

## On the ultrasonic assessment of adhesion between polymer coating and concrete substrate

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### Abstract

In this paper, the ultrasonic methods were used to assess an adhesion between polymer composite and concrete substrate. The usability of indirect (surface) ultrasonic methods was evaluated on the example of commercial polymer coating. The relationships between pull-off strength and propagation of ultrasonic wave were established and analyzed. The effect of chemical composition and thickness of PC system was discussed. The results confirmed usefulness of indirect ultrasonic method for non-destructive mapping of adhesion between polymer composite and concrete substrate.

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### 1. Introduction

The repairing and anti-corrosion protection of the building structure is one of the most important application of the polymer composites [1,2]. In such case, a two component system: cement concrete substrate (CC) in contact with polymer composite (PC) is produced. The compatibility of PC–CC systems is here the main problem [3–5]. The adhesion between overlays and concrete substrate is one of the most important factors that affect the reliability and durability of repair [3,6,7]. The high adhesion causes higher tolerance on non-compatibility of properties of the both materials [3]. According to the many standards and guidelines, e.g. new European Standard EN 1504-10 [8] and ACI Concrete Repair Manual [9], a pull-off test is recommended for assessment of a bond strength and a bond quality in repair systems. The need for the adhesion monitoring exists in the case of large area objects like industrial floors, bridge decks, injected concrete structures [7,10].

Fig. 1 shows the example of the pull-off strength distribution for industrial floor (size of approx.:  $60 \times 160$  m). In this case, the measurement of pull-off strength, due to its destructive character, was restricted by contractors to the local places. Therefore, the elaboration of reliable non-destructive method for adhesion mapping is one of the most important tasks. Recently, it is noted growing interest in application of non-destructive techniques (NDT) for evaluation of concrete structures. However, these investigations are rarely focused on assessment of adhesion in repair systems.

The aim of this work was to evaluate the usability of ultrasonic methods to assess adhesion between polymer composites and concrete substrate. The industrial polymer floors were used as an example.

### 2. Non-destructive assessment of adhesion of two-layer systems

The possible defects in the system created by PC and CC can be categorized into two main types [14]:

- “adhesion type” (at contact zone of PC–CC system): delaminations, flaws, poor adhesion area,

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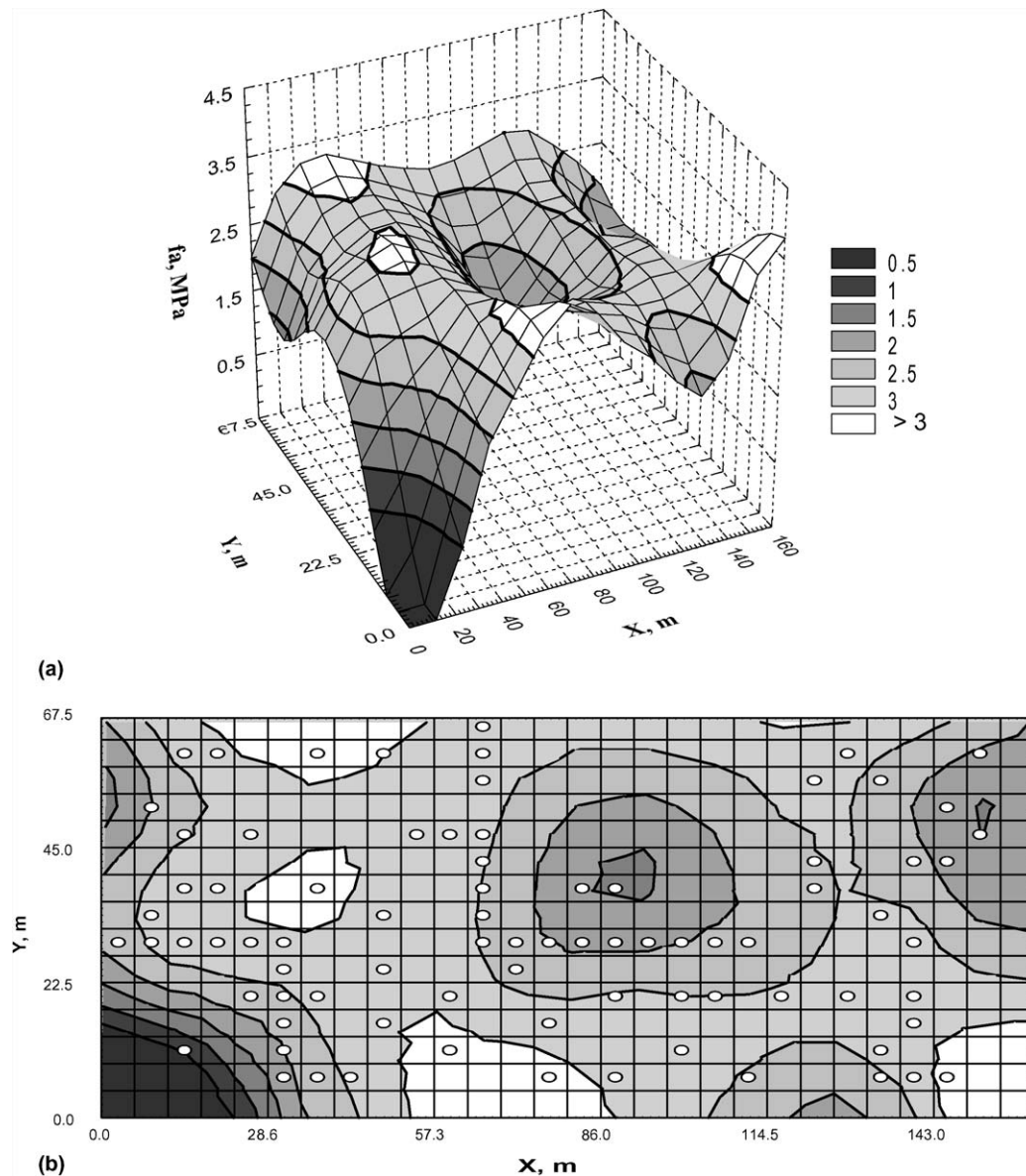


Fig. 1. Example of pull-off strength distribution for surface-hardened industrial floors (area size approx.: 67.5 m × 160 m) estimated on the base of data obtained for pull-off measurements (○): (a) 3D distribution and (b) contour line of pull-off strength; non-uniform distribution of measurement points resulted from arbitrary decision of contractors.

- “cohesion type” (in polymer composite or/and concrete substrate): porosity, cracks, honeycombing or completely hardened resin in the case of PC.

The non-destructive evaluation of quality of multi-layer systems (layer thickness, flaws, delaminations, areas of poor adhesion) is a difficult. The relation between the specific acoustic impedance of the system components should be taken into account as the important factors influencing ultrasonic wave propagation in particular elements of the system, as well as through the internal interfaces. The effect of surface roughness resulted from preparation of concrete substrate before the repair should be also considered. The roughness of significant value can cause an additional distortion of ultrasonic wave [12]. For example, Jósko

observed a strong wave scattering as a significant effect of the surface roughness for polymer coated steel substrate if the total profile height,  $R_z$ , was higher than 0.5 mm [13]. In the case of building structures, the NDT technique selected should also give a possibility for testing from one side of the object. This implies that echo method or indirect pulse velocity method are mainly considered.

A large majority of NDT methods applicable to multi-layer systems are focused on defects detection at the interface surface [10,11,14,15]. A few papers only are related to evaluation of the adhesion strength between repair material and concrete substrate [16–18]. In those studies, the adhesion assessment was based on the assumption that the places with poor adhesion influenced the recorded waveform and the adhesion strength is related to value of

selected parameters for reference state – good bonding joint. For example, Tan et al. (1996) have used relative amplitude method for evaluation of cement adhesion in wall tiles. They have assumed that the measure of bond quality is relation between intensity of reflected signal from a free (unbounded) tile and from a tile–cement interface. If the bond strength decreases the intensity of reflected signal is higher. They have obtained maximum of the pull-off strength and the minimum of relative signal amplitude for cement adhesive with water to ratio,  $w/c = 0.5$ .

The evaluation of the adhesion in PC–CC systems with an ultrasonic echo method is more difficult because of the high difference between the acoustic impedances of the PC coating and concrete substrate (reflection coefficient  $R = +0.40$ ). Garbacz and Garboczi [19] have evaluated the adhesion between polymer coating and concrete substrate. The value of amplitude amplification needed to reach 0.8 of maximum amplitude (i.e. 80% of full screen height), called here as the coefficient,  $W_{0.8H}$ , was treated as a measure of polymer coating adhesion to concrete substrate. They have used two commercial transducers for longitudinal wave (frequency 0.8–3 MHz and 10 MHz with delay line). Simultaneously the measurements for free (unbounded) polymer coating (2 mm thickness) have been made. Analysis of the amplification coefficient ( $W_{0.8H}$ ) results showed that a relation between the level of amplification and the pull-off bond strength did not exist. The value of attenuation  $W_{0.8H}$  for a “zero-adhesion” state of polymer coating was located between the points corresponding to different levels of adhesion (Fig. 2(a) and (b)). The observed differences in the attenuation were explained, at least in 70%, by variation in coating thickness (Fig. 2(c)).

On the basis of literature data other parameters describing the propagation of stress waves might be considered as the parameters for characterization of the adhesion strength in the PC–CC system [20–25]. For example: pulse velocity or amplitude of the P-, S- and R-waves or their combination, e.g. peak-to-peak amplitude or an attenuation coefficient. However, parameters mentioned above correspond to the particular point in the time domain and require high repeatability of the signal. This is not easy to ensure in the case of such non-homogenous systems like PC–CC.

In this work the approach proposed by Garbacz et al. [26] was used for evaluation of the adhesion between polymer coating and concrete substrate. Experimental studies were carried out to determine effects of the chemical composition and thickness of polymer coating as well as the substrate surface quality on the ultrasonic wave propagation through the PC–CC system.

### 3. Experimental procedure

#### 3.1. Ultrasonic test procedure

The investigations were carried out with the indirect (surface) ultrasonic pulse velocity method using a commercial concrete tester CT1 Unipan-Ultrasonic with set of associated transducer pairs. The normal transmitting and receiving transducers with build-in amplifier have diameter of 20 mm were used. The source pulse frequency of the compressive wave was 100 kHz. The distance between middle of transducers was fixed and equal to 80 mm. A gel was used as a coupling medium to improve acoustic contact between the samples and the transducers. The transmitted wave pulse was transformed into digital signals (sampling period 0.2  $\mu$ s) by an A/D converter system and then fed into a microcomputer. The time versus voltage record was averaged with six previously recorded pulse signals to reduce the effects of random noise and the heterogeneity of the microstructures of both the polymer composites and the concrete substrate. Each ultrasonic pulse was recorded after tester stabilization was indicated. These non-destructive measurements were carried out after 7 days of hardening of each type of coating.

The propagation of ultrasonic waves through the PC–CC system was characterized by the pulse velocity and by the changes of a mean square value parameter,  $MS(t)$ . Pulse velocity was calculated dividing distance between transducers by the transit time. The “surface” pulse velocity obtained in this way is a little different. In the comparative analysis presented here the pulse velocity was not recalculated. Second parameter, the MS value at a given point of time domain was calculated from the formula

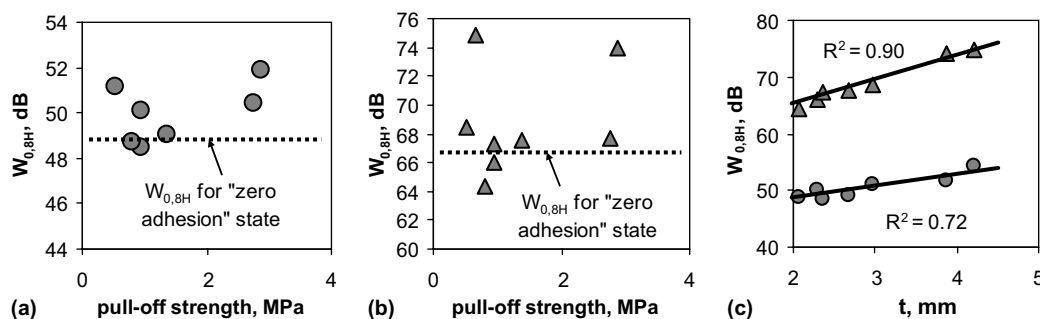


Fig. 2. The pull-off strength against amplification  $W_{0.8H}$  for tested epoxy floor system after 28 d of floor hardening with transducers: (a) S12HB0.8-3 (●) and (b) 10V202 (▲); (c) amplification coefficient  $W_{0.8H}$  vs. “true” thickness for both transducers.

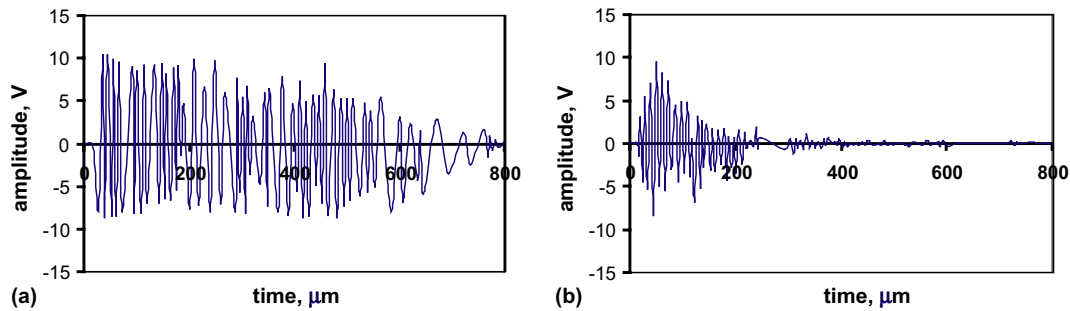


Fig. 3. Example of received ultrasonic pulse for polymer coating with (a) good adhesion (>2 MPa) and (b) “zero-adhesion” in the PC–CC system (indirect pulse velocity method).

$$MS(t) = \frac{\sum_{n_0}^{i-n_0} A_i A_i}{(n_i - n_0)} \quad (1)$$

where  $A_i$  is the amplitude of the  $i$ th-recorded point [V] and  $n_0$  is the number of the first point with amplitude different than zero.

The plot of the MS value describes the amplitude variance in the time domain and is a representation of the attenuation of the wave pulse for the given period in time domain. To certain degree, this approach corresponds to characterization of profile roughness with root mean square deviation [27]. In this work, it was assumed that for a sample area of poor adhesion, the MS value would statistically decrease faster than for an area of high adhesion. This assumption was justified by waveforms obtained for PC–CC system with good and poor adhesion (Fig. 3).

### 3.2. Materials and tested floor systems

The investigations were carried out for the polymer industrial floor systems described in Table 1: water dispersion of epoxy resin (EP-1), solvent epoxy resin (EP-2) and polyurethane resin (PUR) coatings. All coating systems (2 mm thickness) were layered on a cement concrete substrate (C25/30 acc. EN 206-1:2003), prepared from the same concrete mix. This concrete class was selected to be close to the real concrete substrate used in the floor industry (concrete with compressive strengths greater than

25 MPa). For the evaluation of the usability of the pulse velocity method for assessment of the adhesion, three experiments were carried out

- ultrasonic evaluation of adhesion at maximum and minimum adhesion levels,
- analysis of relationship between pull-off strength and ultrasonic parameters,
- ultrasonic detection of defects in the PC–CC system.

Additionally, the results previously obtained for vinyl-ester and epoxy coatings (3 mm) and polyurethane coating (thickness of 1 mm) was also analyzed [26].

### 3.3. Adhesion measurements

The adhesion between polymer coating and concrete substrate was characterized by the results of a pull-off test. The pull-off test was carried out with a digital apparatus acc. to EN-1542 after ultrasonic tests. The steel disks of 50 mm diameter were bonded to the top of polymer coating with epoxy glue. The failure mode was also registered.

## 4. Ultrasonic evaluation of adhesion at boundary conditions

The pulse waveforms were analyzed for two boundary conditions:

- maximum adhesion (for a given type of polymer coating),
- zero-adhesion (corresponding to delamination).

The polymer coating was prepared separately and next put onto a cement concrete substrate to simulate delamination. The investigations were carried out for three commercial floor systems: EP-1, EP-2 and PUR, of nominal thickness 2 mm and containing a quartz fine filler (maximum grain diameter  $D_{\max} < 0.1$  mm).

The average pull-off strength obtained for the EP-1, EP-2 and PUR coating systems was equal to 3.15 MPa, 2.10 MPa, and 3.20 MPa, respectively. Cohesive failure in the concrete substrate was obtained during the pull-off test for the EP-1 and EP-2 coatings. In the case of the PUR

Table 1  
Chemical composition and pull-off strength of tested polymer coatings (acc producer data)

Property	Symbol of polymer coating		
	EP-1	EP-2	PUR
Chemical composition	Water dispersion of epoxy resin	Epoxy resin	Polyurethane resin
Number of components	2	2	2
Max. grain size of fine filler, mm	<0.1	<0.1	<0.1
Nominal thickness, mm	0.7–3	1–5	1–4
Pull-off strength (concrete substrate compressive strength >25 MPa)	>1.5 MPa	>2.0	>2

coating, a mixed failure mode (70% cohesive failure in concrete and 30% adhesive failure in the PC–CC bond line) was observed. This confirmed that for the “maximum adhesion” state high adhesion in the PC–CC system was actually developed. The transit time and the MS value were selected as useful parameters for further investigations. The amplitude values registered by the tester were not significantly correlated with the adhesion level for all tested systems. This was confirmed by the MS value distribution, which were similar up to 100  $\mu$ s for the both “maximum” and “zero” adhesion states.

The pulse velocity obtained for maximum adhesion state was at least about 1000 m/s higher in comparison to “zero

adhesion” state for all tested systems (Table 2). In the case of the epoxy coating, the pulse velocity for the zero-adhesion state,  $v_p = 2960$  m/s, was close to the pulse velocity determined with the direct method,  $v_p = 2650$  m/s [19,26]. This result confirmed that the ultrasonic wave penetrated both the polymer coating and the concrete substrate in the case maximum bonding. The ultrasonic wave traveled only through the polymer coating in the delamination place. The similar results were obtained for other epoxy and polyurethane coatings of 3 mm thick [26].

The MS values for coatings with maximum adhesion and zero adhesion were also significantly different (Fig. 4). The MS plot determined for the concrete substrate

Table 2

The pulse velocity and pull-off strength for polymer coating at the maximum adhesion and “zero-adhesion” stage

Polymer coating	Pulse velocity (km/s) (stand. dev.)	Adhesion strength (MPa)	Symbols in Fig. 4	Pulse velocity (km/s) (stand. dev.)	Symbols in Fig. 4
<i>Max adhesion for given coating</i>				<i>Zero adhesion (delamination)</i>	
EP-1 (2 mm)	3.94 (0.03)	3.15	○	2.96 (0.16)	●
EP-2 (2 mm)	4.10 (0.05)	2.10	□	2.79 (0.42)	■
PUR-1 (2 mm)	4.59 (0.11)	3.20	△	3.06 (0.43)	▲
EP-3 (3 mm) <sup>a</sup>	4.46 (0.32)	3.23	1	2.63 (0.07)	3
PUR-2 (3 mm) <sup>a</sup>	4.42 (0.20)	2.10	2	2.33 (0.10)	4
Concrete substrate, CC: Pulse velocity 4.908 km/s					

<sup>a</sup>According to [26].

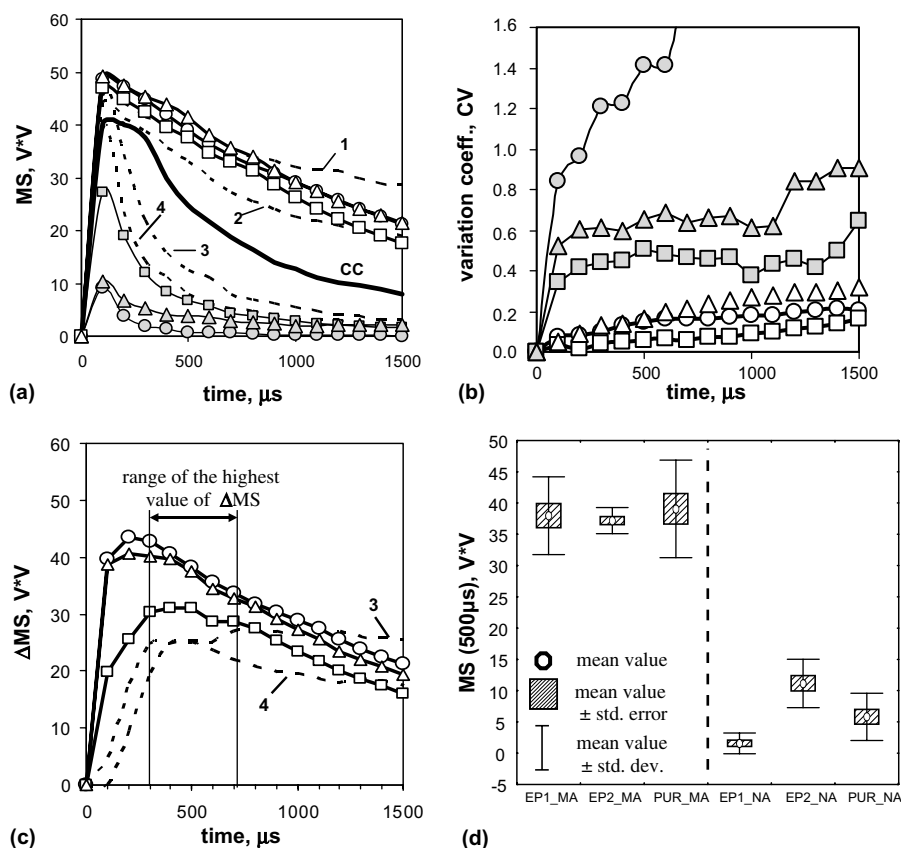


Fig. 4. The results in time domain for boundary conditions: (a) MS( $t$ ) distribution, (b) variation coefficient of MS( $t$ ), (c) absolute difference of MS( $t$ ) for maximum adhesion – “MA” and “zero-adhesion” – “NA”; (d) parameters of MS(500  $\mu$ s) distribution; symbols like in Table 2.

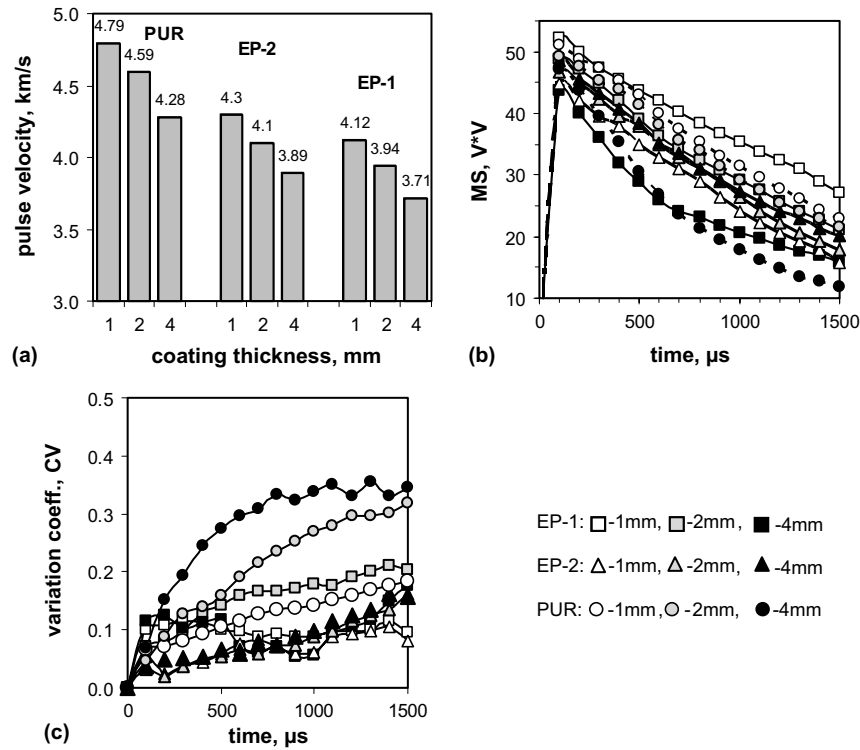


Fig. 5. Pulse velocity (a), MS value distribution (b) and variation coefficient of  $MS(t)$  (c) for the polymer coatings of different thickness.

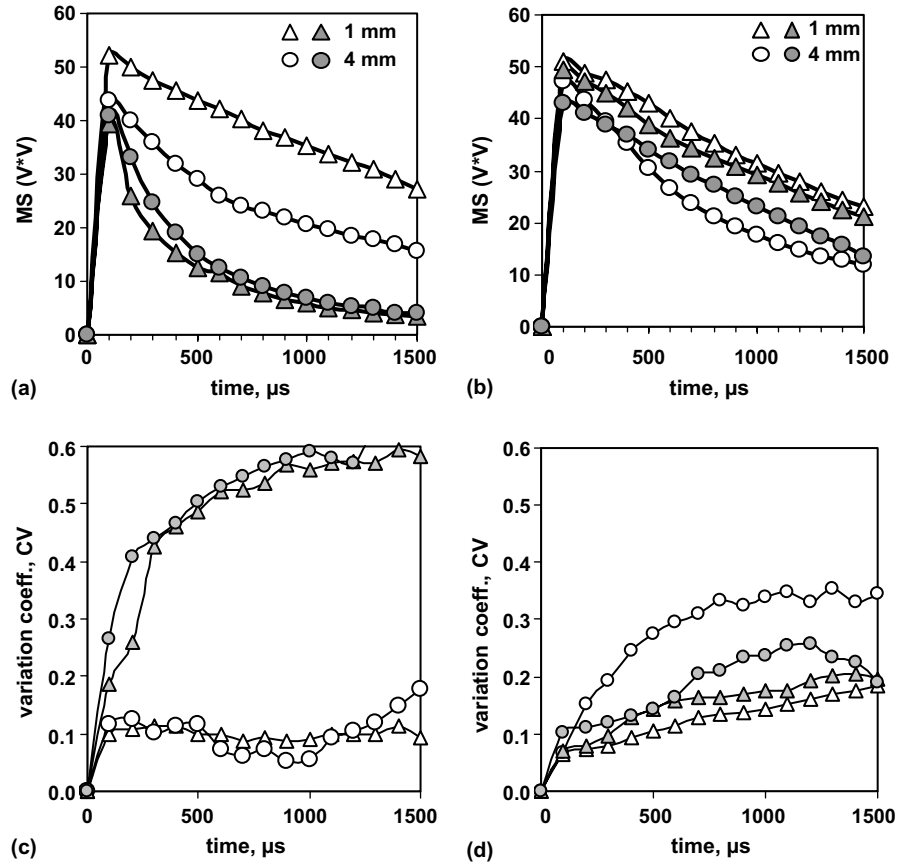


Fig. 6. The effect of filler admixture (△, ○) on the distributions of  $MS(t)$  (upper) and variation coefficient (bottom) for EP-1 (a,c) and PUR (b,d) coatings with thickness of 1 and 4 mm.



without any polymer coating was located between the MS plots for the coatings with maximum adhesion and with zero-adhesion (Fig. 4(a)). The values of variation coefficient, CV, for the both adhesion states were also significantly different (Fig. 4(b)). For maximum adhesion state the CV values for MS(500  $\mu$ s) were small ( $<0.1$ ) and close for all tested coatings, while for “zero-adhesion” state the corresponding CV were higher – CV = 0.5 for EP-2 and PUR and CV = 1.1 for EP-1. The highest difference of the MS value between the maximum adhesion state and the zero adhesion state was observed in the range of 400–700  $\mu$ s in the time domain (Fig. 4(c)). The MS value at 500  $\mu$ s in the time domain was used for further analysis (Fig. 4(d)).

### 5. The effect of chemical composition and geometry of polymer coating

Ultrasonic wave propagation through the PC–CC system was analyzed for each type of floor system and for three thickness values of the polymer coating: 1 mm, 2 mm, and 4 mm. Additionally, the effect of using filler, 0.3–0.7 mm in size, on wave propagation was tested. This kind of filler is commonly added to improve the abrasion resistance of a polymer coating. All tested coatings were applied to obtain the maximum bonding (for a given coating type).

The results obtained indicate that ultrasonic wave propagation through the PC–CC system is affected by the type of resin binder, the presence of coarse filler, and the thickness of the polymer coating. As the coating thickness increased, the pulse velocity (Fig. 5(a)) and MS value (Fig. 5(b)) decreased and the CV value increased (Fig. 5(c)). The largest changes were observed for the EP-1 and PUR floor types. In the case of the EP-2 coating, the effect of coating thickness was less significant. The addition of the filler (Fig. 6) caused the decrease of the MS value and the increase of CV value for the EP-1 coating, while for the PUR system this effect was not significant. The results indicate that for proper ultrasonic evaluation of adhesion in a PC–CC system, a suitable reference curve should be determined, with coating composition and thickness considered as factors affecting the propagation and attenuation of ultrasonic waves. The value of the coating thickness can be verified using the echo method.

### 6. Relationship between pull-off strength and ultrasonic parameters

The relationship between the “pull-off” strength and the ultrasonic parameters was tested for two floor system types: EP-1, EP-2 (2 mm thickness). In opposite to compressive strength evaluation with NDT (ISO–strength reference curve) there are not guidelines for development of pull-off strength reference curve. What is more, it is not easy to obtain an uniform and wide distribution of pull-off strength for tested system. In this work the concrete

substrates differed in moisture content, and were prepared with and without primer in order to obtain a continuous range of adhesion in the PC–CC system. Each variant of polymer floor was prepared on a concrete plate of dimension 300 mm  $\times$  300 mm  $\times$  50 mm. The ultrasonic measurements were tested at fifteen different places for each floor specimen in order to determine average values. The pull-off test was measured at five different locations. Additionally, the results obtained [19] for vinyl-ester coating (3 mm thickness) and PUR (0.7 mm thickness) were taken into account for statistical analysis of relationship between the “pull-off” strength and the ultrasonic parameters.

In the case of the EP-1 and EP-2 coatings, the pulse velocity was approximately constant for a wide range of

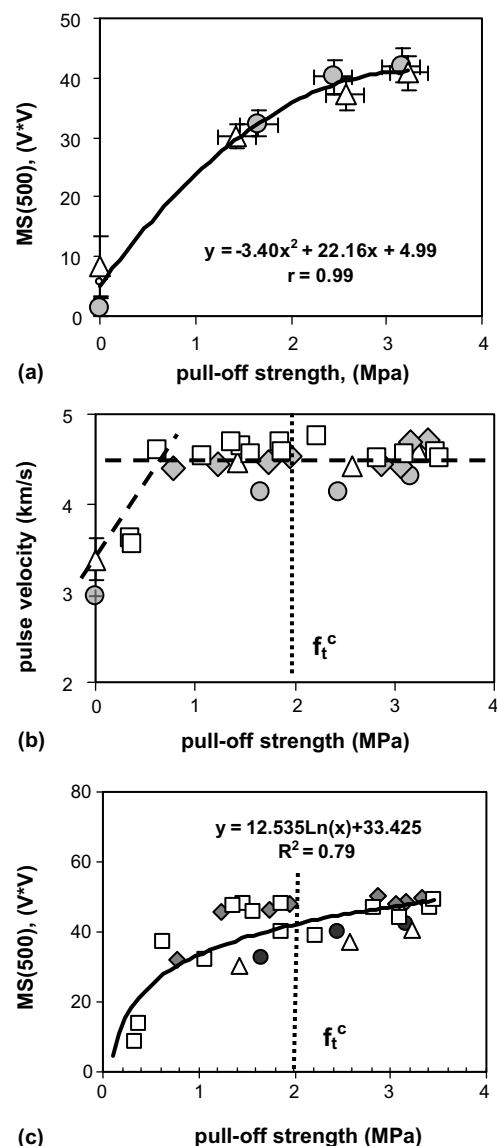


Fig. 7. MS(500  $\mu$ s) value vs. the pull-off strength for EP-1 and EP-2 coatings of 2 mm thickness. The pulse velocity (b) and MS(500  $\mu$ s) value (c) vs. the pull-off strength or four coatings differ in composition and thickness: water-dispersed epoxy, EP-1 – 2 mm ( $\bullet$ ), solvent epoxy, EP-2 – 2 mm ( $\Delta$ ), polyurethane – 1 mm ( $\square$ ) and vinyl-ester 4 mm ( $\diamond$ );  $f_t^c$  – nominal tensile strength of concrete substrate.

the pull-off strength, 1–3.5 MPa, and its value was significantly higher in comparison with the velocity measured for the “zero-adhesion” state. For both epoxy coatings, changes the MS(500  $\mu$ s) with the pull-off strength were similar. Taking the results for the both coatings the relation for above parameters was characterized by a high correlation coefficient value:  $r > 0.99$  (Fig. 7(a)). The same changes in the pulse velocity (Fig. 7(b)) and the MS(500  $\mu$ s) (Fig. 7(c)) were obtained taking into account the results for four coating differ in resin type and thickness. The relationship of MS(500  $\mu$ s) vs. the pull-off strength can be described logarithmic function (best fitting regression function) with relatively high determination coefficient:  $R^2 = 0.79$ . This indicates that the increase of the value of MS(500  $\mu$ s) can be explained at least in 75% by the increase of the bond strength. For all coating systems tested, adhesive or mainly adhesive failure modes were observed up to approximately 2.0 MPa. As the pull-off strength increased above this point, the failure mode approached pure cohesive failure in the concrete substrate.

The results obtained for the both epoxy coatings were also analyzed using ANOVA procedure [28] with post hoc Sheffe test (conservative test) at the significance level  $\alpha = 0.05$ . The pull-off strength levels: “0–1”, “1–2”, “2–3” and “>3” MPa were selected as the group variables. In the case of EP-1 coating (Fig. 8(a)) the differences in the mean value of MS(500  $\mu$ s) were statistically significant up to 2 MPa. The distribution of MS(500  $\mu$ s) values corresponded to the pull-off strength above 2 MPa had the same mean value with probability higher than 95%. In the case of EP-2 coating the pull-off levels “1–2” and “2–3” had the same mean value between those levels and significantly different mean value of the MS(500  $\mu$ s) from the levels “0–1”, “>3” (Fig. 8(b)). The MANOVA analysis showed also statistically significant interaction effect between binder type and pull-off strength level.

On the base obtained results it can be concluded that relationships between the pull-off strength and the parameters of ultrasonic wave propagation are valid for only a limited range of pull-off strength. This is a result of the nat-

ure of the pull-off test – maximum bond strength corresponds to the tensile strength of the concrete substrate. Fig. 7(a) and (c) show that the MS(500  $\mu$ s) value attained a maximum when the pull-off strength achieved a maximum value close to the tensile strength of the concrete substrate.

## 7. Ultrasonic detection of defects in PC–CC system

The possible ultrasonic detection of defects in a PC–CC system was tested for the EP-1 and EP-2 epoxy coatings that had artificial defects introduced at the PC–CC interface. Artificial defects were prepared in the form of polyethylene thin film sheets of different shape and dimensions, which were put on the concrete substrate under the top coating. The EP-1 and EP-2 epoxy coatings (2 mm thick) were layered on concrete substrates of dimensions 300 mm  $\times$  300 mm  $\times$  50 mm, and the specimens were divided into a regular grid. At the nodes of this grid, eight ultrasonic measurements were carried out in directions inclined at 45° to each other. In selected nodes, the pull-off strength was also determined. The results of the ultrasonic measurements were statistically analyzed. The least squares method of estimation was used to determine the distribution of the MS(500  $\mu$ s) value for the EP-1 and EP-2 coatings (Fig. 9(a) and (b), respectively). A low pull-off strength was obtained only when a steel dolly was placed completely in the defect area (Fig. 9(a) and (b) – white circles). In general, the experimental pull off strength was higher than 2 MPa, even at the points where steel dollies were partially placed in the defect area (Fig. 9(a) and (b) – white dots). This can lead to overestimation of the pull-off strength. Contrary to the experimental results of the pull-off strength, the MS value distribution properly indicated the presence of defects. The MS(500  $\mu$ s) value decreased at defect sites and the corresponding coefficient of variation for the MS value increased (Fig. 9(c) and (d)).

On the basis of the results obtained, the following steps for the non-destructive mapping of adhesion between

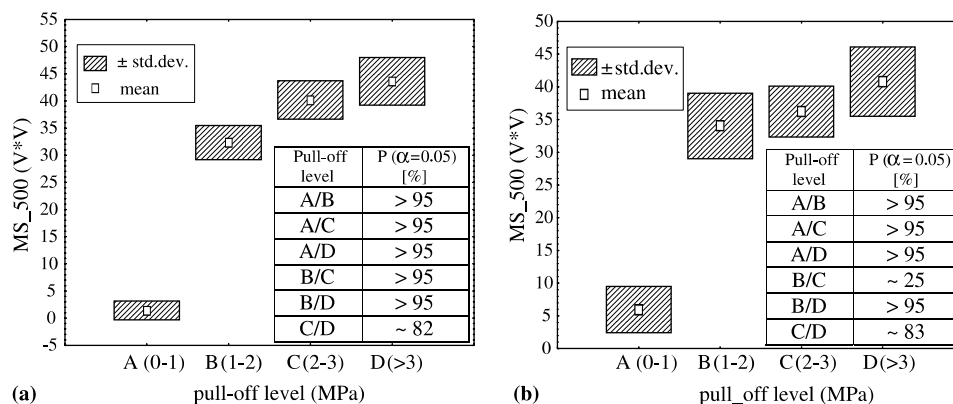


Fig. 8. Parameters of MS(500  $\mu$ s) distribution and results of ANOVA analysis for two epoxy coating EP-1 (a) and EP-2 (b);  $P$  ( $\alpha = 0.05$ ) – probability (in per cent) at  $\alpha = 0.05$  that two pull-off strength ranges have different the mean value of MS(500  $\mu$ s).



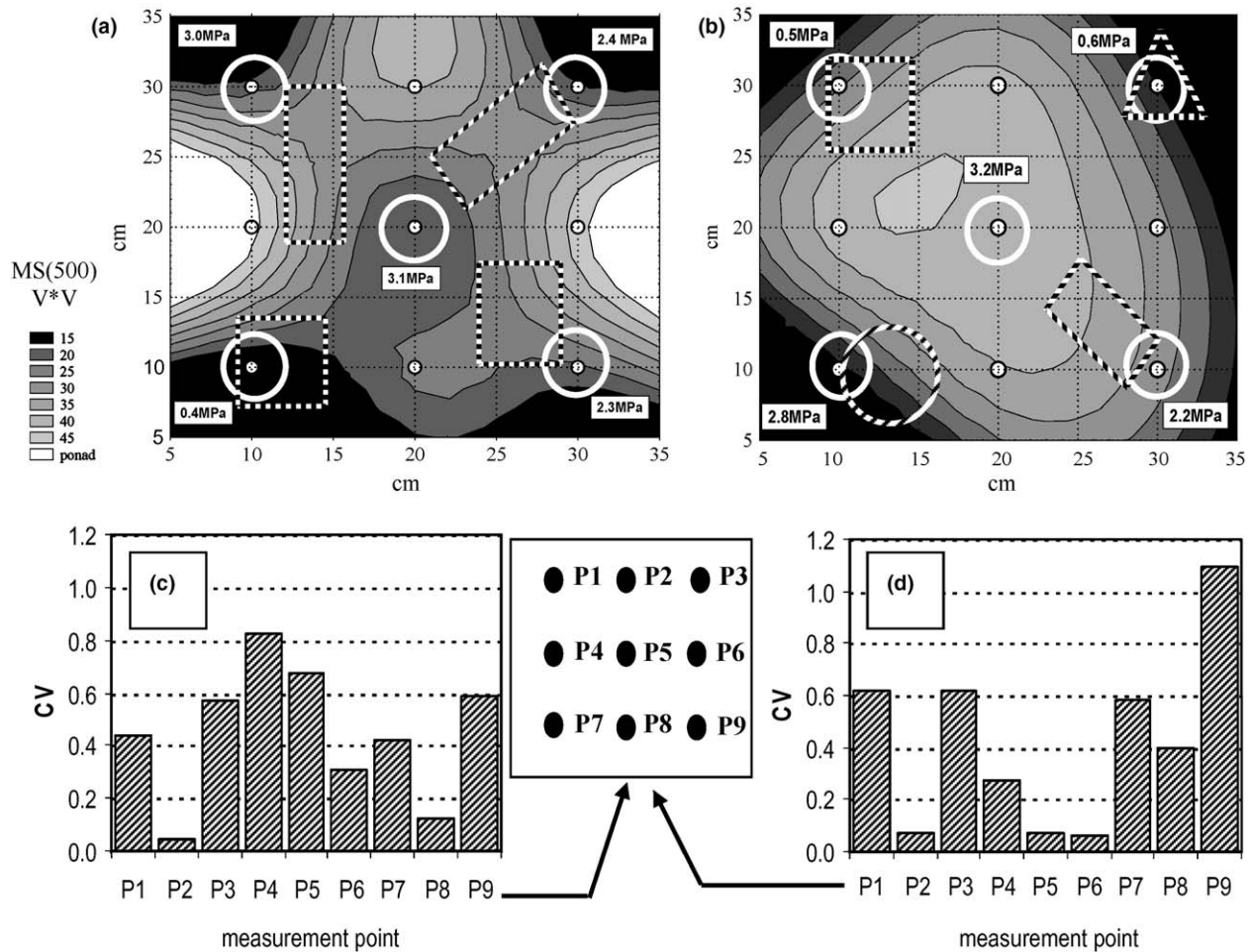


Fig. 9. Results of the ultrasonic mapping of the adhesion for (a) EP-1 and (b) EP-2 coatings with nominal thickness of 2 mm and artificial defects (dotted line contour); (c and d) corresponding variation coefficient, CV in measurement points P1–P9. In white rectangles the experimental values of the pull-off strength (measured in the solid white line circle); white points stand for the points of ultrasonic measurements.

polymer coating and concrete substrate (Fig. 10) can be formulated:

- selection of measurement points grid,
- experimental determination of  $MS(500 \mu s)^{EXP}$  value in the given points (at least four measurements at each point) with indirect pulse velocity methods,

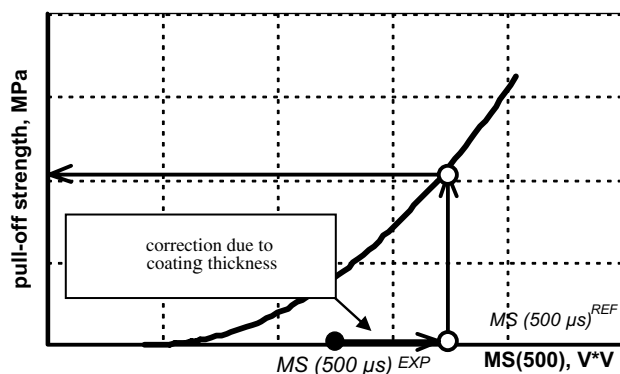


Fig. 10. General scheme of ultrasonic evaluation of the adhesion between polymer coating and concrete substrate (description in text).

- evaluation of the coating thickness with ultrasonic-echo method,
- recalculation of  $MS(500 \mu s)^{EXP}$  into  $MS(500 \mu s)^{REF}$  due to the variation in coating thickness (e.g., on the basis of a suitable regression function or graph  $MS(500 \mu s)$  value vs. coating thickness),
- estimation of adhesion strength from the reference curve: pull-off strength vs.  $MS(500 \mu s)^{REF}$ ,
- determination of the  $MS(500 \mu s)^{REF}$  value distribution.

The ultrasonic methods belong to “point” NDT methods. It means that measurements should be carried out in using a grid of measurement points. For example, in ACI Concrete Repair Manual [9] grid:  $1.0 \times 1.5$  m is recommended. The coating samples, tested in this work, can be treated as a node of this kind of grid. On the basis of parameters obtained for all nodes and using procedures of statistical estimation the adhesion distribution (mapping) can be determined. The high value of the coefficient of variation for  $MS(500 \mu s)$  can be treated as the additional confirmation of the poor adhesion in tested place.

## 8. Conclusions

The obtained results justify the possibility of application of ultrasonic methods for non-destructive evaluation of adhesion between polymer coating and concrete substrate as well as for the defect detection. For purpose of non-destructive adhesion evaluation the suitable reference adhesion curve (similar to the ISO-curve for concrete compressive strength assessment) should be developed for the given type of polymer coating. The material composition, e.g. kind and content of binder and filler as well as content of porous should be taken into account to increase accuracy of the regression function describing the reference adhesion curve.

Simultaneously, the obtained results indicate the need for further investigation towards ultrasonic signal analysis, which will decrease an effect of the heterogeneity of polymer coating and concrete substrate.

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