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Electrochemical investigations of conductive coatings applied as anodes in cathodic protection of reinforced concrete

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Abstract

Results of electrochemical measurements of conductive coatings, based on the mixing of pigmentary graphite in a polymer matrix are presented in this work. Electrochemical parameters are determined for the investigated coatings during long-term anodic polarisation on reinforced concrete. Based on impedance measurements the electrochemical parameters of conducting coatings are calculated. It is shown that the investigated coatings can be used in cathodic protection of reinforced concrete. The investigations show that the optimum graphite contents in coatings used for protection of concrete should be in the range from 40% to 45%.

Keywords: Conductive coatings; Cathodic protection; Impedance; Electrochemistry

1. Introduction

Coating protection is one of the most popular method of anticorrosion protection of reinforced concrete structures. In this case the aim of the coating is to ensure a tight barrier from access of moisture and diffusion of aggressive ions such as: chlorides, sulphates, nitrates to the porous structure of concrete [1–3]. Protective coatings also ensure protection from access of aggressive gases such as carbon dioxide, sulphur dioxide etc. [2,3]. Due to direct contact with the surrounding environment protective coatings applied on concrete surfaces should be characterised by special physical and chemical properties such as: resistance to water and moisture, gases present in the atmosphere, water vapour, ultraviolet light and erosion [4]. Most of the presented requirements are fulfilled by coatings based on epoxide resin binder [5]. Investigations performed by Flounders et al. [6] indicate, however, that the epoxide coating applied as anticorrosion protection of concrete should be made up of many layers with application of differentiated chemical and physical properties of each layer. Application of protective coatings directly on reinforcing bars is another form of anticorrosion protection of reinforced concrete [7–9]. Investigations of Kilareski and Walter [10] confirm that this type of solution is the most effective form of protection of newly constructed bridge structures [10]. Due to difficulties in painting of openwork reinforcement, reinforcing bars are painted with powder paints [11]. Application of coating protection does not always ensure full anticorrosion protection. Another alternative method is the application of impressed current or sacrifical cathodic protection [12,13]. Investigations of Brousseau et al. [14] have shown that the good protective properties are obtained by using zinc metal spraying, in contradistinction to aluminium coatings which application of which does not bring the expected results. Application of titanium as anode is another form of metallisation protection [15]. Application of titanium extends the failure-free time of impressed current protection systems due to very good mechanical properties of the anode [16]. The effectiveness of metallisation coating protection, as shown by Covino et al. [17], are connected with parameters of metal spray coating connected with the outflow rate, the particle size, spraying pressure and physical and electrochemical effects, namely as: processes occurring on the anode–concrete phase boundary and the water penetration rate.

Cathodic protection through impressed current is another method. In this case the anode should be made of

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an insoluble material and be connected to an external power supply [18]. The most typical form is installation of anodes on the concrete surface, covered by a thin surface layer of the concrete [18,19]. In such systems anodes can be made of titanium or titanium covered with active oxides or platinum. Due to very good electrochemical properties of these materials the current densities can be very high [20], but the disadvantage is a relatively high anode installation cost, connected with the application of the external concrete coating layer. One of the methods of avoiding some flaws of classic concrete cathodic protection solutions is modification of the concrete structure with conductive components. Xuli and Chung [21] noticed that addition of carbon fibers to the concrete structure distinctly limits its resistivity. On the other hand, according to Jiangyuan and Chung [21], addition of carbon fibers to the concrete decreases the risk of occurrence of overprotected and unprotected places [22]. According to Chung introduction of carbon fibers into the concrete [23], apart from improving electric properties, increases thermal resistance and mechanical parameters of the concrete. In spite of many improvements in methods of anode installation, the process still requires relatively expensive construction works connected with drilling of holes and removing of the external concrete layer. Problems can also be caused by likely damages during mounting of anodes. The different approach to anodic systems makes possible simultaneous realisation of coating and cathodic protection. It is a problem not encountered up till now in the world literature.

The aim of the work is to perform of electrochemical measurements on specially prepared conductive coatings in order to evaluate their suitability as anodes in cathodic protection of reinforced concrete structures. The main aim was to choose an optimum content of conducting component in the epoxide coating, to ensure correct anode working parameters and perform laboratory cathodic protection of reinforced concrete with the use of conducting coatings as anodes.

2. Measurement methodology

Coating samples for investigations were made from an epoxide resin base of average molecular mass. The graphite was introduced to epoxide resin (25–60% contents by weight). The coating was applied with a paintbrush in four layers of 300 μm total thickness on ST3 carbon steel cleaned by sanding and on reinforced concrete samples (the free w/c ratio was 0.4). Measurements were performed each time on five samples for each graphite contents.

Application of coatings on steel samples was performed in order to determine their account of resistance. Coating resistance was assessed by measuring the impedance in a two-electrode system with application of a mercury electrode. The geometric area of the electrode was equal to 1.8 cm^2. The measurement set-up was made up of the SCHLUMBERGER SI 1255 transmittance analyser with an ATLAS 9181 attachment for high-impedance measurements.

Concrete specimens were covered with conducting coatings of 30–50% graphite contents for performing laboratory cathodic protection of concrete reinforcement. The coating was applied on concrete, by a paint brush in four layers of 300 μm total thickness. Concrete samples were in a form of cylinder: 1.5 cm in diameter and 6 cm in height. The diameter of the reinforcing bar was equal to 0.6 cm, and the height was 5 cm. The lower part of reinforcement, 1 cm in length, was covered with a casing in order to form an artificial crevice. Physicochemical investigations of coatings included exposure of unprotected samples in a 1% NaCl environment, hardness measurement by the Shore method and adhesion of coatings to the base surface. Fig. 1 describes measurement cell of cathodic protection reinforced concrete.

The reinforcement cathodic protection system was made up of the cathode—steel reinforcement, the anode—a conducting coating applied on the surface of the concrete cylinder. Concrete samples were placed in 2 dm^3 vessels containing a solution of NaCl 1% at a temperature of 16–18 °C (with no stirring). The electric terminals were connected to the steel reinforcement and to the conducting coating on the concrete surface. The computer-controlled galvanostat–potentiostat ATLAS 9232 (ATLAS-SOLLICH, Poland) was used for cathodic protection system. The electrolytic environment was specially chosen in order to simulate an resistivity identical to that of the sea environment (about 10 Ω m). Before starting cathodic protection the samples were placed in a solution of NaCl for 90 days. After this time potential and impedance measurements of concrete reinforcement had shown that the electrolyte penetrated to the concrete structure. In order to ensure identical anode (conducting coating) working conditions the current cathodic protection criterion was applied. In this case through each electrode the same charge will flow, independently of the environment resistance or electrode resistance. Working current values applied in cathodic protection of reinforced concrete with conducting coatings are up to 10 mA/m^2. For determination of the electrochemical characteristics of conducting coatings in a relatively short time the current density was 45 mA/m^2. Cathodic protection was performed during 72 days. In the same environment painted concrete samples with no protection were placed for determination of weight loss of reinforced rebar. During exposure, potential measurements were made on the coatings using a silver - silver chloride reference electrode. Impedance measurements were also performed on the conducting coatings every 18 days using a three-electrode system (auxiliary

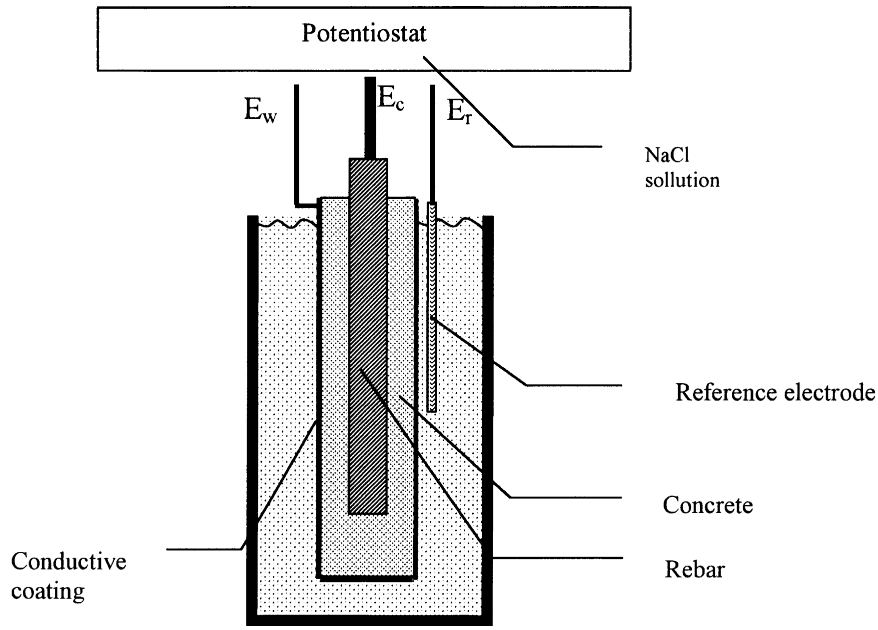

Fig. 1. Measurement cell of cathodic protection reinforced concrete.

electrode in the form of a platinum mesh placed in the vessel together with the reinforced concrete sample covered with a conducting coating). The set-up for impedance measurements of conducting coatings was made up of an ATLAS 9121 transmittance analyser, generator, an Atlas 9131 analogue–digital converter and a PC computer. Measurements were performed in the potentiostatic mode in the 100 kHz to 0.5 Hz frequency range. After finishing exposure visual inspection of investigated reinforced concrete samples was performed.

Table 1
Results of adhesion measurements of conducting coatings as a function of the graphite contents

Graphite content [%]	Adhesion of coating before exposure [N/mm^2]	Adhesion of coating after exposure [N/mm^2]
30	4.1	3.6
35	3.5	3.8
38	3.8	4.1
40	3.4	3.3
45	3.7	3.7
50	3.5	3.4

3. Results and discussion

3.1. Physicochemical investigations of coatings

Physicochemical investigations of coatings were performed attending to the NACE RP0281 standard covering atmospheric exposure of electroconducting coatings [24]. Laboratory investigations of reinforced samples were performed in 1% NaCl for a period of 90 days.

In spite of application of significantly more drastic exposure conditions no occurrence was stated of coating defects according to RP0281.

Physicochemical investigations of conducting coatings on concrete were performed basing on NACE standard no. RP0591 covering exposure of coatings applied on the concrete structure in the atmosphere [25]. Also in this case the immersion test was applied. Table 1 describe results of adhesion measurements of the coatings depending on the graphite contents.

Results presented in Table 1 show that application of graphite contents in the 30–50% range does not affect the adhesion of the coating to the base before, as well as after exposure in 1% NaCl. Exposure of the specimens to 1% NaCl also does not influence the bond strength.

3.2. Electrochemical investigations of electric properties of coatings

Impedance investigations in a two-electrode system were performed in order to observe the effect of graphite contents on the coating resistance. Application of a mercury electrode for measurements allows obtaining direct contact of the electrode with the coating surface. The impedance measurement allows recording the coating resistance on the basis of the obtained impedance spectrum. Fig. 2 describes the impedance spectra for a conducting coating in a two-electrode system determined for different graphite contents.

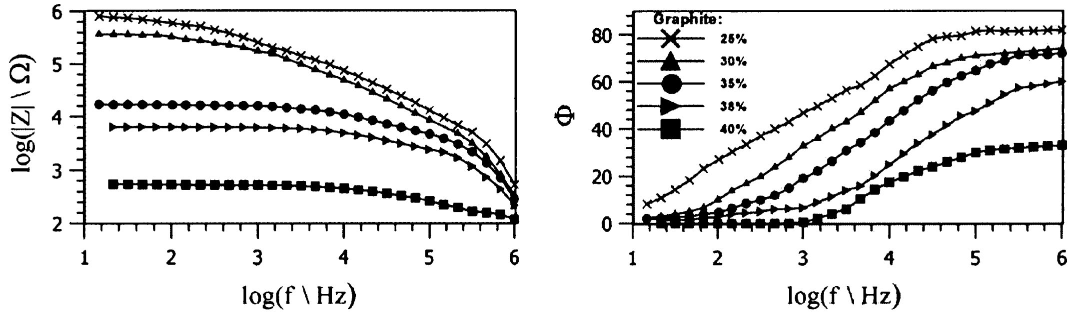

Fig. 2. Impedance spectra of steel coated with coatings containing variable quantities of graphite (contents by weight).

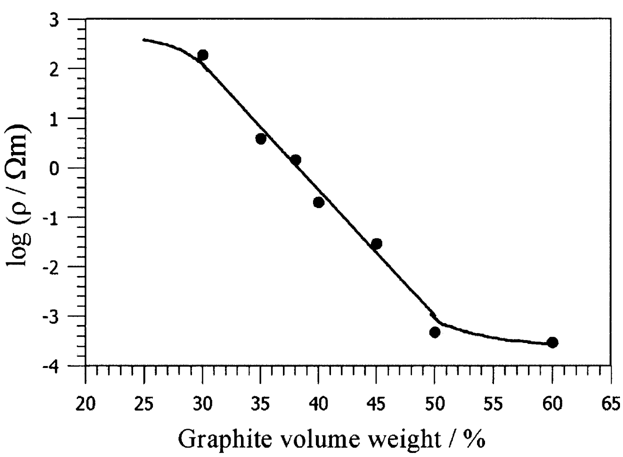

Fig. 3. Graph of coating resistivity as a function of graphite contents in percent.

Resistance values obtained from the impedance spectra are several orders lower in relation to the resistance of epoxide resin ($\sim 10^{10}$ Ω) used as the binder of investigated coatings. Analysis of the impedance spectra was performed with Boukamp software. Fig. 3 presents the resistivity values of conducting coatings in the function of graphite contents in the coating. The presented dependency has been formulated on the basis of five results for each graphite contents.

As expected, the coating resistivity decreases with the graphite content. Two intersection points can be seen on the graph for graphite contents of ≈30% and 50%. For 30–45% graphite contents a significant resistivity decrease occurs. The observed coating resistivity graph is in accordance with the percolation theory [26]. The percolation theory allows estimation of the conductivity in function of the conducting component contents in relation to the mass of the whole composite. Factors affecting conductivity are: size of conducting and non-conducting particles in the composite, their conductivity and spatial structure of the material [27]. When the content of the conducting component in the coating is relatively low (below 30% of graphite) conducting pigment particles are not connected with each other and hence conductivity of such material is very low. With increase of conducting component contents filling in of the spatial network with its particles increases. At higher contents conducting particles conductivity of composite material significantly increases. Above 50% of graphite in coatings additional conducting component contents do not significantly affect the resistance decrease of composite [27].

In the case of conducting coatings it is important for the conducting component contents to be higher than the percolation threshold, as anode material electric properties can significantly deteriorate as the result of consumption of conducting component due to occurring anodic reactions [27,28].

Based on the performed measurements is possible to state that coatings containing over 38% of graphite exhibit satisfactory resistivity properties. In the case of resistivities greater than 5 Ω m the decrease in protective current during realisation of cathodic protection would be too high [28]. In this case one should take into account also the economic factor connected with use of greater power of the cathodic protection station and faster consumption of the coating anodic material. Measurements of conducting composites on an ethylene–propylene terpolymer base had shown that samples characterised by greater resistance were distinguished by poor stability as anodes in cathodic protection [28].

3.3. Electrochemical investigations of coatings under current load

3.3.1. Impedance measurements

During application of cathodic protection to the concrete reinforcement impedance measurements were performed on the conducting coatings. The impedance spectra determined for each graphite contents in the coating are presented on graphs.

Fig. 4 presents Nyquist impedance spectra of a coating containing 50% graphite applied on concrete measured during cathodic protection of concrete reinforcement.

Impedance investigations were performed in order to estimate the resistance of the conducting coatings. In

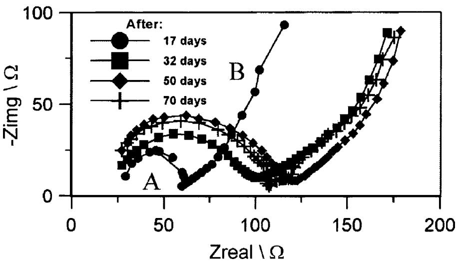

Fig. 4. Impedance spectra of concrete specimen coated with a coating containing 50% graphite during cathodic protection of concrete reinforcement.

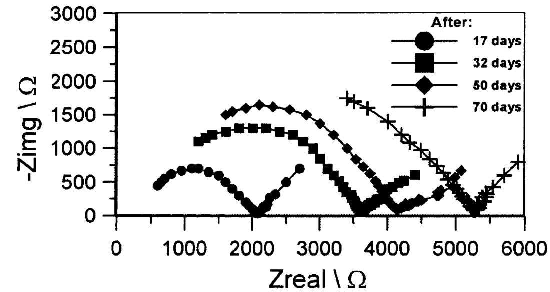

Fig. 5. Impedance spectra of a coating containing 35% of a graphite during cathodic protection of concrete reinforcement.

order to explain the physical sense of an impedance spectrum of a conducting coating the measurement after 17 days exposure was analysed. Two characteristic fragments can be distinguished designated with letters A and B in Fig. 4. In the high-frequency part a semi-circle is seen connected mainly with electric properties of the coating (A). Similar relations in the same frequency range are observed in impedance spectra of coating material in a two-electrode system (Fig. 2).

The low-frequency part (B) of the impedance spectrum is connected with anodic processes, which are the result of oxygen and chlorine evolution (charge transfer process) [29]. Both these processes are several step reactions, complicating analysis of obtained impedance spectra in the low-frequency range.

Based on the analysis of impedance spectra presented in Fig. 4, is possible to state that resistance of the coating changes during application of cathodic protection. In the initial stage of operation the coating resistance increases, this being especially visible for the first 17 days of exposure. After 32 days the increase of coating resistance is minimal. In spite of the fact that during application of cathodic protection combustion of graphite occurs [28], the contents of this component are so high that no distinct deterioration of electric properties of the coating occurs. In the case of such high graphite contents in the coating overpigmentation can occur which is characterised by poor barrier properties. The problem of coating overpigmentation was widely discussed by Bierwagen et al. [30]. During cathodic protection of reinforced concrete the poor barrier properties of the coating affect the rate of penetration of aggressive electrolyte to its structure. This phenomenon is very disadvantageous, however, the increase of porosity of conducting coating leads to an increase of the coating surface, this being connected with an increase of the anode area. In this case the current density flowing through a geometric unit of the anode surface will be smaller. In the case of cathodic protection of underground structures, in order to increase the anode working area, porous backfills are used from a conducting carbon material. Taking these aspects into account the coating containing 50% of graphite due to its porosity is characterised by very good electric properties, advantageously affecting working parameters of the cathodic protection system.

In Fig. 5 examples have been presented of impedance spectra of coatings containing 35% of graphite applied on concrete, working as anodes in cathodic protection of concrete reinforcement.

Based on the analysis of impedance spectra presented in Fig. 5 is possible to state that the coating containing 35% graphite is characterised by a significantly higher resistance than the coating containing 50% graphite. In the case of a coating containing 35% graphite the conducting component contents are much lower, unequivocally showing that barrier properties of this coating are much better. Electric resistance of coating containing 35% of graphite increases as the result of combustion of conducting component [31]. Relatively good barrier properties of the coating prevent access of electrolyte to the coating structure containing greater quantities of graphite than in the surface layer. Hence, during the operation of the cathodic protection system a continuous increase of coating resistance can be observed.

In Fig. 6 depicts the impedance spectra of the coating containing 30% graphite applied on concrete, during application of cathodic protection.

By analysing the impedance spectra of the coatings containing 30% graphite during cathodic protection of concrete reinforcement it may be stated that the resistance of this coating is very high. It is connected with initial the electric properties of this coating, as graphite contents are very low, close to the percolation threshold value. Hence, the number of conducting paths in the coating structure is very low. If we add to such disadvantageous parameters, the effect of the graphite combustion as the result of anodic processes, the resistance of the coating can be significantly increased. The resistance values of the coatings containing 30% graphite increase ten-fold after 32 days (Fig. 6), confirming initial assumptions. After 52 days of cathodic protection the resistance of the coating containing 30% graphite increased by such a degree that in order to ensure the required current density ($i = 45$ mA/m^2), cathodic protection was interrupted.

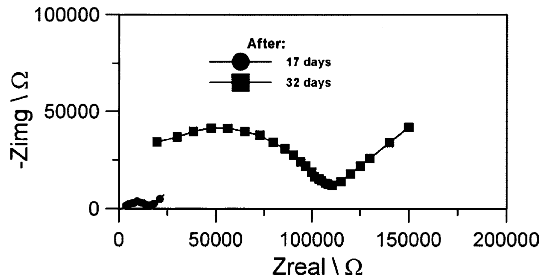

Fig. 6. Impedance spectra of a coating containing 30% graphite during cathodic protection of concrete reinforcement.

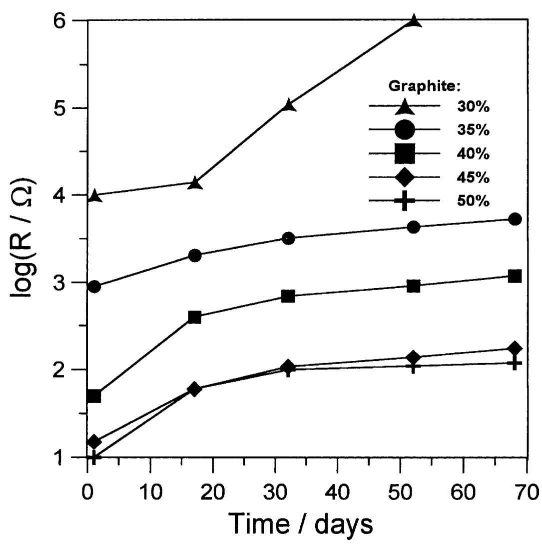

Fig. 7. Graph of resistance changes of conducting coatings as a function of the exposure time for graphite contents in coating.

Fig. 7 depicts the resistance changes of all conducting coatings as function of the cathodic protection exposure time.

During cathodic protection the coating resistance increases as the result of graphite combustion during the anodic processes. The highest resistance increase can be observed for the first 17 days of operation, especially for coatings containing large graphite quantities. After 17 days of exposure an increase in the coating resistance is not so significant, except for the coating containing 30% graphite in which resistance distinctly increases with the exposure time.

3.3.2. Measurements of potential of conductive coatings

Fig. 8 presents the conducting coating potential changes in the function of cathodic system operation time.

Impedance measurement results are reflected in electrode potential measurements. The higher the graphite content in the coating, the lower the measured electrode potential values (Fig. 8). The coating potential value measured during operation of cathodic protection in-

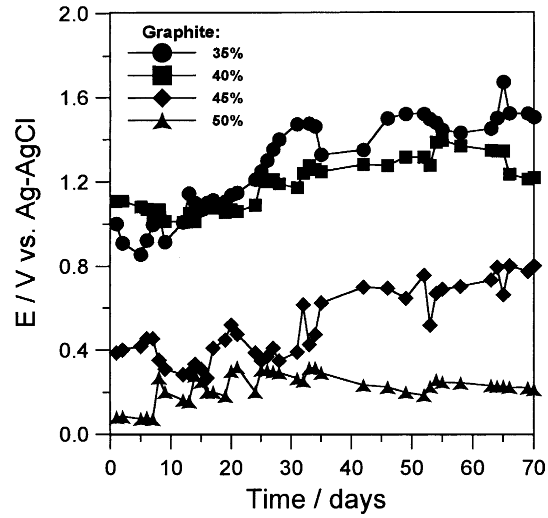

Fig. 8. Graph of conducting coating potential changes as a function of reinforced concrete cathodic system operation time for graphite contents in coating.

corporates the value of the potential ohmic drop. Hence, coatings containing greater graphite quantities are characterised by lower resistances, reflected by lower measured potentials. During cathodic protection the coating potential increases due to an increase of the coating resistance except for the sample containing 50% of graphite. The potential of this sample decrease with the polarisation time due to an increase of the surface of the electrode caused by the anodic reaction. In Fig. 8 the potential measurements for the coatings containing 30% graphite are not present due to large potential value difference. In the first day of cathodic protection system operation the potential of coating containing 30% graphite was equal to +5 V, during 17 days it reached +45 V while after 52 days it exceeded +100 V. Application of such voltages is not in accordance with safety regulations, hence after 52 days of polarisation the system was turned off.

At the end of cathodic protection tests, evaluation was performed in order to observe the state of the conducting coating and steel reinforcement in concrete. Samples of unprotected concrete were also analysed to determine the effectiveness of cathodic protection. In the case of coatings applied on protected as well as unprotected samples no deterioration of mechanical properties of coatings was stated. The hardness and adhesion of coatings to the base did not change after completion of exposure in relation to initial conditions.

3.3.3. Gravimetric measurements

Presence of corrosion products was observed on the surface of reinforcement. Corrosion products were removed using Clark's solution. Table 2 presents mass decrements of reinforcing steel samples.

Table 2
Results of mass decrement of reinforcing steel without cathodic protection

No. of sample	Mass decrement Δm [g]
1	0.087
2	0.094
3	0.073
4	0.086
5	0.092

Table 3
Results of concrete steel reinforcement samples after cathodic protection in the function of graphite contents in the coating

Graphite contents in coating (%)	Mass decrement Δm [g]
35	0.001
40	0.003
45	0.002
50	0.001

The investigation of the steel concrete reinforcement has shown that crevice corrosion takes place. Visible corrosion products were found under a specially prepared crevice formed inside the concrete structure. Also it was noted that during the experiment general corrosion of concrete reinforcement did not take place.

Investigations of the coating structure containing 35–50% graphite have not shown concrete cracks or damages after finishing exposure. Investigation of steel reinforcement after removal of the concrete has not shown the presence of corrosion products. Mass decrements of steel reinforcement of concrete samples have been presented in Table 3.

Mass decrement results are overstated due to the sample etching process before weighing.

Examination of the conducting coating containing 30% graphite shows significant surface damage after completing cathodic protection. Numerous blisters were stated, the coating containing 30% of graphite lost its barrier properties.

The proposed conducting coatings based on the graphite and epoxide binder can be successfully used in cathodic protection systems of reinforcement concrete.

4. Summary

Results were presented for a conducting coating fulfilling the role of anode in an impressed current cathodic protection of reinforced concrete structures. This unconventional protection method requires electroconducting coatings of special electric and electrochemical properties. The following conclusions were drawn on the basis of performed electrochemical investigations of conducting coatings:

The electrochemical properties of the coatings depend on the contents and type of graphite. Coatings containing the highest graphite quantities (45–50%) are characterised by low resistances and potential stability during long-term anodic polarisation. In spite of the fact that in other exposure measurements occurrence of porosity was stated already after two days of polarisation, investigations of cathodic protection of concrete reinforcement did not confirm a negative effect of coating porosity on protection process parameters [32]. Therefore coatings with such high graphite contents should be submitted to more detailed investigations in order to explain the effect of their porosity on the effectiveness of corrosion protection of reinforced concrete.

For coatings with smaller graphite contents (below 35%) the resistance sharply increases during anodic polarisation. Also occurrence of blisters is visible on the coating surface. Performed investigations have shown that the optimum graphite contents in coatings used in protection of concrete should be in a range between 40 to 45%, as these coatings are characterised by low potentials and relatively low resistances.

The results show that conducting coatings can fulfil the role of anodes in cathodic protection of reinforced concrete structures. The coating binder based on epoxide resin allows maintaining the required barrier properties during anode operation. Application of conducting coatings allows maintaining of anode electric parameters under a current load equal to 20 mA/m^2. Good protection parameters connected with advantageous protective current distribution can be obtained due to a large conducting coating surface allowing additional protection of places hard to reach. Conducting coatings, contrary to classic protection methods, are easy to renovate, allowing long-term trouble-free anticorrosion protection. Hence, application of conducting coatings can be an alternative in relation to classic cathodic protection system solutions [33].

Improvement of electric and electrochemical properties can be obtained by changing the of coating binder to one more resistant to the aggressive anode working conditions or by modification of graphite with platinum group active oxides. Advantageous parameters were stated of such modification in the case of conducting composites on the ethylene–propylene terpolymer base [28]. Also, it is possible to apply a several-layer coating made up of conducting coatings (internal layer) and non-conducting coatings (external layer)—of even better barrier properties than coatings containing large graphite quantities.

However, many technological problems need to be solved connected with, e.g., the ageing process of coatings and the state of the coating electric connection. Most of these problems will be discussed in future publication when cathodic protection of concrete samples will be finished.

Acknowledgements

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