third component, a filler material, the ALESTA HR polymer initially developed as a powder coating by Dupont and characterized by heat resistance up to 550 °C was used. It was included in a proportion of 8% by weight, meaning that in the final specimen, the polymer accounted for 8% of the total mass.

After the hand layup process, the plates were inserted in an oven during 24 h at 60 °C in order to cure. Afterwards a number of specimens received an additional heat treatment at 300 °C during 1 hour. The aim was firstly to thermally degrade the plain specimens and secondly to investigate the self healing effect in the specimens containing the heat resistant polymer powder.

The plates were prepared for testing by cutting them and cleaning the surface for the AE sensors. The loading setup used was typical three point bending for beams as shown in Fig. 2a and b, and a point loading for the circular plate geometry (c). The experiment was performed using the INSTRON 5885H with a speed of 1 mm/min. For the case of circular plates the loading was continued until a maximal deflection of approximately 7.5mm, while for the beams the mid span deflection reached even 48 mm for the longest span.

Among the various specimens tested, in the present paper the behavior of four beams (two sound, two thermally degraded) and four plates (two reference, one thermally degraded, one healed) are discussed.

## 2.2. AE monitoring

Two “pico” sensors of Mistras Holdings were used (Fig. 2a) to monitor the occurring failure during the bending test. They are relatively broadband, with maximum sensitivity at 500 kHz. Vaseline grease was applied between the sensors and the specimens for acoustic coupling and the sensors were secured by tape. The threshold was set at 35 dB and the signals were recorded in a micro-Samos acquisition board after pre-amplification of 40 dB. Photograph of the setup can be seen in Fig. 2b. Two span lengths were tested: 330 mm (“long” span) and 150 mm (“short” span). The ratio between normal and shear stresses differ for the two spans, leading to a relatively earlier occurrence of the interlaminar shear damage type for the short span compared to the matrix cracking damage type, than for the long span. TRC circular plates were also tested in bending through central point loading. In this case four sensors were used defining a square of 100 mm side as seen in Fig. 2c. In this case planar localization of the AE sources was enabled with an “event” defined when at least 3 sensors received the response in a limited pre-defined time window.

## 3. Mechanical Results

The load deflection curves are presented in Fig. 3. For long spans the maximum load is lower while they undertake larger mid-span deflection. The thermally degraded specimens exhibit notably lower maximum load than the intact ones since their matrix is already cracked due to the thermal exposure. In addition, their stiffness is much lower compared to the corresponding beams of the same length, as evident from the slope of the curves.

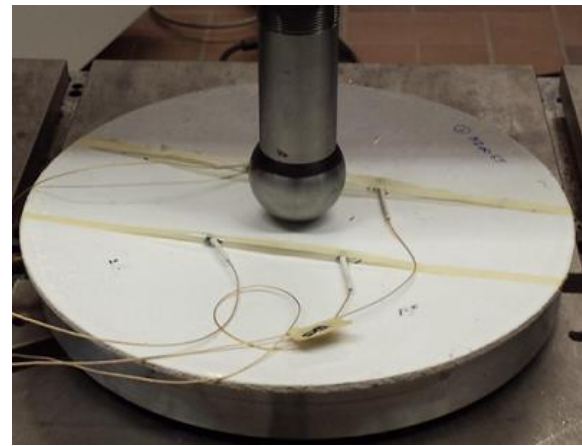
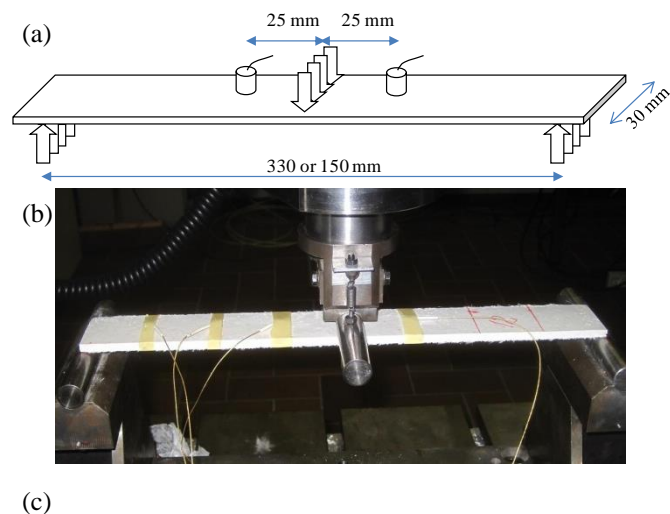


Fig. 2 (a) sketch of bending setup and AE sensors, (b) photograph during the bending test (the two sensors at the left are for reference), (c) during the plate test.

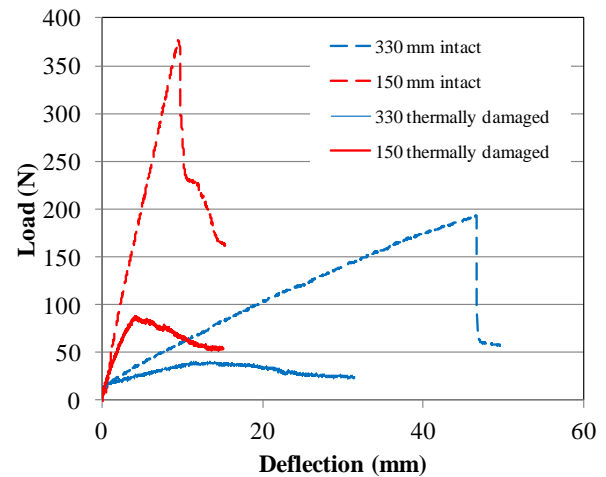


Fig. 3. Load vs. deflection for two sound and two pre-cracked TRC laminates with different bending spans.

## 4. AE results

### 4.1. Effect of thermal cracking

The total AE activity of the beams in bending is shown in Fig. 4 separately for the two different spans. The activity of the thermally degraded sample starts much earlier as it already contains numerous cracks that either propagate under low load or they trigger the next failure mechanisms in the sequence: delaminations and fibre pull out. On the other hand the AE activity of the intact specimens delays considerably since during the elastic stage neither serious fracture nor AE is expected. However, since the intact ones have not cracked yet, their potential for giving new events is higher in total and as normal at some point in the test their accumulated activity overpasses that of the thermally damaged ones. The situation is very similar for Fig. 4b for the shorter beams of 150 mm span. In this case the experiment lasted less due to the smaller maximum deflection that these specimens can bear. The activity is much higher than the long span beams since apart from the normal stresses, considerable shear stresses develop as well for the same external load [12]. In any case it is clear

that the thermally degraded beams emit less total activity than the intact ones, which is reasonable since the failure mechanism of matrix cracking has already been exhausted in the pre-treatment in the oven.

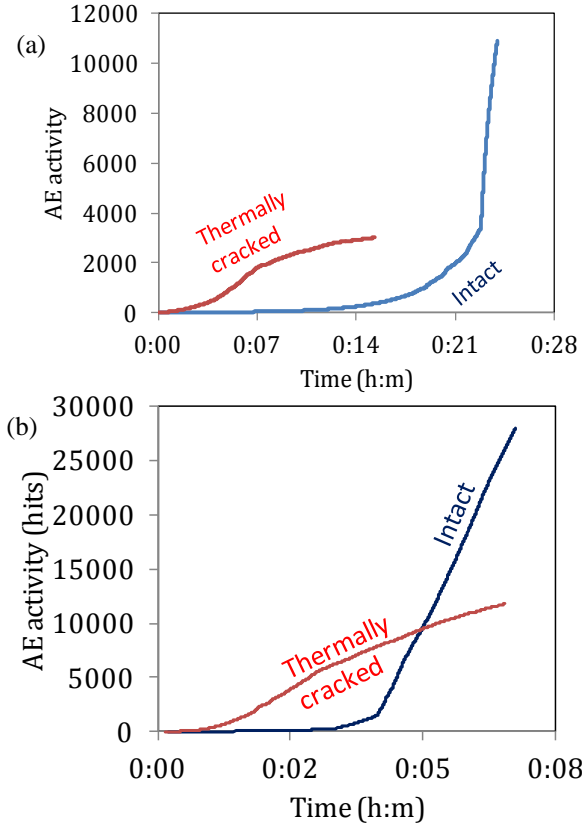


Fig. 4. AE activity history for TRC beams of span (a) 330 mm, (b) 150 mm

It is interesting to check the AE waveform parameters since it has been shown that they are indicative of the failure mode. Fig. 5a shows the AF data as well as the moving average trend line. For the intact beam the average line starts just below 300 kHz, moving smoothly downwards before collapsing at the moment of macroscopic fracture after 21 min. However, for the thermally cracked specimen the initial value is 200 kHz moving faster down to 100 kHz.

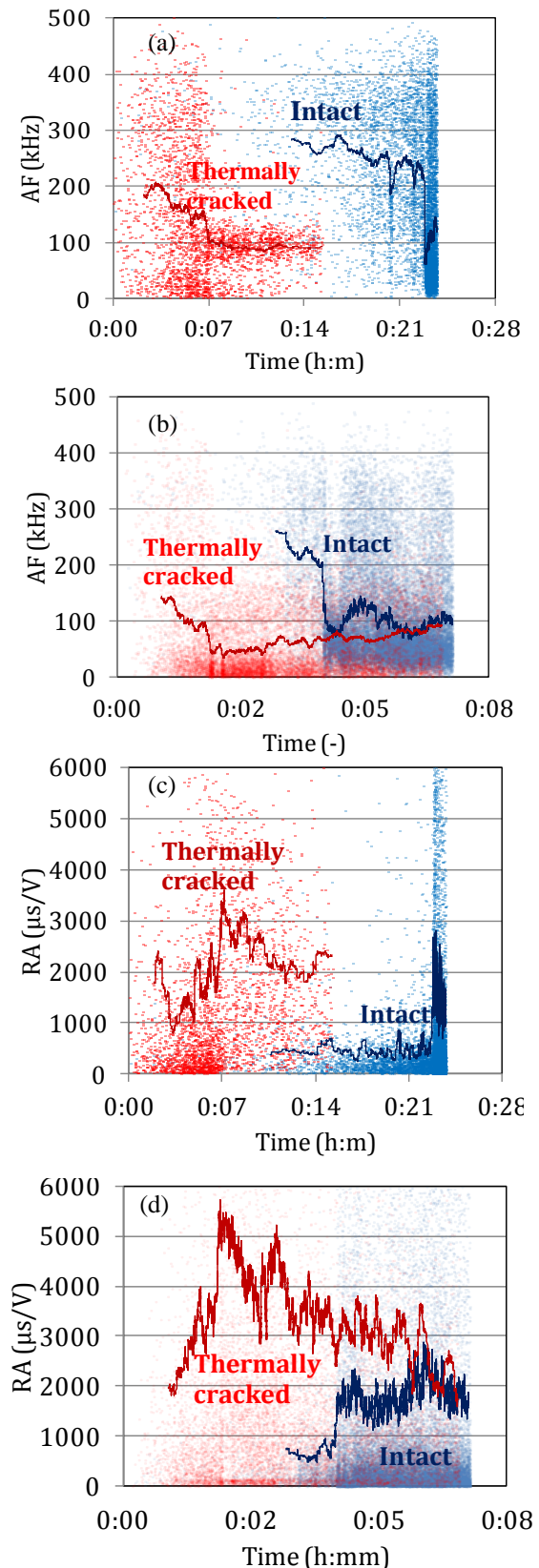


Fig. 5 AF vs. time for (a) long span, (b) short span. RA value vs. time for (c) long and (d) short span. The solid lines are moving average of recent 300 points.

Similar trends can be seen in Fig. 5b for the shorter span beams. Again the thermally cracked starts with AE activity approximately 100 kHz lower than the intact. This is indicative of the different failure mechanisms that are

activated from the start. Typically for the intact beam the first events of fracture come from the cracking of the brittle matrix under the tensile stress of bending. After substantial matrix cracking has developed, mixed mode damage is activated which is evident by the lower frequencies that are progressively reached. On the other hand the degraded sample simply cannot crack anymore and since the matrix does not carry any external load the mixed mode is activated early. This is the reason that lower frequencies are recorded even under low load at the first minutes of the test.

Figs. 5c and d show the behavior of the RA parameter in the same fashion as AF. The RA values of the degraded samples are more than double than the intact from the start indicating that the failure processes contain a higher proportion of delaminations and fibre pull out compared to cement cracking, which is dominant at the early stage of loading in the intact specimens. Only at the end and during the macroscopic failure of the specimens the levels of the lines seem to converge since in all specimens delaminations are the final dominant mechanism.

In order to quantify the differences, the initial population of 200 hits was isolated in each case and the results are included in Fig. 6 for AF (a) and RA (b). In both cases of spans the thermally degraded specimens exhibit lower frequency by approximately 100 kHz compared to the intact specimens. At the same time their RA values increase compared to the intact indicating that even at the very early stages of the test when the load is still a small fraction of the ultimate value, the preliminary manifestation of the failure mechanisms is already available. The differences are quite clear, and show the sensitivity of AE features to the source mechanisms. Frequency changes by more than 30% while RA increases by several hundred percent when delaminations are dominant as happens in the thermally degraded samples.

It is noted that the span has also an effect as can be seen by the differences between the 330 mm and 150 mm corresponding specimens, a result that has been investigated previously [12]. As the span becomes shorter, the proportion of shear stresses becomes higher and therefore, the AE frequency becomes lower and RA higher as seen for the intact beams. Therefore, comparisons between thermally degraded and intact specimens should be made only for the same span.

#### 4.2. Influence of self-healing agent

Since it was verified that the technique is sensitive enough and could capture the differences between material conditions (intact/thermally damaged) and loading conditions (long/short span) it was deemed necessary to proceed to the next, more realistic step which was the plate geometry and apply the healing powder there. Concerning the plate tests, the analysis of the AE data was similar. The results in terms of frequency and RA are presented in Fig. 7a. The thermally degraded plate exhibited a substantially lower frequency (195 kHz) compared to the intact plates (295 kHz). However, the thermally degraded plate which contained the self-healing agent exhibited a frequency in between (230 kHz).

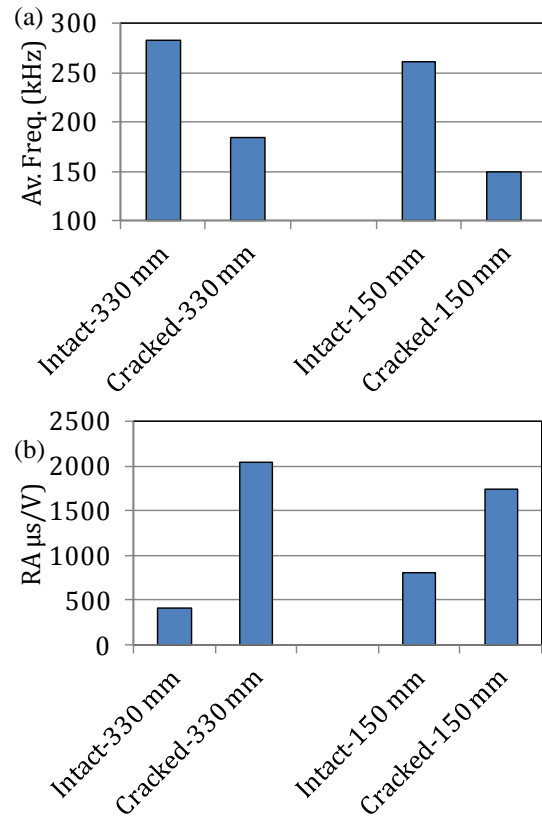


Fig. 6 AE average parameter values for the first 200 hits (a) AF, (b) RA.

In Fig. 7b the corresponding RA values are shown for the same initial population. The intact plates (with and without the self-healing agent) exhibited values around 500-600  $\mu\text{s/V}$ . The degraded plate exhibited much higher values at the level of 1500  $\mu\text{s/V}$  showing again the difference in the initially activated mechanisms. However, the self-healed plate exhibited RA values much restored to the intact ones at less than 800  $\mu\text{s/V}$ . Both these features show that in the self-healed plate the fracture processes are in between the two extreme cases. In case the self-healing agent was not active at all, the failure would start by delaminations directly and the AE features would be close to the thermally degraded one. On the other hand if the polymer agent was effective in completely sealing the cracks and fully restoring the structural capacity of the plates, the AE features would resemble the ones of the intact plates. These results show that the polymer certainly makes a difference and restores the properties at least up to a significant degree.

Finally a wave propagation feature of the AE signals is considered. Since the planar localization of sources was active, this enabled to locate the AE events. Each “event” produced waves (hits) received by all four or at least three different transducers. According to the original position of the event, the waves propagated over a different distance towards the different sensors. This information is available through the planar localization algorithm and along with the amplitude of these hits it allows evaluation of the attenuation characteristics in all plates. Attenuation is known to be sensitive to the heterogeneity of the cementitious materials and allows accurate assessment of their internal nature



[18,19]. The hits were sorted according to their distance from the source and the function between amplitude and distance can be seen in Fig. 8 for the different cases. For the intact plate the average value of a hit received at the closest sensor is around 46 dB while it is below 40 dB for distances longer than 80 mm, leading to an attenuation value of 0.083 dB/mm as seen by the slope of the linear fit to the data. For the thermally degraded (pre-cracked) plate this slope becomes much steeper leading to an increased absolute value of almost 0.14 dB/mm. This is a reasonable result since the distributed micro-cracking hinders propagation of the elastic waves creating several interfaces and causing multiple reflections. Interestingly, the self-healed plate showed a complete restoration of the slope to the initial value (0.084 dB/mm). This shows that the cracks were physically closed allowing the waves to propagate without many losses due to reflections. Although the restoration of the mechanical properties depends on the properties of the polymer and the match to the matrix and may or may not be reached in full, the attenuation shows that at the high temperature the melt polymer sealed the empty space created by the cracks.

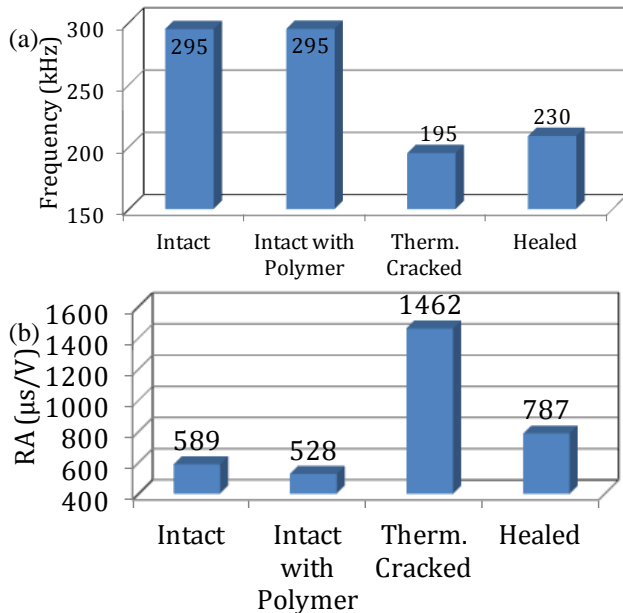


Fig. 7 (a) AF and (b) RA of different plates.

## 5. Conclusions

This study discusses the possibility of using simple AE and wave features as indicators of the internal conditions in TRC laminates. The focus is put on the characterization of the failure mode (matrix cracking and the more complex mode including delaminations and pull-out), on the initial condition of the laminates (intact or cracked) and the effect of self-healing agent. The results show that AE parameters are indicative of the processes that occur within the material. The effect of cracking is shown by a strong drop of the emitted frequencies and a high increase of RA value. AE also provides the possibility to characterize the restoration action due to the existence of self-healing agent aiming at closing the matrix cracks. The AE parameters seem to be partially restored meaning that the self-healed specimens start to crack

again, instead of moving directly to the next failure stage of delaminations. In addition, the attenuation of the acoustic waves is also sensitive enough to indicate the physical filling of the cracks by the self-healing agent. This is the first time that the difference between intact, cracked and self-healed TRC laminates is studied and shows very encouraging results as to the health monitoring of structures, reinforced or entirely built by TRC.

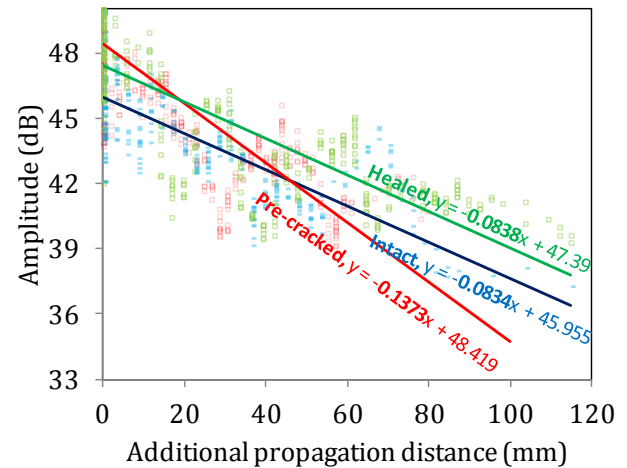


Fig. 8 Amplitude of the AE hits vs. propagation distance. The points are shown in transparent mode in order not to mask the trend line.

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