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Review of the possible application of laboratory testing techniques for corrosion protection of steel structures

Przegląd możliwości zastosowań laboratoryjnych technik badawczych zabezpieczeń antykorozyjnych konstrukcji stalowych

The article includes examples of problems with the corrosion protection of steel structures after a few or several years of operation and presents selected laboratory testing techniques used during technical assessments. The research techniques used included infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). These enabled the following questions to be answered: whether the paint was applied according to the required technology, whether a conversion coating was applied before the powder coating, why a correctly applied zinc coating did not provide sufficient corrosion protection for the fasteners of the steel structure. The paper also details other applications of the presented research methods, not only in the field of performing technical expert reviews, but also in testing systems used in the corrosion protection of steel structures.

Keywords: SEM, EDS, FTIR, testing techniques, paint coating, zinc coating

W artykule opisano przykłady problemów z zabezpieczaniem antykorozyjnym konstrukcji stalowych po kilku lub kilkunastu latach eksploatacji obiektów oraz przedstawiono wybrane laboratoryjne techniki badawcze wykorzystywane podczas wykonywania ekspertyz technicznych. Zastosowano następujące metodyki badawcze: spektroskopię w podczerwieni (FTIR), skaningową mikroskopię elektronową (SEM) oraz spektroskopię promieniowania rentgenowskiego z dyspersją energii (EDS). Pozwoliło to uzyskać informacje, czy zastosowano farbę zgodną z wymaganą technologią, czy naniesiono powłokę konwersyjną przed malowaniem proszkowym, a także dlaczego poprawnie wykonana powłoka cynkowa nie była wystarczającym zabezpieczeniem antykorozyjnym elementów złącznych konstrukcji stalowej. W pracy wyszczególniono również inne możliwości zastosowania przedstawionych metodyk badawczych, nie tylko w zakresie wykonywania ekspertyz technicznych, lecz także w obszarze badań systemów stosowanych w ochronie antykorozyjnej konstrukcji stalowych.

Słowa kluczowe: SEM, EDS, FTIR, techniki badawcze, powłoka malarska, powłoka cynkowa

1. Introduction

1.1. Selected research methodologies and overview of application possibilities

In the case of premature corrosion damage to steel structures or accelerated deterioration of the corrosion protection system in relation to the design objectives, it is necessary to carry out a technical expert review that goes beyond standard field-testing methods. Laboratory testing of corrosion protection systems can also be useful, for example, at the design stage of restoration work on decades-old or historic steel buildings, for which no complete documentation is available and where there is a risk of having to factor in the disposal of coatings containing elements such as lead or chromium, among others.

The advantage of laboratory testing is that there is a wide range of test methods available to answer many questions about the quality of corrosion protection. When defining the scope of a technical expert review, engineers involved in the design of corrosion protection systems are often surprised at how much information can be obtained about a given system through laboratory testing.

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Therefore, this article will introduce some basic test methods and, based on a literature review and specific cases, will discuss the potential of these methods in the field of corrosion protection provided by paint systems, duplex systems (paint on metallic coating) or metallic coatings.

When commissioning laboratory tests, it is important to bear in mind that these are destructive tests, which not only involve the removal of coatings, but also often involve the cutting out of a section of the structure or the component under examination. In addition, the cost of this type of testing is usually determined on a case-by-case basis and is related to the method-specific preparation of samples, the cost of standard samples or consumables and the working time of highly qualified personnel.

This article presents selected capabilities of the Fourier infrared spectrometer and the energy dispersive spectrometer coupled to a scanning electron microscope. Although advanced analytical techniques are mainly used in research and development laboratories and in product quality control departments for corrosion protection applications, they can also support the work of corrosion protection inspectors, especially at the stage of determining the causes of premature corrosion damage in steel structures.

1.2. Fourier transform infrared spectrometry

The primary identification of the type of paints and coatings is carried out by Fourier transform infrared spectrometry (FTIR). It is an analytical methodology for investigating the structure/composition of solids, gases or liquids and uses electromagnetic radiation with wave numbers usually in the range of 4000–400 cm^{-1}. FTIR spectroscopy exploits the phenomenon of absorption of radiation of a given frequency by vibrations of chemical bonds of a given energy. The kinetic energy of a molecule is related to its oscillatory and/or rotational motion. As a result of the selective interaction of a molecule/functional group with infrared radiation (of a given energy), it is possible to change the oscillatory/rotational energy into so-called normal vibrations of a different nature. One can distinguish between stretching (symmetrical and asymmetrical), bending (i.e. scissoring, rocking, wagging, and twisting) and skeletal vibrations. The result of the study is a spectrum, presented in an absorbance or transmittance relationship to the radiation energy expressed by the wave number, on which the position of the peaks corresponds to the absorption bands of the functional groups present in the sample. Almost every substance has its own unique spectrum, especially in the 1300–900 cm^{-1} wave number range, which allows qualitative analysis of the material under study by comparing the spectrum obtained with databases. Quantitative analysis is possible due to the linear relationship of band intensities to the number of functional groups [1]. Performing a computerised Fourier transform allows the signal-to-noise ratio to be improved by analysing multiple signals from successive measurements. Improvements in resolution, sensitivity, and speed of analysis on a Fourier transform spectrometer also result from the replacement of monochromators by interferometers relative to classical dispersive spectrometers. FTIR spectroscopy distinguishes several measurement techniques depending on the geometry of the measurement system: transmission (TS), internal reflection (IRS), attenuated total reflection (ATR/IRS), external reflection (ERS), diffuse reflection (DRS), emission (ES) and photoacoustic (PAS) [2, 3].

Regarding coating-based corrosion protection, infrared spectrometry can be used, for example, to:
- identify resin and other paint components and confirm the composition of paint products with that declared by the manufacturer ('fingerprinting' – identification testing of paints and other construction products) [4, 5];
- reveal the degradation of chemical bonds under the influence of various corrosive factors such as UV radiation, moisture [6];
- determine the degree of cross-linking/curing of thermosetting and UV-cured coatings [5, 7, 8];
- determine the chalking resistance of coatings in relation to the type of resin and the amount and type of paint additives, i.e. pigments, fillers [9];
- conduct research work in the development of new paint additives [10], new coating compositions [11];
- conduct quality control on the production line [12];
- verify paint storage conditions, exceeding of shelf life [13];
- test coatings for water absorption and desorption [14];
- identify sub-film corrosion products, e.g. during blistering of the coating [15].

1.3. Scanning electron microscopy

Scanning electron microscopy (SEM) is a technique for observing the structure or topography of materials [16]. The image is formed by the interaction of a concentrated electron beam with the test sample. The beam of electrons emitted from the cathode is focused by an electromagnetic lens system and then scans the surface of the test sample and interacts with it. With regard to the design of scanning microscopes, the most common types include: high vacuum SEM HV microscopes, e.g. with a tungsten or LaB6 cathode, high vacuum SEM FE field emission microscopes, low vacuum (SEM LV) or variable vacuum (SEM VP) microscopes, and environmental ESEM microscopes, which allow materials to be examined under high temperature conditions.

For imaging, backscattered electrons (BSE/BECs) and secondary electrons (SEs) are used, which are captured by their corresponding detectors. The BSE/BEC observation mode makes it possible to distinguish areas of different chemical composition, while the SE mode is mainly used to study the topography of a sample. The resolving power of electron microscopes depends on the energy of the electron beam controlled by the accelerating voltage. High-resolution scanning microscopes have a resolution of a few nanometres [17].

The imaging capability of a scanning electron microscope in secondary electron SE and backscattered electron BSE/BEC modes, as related to corrosion protection systems (metal, coating or complex duplex), can be used for:
- surface quality control of coatings [18], determination of surface morphology of the anticorrosion system, surface structure of conversion coatings [19];
- determination of the number of coatings in a corrosion protection system [20] and the thickness of the individual coatings [21];
- confirmation of the presence of conversion coatings [22], electrophoretically applied coatings [23];
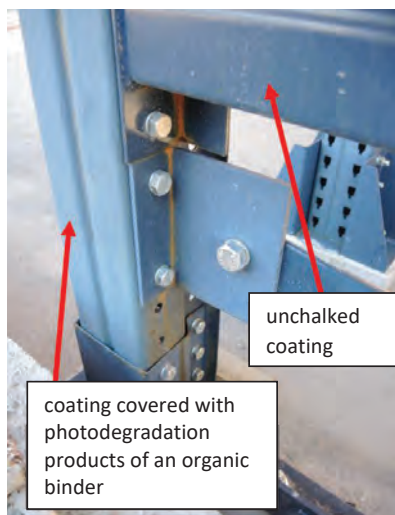
Fig. 1. A fragment of the structure under assessment

Source: photo by Damian Wojda, IBDiM.

Rys. 1. Fragment konstrukcji będącej przedmiotem ekspertyzy technicznej

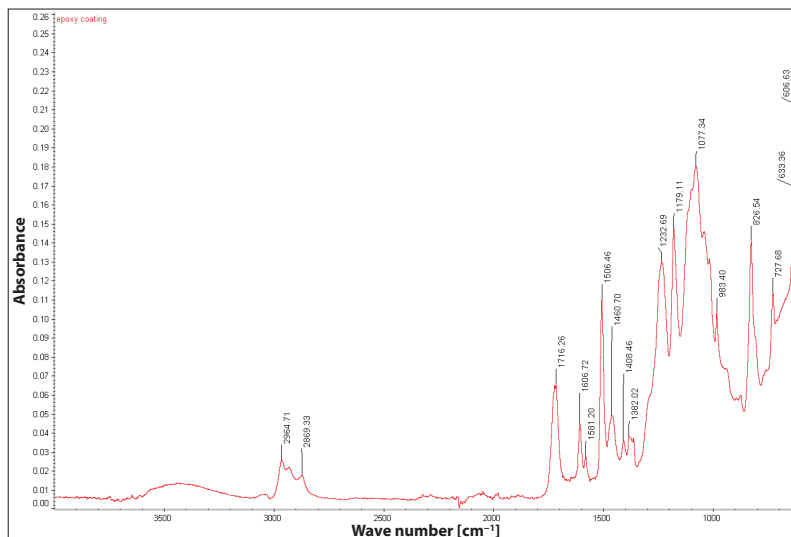Źródło: zdjęcie autorstwa Damiana Wojdy, IBDiM.

Fig. 2. FTIR spectrum of the topcoat: chalked epoxy coating sample after 18 months of UV exposure

Rys. 2. Widmo FTIR powłoki nawierzchniowej: próbka skredowanej powłoki epoksydowej po 18 miesiącach narażenia na promieniowanie UV

— observation of the microstructure of galvanic coatings, e.g. zinc [24], from zinc alloys [25];

— detection of defects in metallic coatings, such as discontinuities, cracks, etc. [26] and in paint coatings, such as porosity, delamination [27], pigment and filler separation [28];

— identification of corrosion/oxidation products of steel or metallic coatings [29];

— testing of new coatings, paint additives [30];

— observation of the steel surface after pretreatment, e.g. shot-blasting [31];

— testing the influence of ageing factors on the condition of the coating [32];

— sealing quality of metallized coatings [33];

— stereological analysis of the precipitates [34] or phases in metallic coatings [35].

1.4. X-ray energy dispersive spectrometry

The interaction of the electron beam with a sample is used in scanning electron microscopy not only for imaging in SE and BSE/BEC modes, but also for chemical composition analysis. For this purpose, the detection of Auger electrons or emitted element-specific X-rays is used [16]. Auger electrons are mainly used in the analysis of the chemical composition of surface layers, while energy dispersive spectrometers (EDS/EDX) or wavelength dispersive spectrometers (WDS) of X-rays are most common. Atom-specific radiation is produced by the emission of photons generated during the interaction of an electron beam with electrons of atoms at different energy levels. EDS and WDS spectrometers analyse the energy or wavelength of the emitted wavelength, respectively. The limitation of this analytical method is therefore the minimum number of orbitals of an atom, EDS and WDS methods are used to detect atoms with an atomic number equal to or higher than 5. Qualitative and quantitative chemical composition analysis by SEM + EDS/WDS can be performed at a given point, in a micro-area, along a line (linear analysis) or indicated on a surface (composition map).

SEM-EDS chemical composition analysis is a useful and frequently used technique in determining the causes of premature corrosion damage in steel structures. In technical expert reviews of corrosion protection systems, this analytical technique can be used to:

— determine the microstructure and chemical composition of metallic coatings and individual phases [36];

— analyse the chemical composition of inclusions, precipitates, or impurities in metallic coatings [37];

— analyse the chemical composition of components and additives in coatings such as pigments, fillers [38];

— determine the presence of pigments/components in coatings subject to specific disposal processes (such as lead, cadmium, antimony compounds) [39];

— analyse the chemical composition of corrosion products (including sub-film corrosion) [40], reveal residual solid contaminants after mechanical surface pretreatment prior to painting [41].

2. Laboratory test results obtained using the presented test methodologies in technical expert reviews

2.1. Determining the cause of chalking of an organic topcoat

One of the objectives of the technical review was to determine the reasons for the chalking of a topcoat of the same RAL colour, found only on certain elements of a coated steel structure, after several months of exposure to UV radiation. Chalking is a process associated with the photodegradation of the organic binders that form the basis of organic coatings. Different types of paint coatings exhibit different levels of resistance to chalking when exposed to UV radiation. This process manifests itself as a change in the colour of the coating, a loss of gloss and the formation of a light chalky dust on the surface of the coating.

Fig. 1 shows a section of the structure, which is the subject of the review. The organic topcoat on the pole is covered with blue pastel dust and exhibits grade 5 chalking according to PN-EN ISO 4628-6 [42], whereas this process was not observed on the beam.
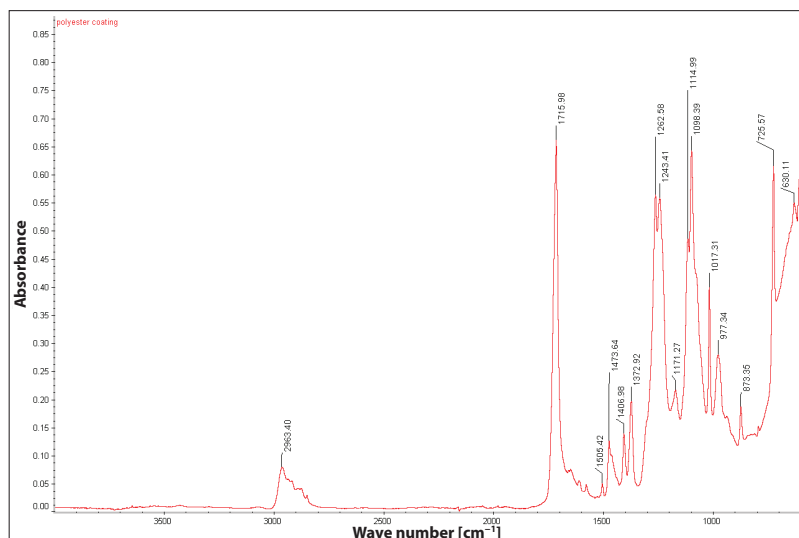
Fig. 3. FTIR spectrum of the topcoat: unchalked polyester coating sample after 18 months of UV exposure

Rys. 3. Widmo FTIR powłoki nawierzchniowej: próbka nieskredowanej powłoki poliestrowej po 18 miesiącach narażenia na promieniowanie UV
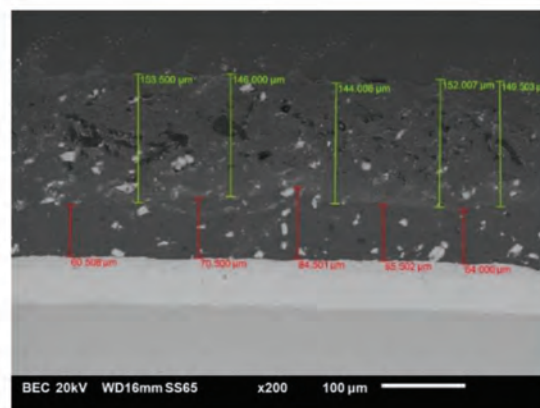

Fig. 4. Cross-section through the coating system produced on the zinc immersion coating – backscattered electron BSE/BEC mode image

Rys. 4. Przekrój poprzeczny przez system malarski wytworzony na zanurzeniowej powłoce cynkowej – obraz w trybie elektronów wstecznie rozproszonych BSE/BEC

One of the objectives of the review was to verify whether products that complied with the corrosion protection specification were used. FTIR spectra of the chalked and unchalked coatings were obtained (according to PN-EN 1767 [43]) using the atomic total reflectance (ATR) method at a resolution of 4 cm^{-1}. The FTIR spectra of the chalked coating taken from the frame column are shown in Fig. 2, while the FTIR spectra of the unchalked coating taken from the beam are shown in Fig. 3.

The FTIR spectrum of the chalked coating is typical of an epoxy coating, but slightly distorted due to its destruction. Peaks characteristic of bonds such as aliphatic C−H stretching (2965 cm^{-1}, 2869 cm^{-1}), aromatic ring (1607 cm^{-1}, 1503 cm^{-1}), C−O stretching (1233 cm^{-1}) can be distinguished on the spectrum. The unchalked coating is a polyester coating. Peaks characteristic of the following bonds can be observed on the FTIR spectrum: C=O stretching (1716 cm^{-1}), CH$_3$ bending symmetric (1373 cm^{-1}), CH$_2$ torsion (1263 cm^{-1}).

FTIR examination of the coatings revealed the use of two generically different coatings on the structural elements. The chalked coating is an epoxy coating with low UV resistance and was not applied according to the corrosion protection execution specification.

2.2. Assessment of whether pre-treatment of the surface with a conversion coating was used prior to powder coating

In duplex corrosion protection systems (zinc coating + paint coating), a common problem is the correct adhesion of the paint coating to the galvanised substrate. The most common reasons for poor adhesion of the coating are errors in the pretreatment of the surface prior to the paint application process, i.e. incorrect mechanical treatment or lack of conversion coatings obtained by chemical treatment. In the cited case, a zinc immersion coating and a powder coating were used, but the corrosion protection specification did not specify the pretreatment. The type and specific thickness of the powder coating was not indicated,

nor, consequently, was the resistance of the protection to the corrosive media specified in years. The purpose of the technical expert review was to determine the thickness of the coatings and to verify whether a chemical pretreatment with the formation of a conversion coating had been applied. SEM was the research technique used to answer the questions posed.

The purpose of the technical expert review was, among other things, to determine the thickness of the coating system and to verify whether a chemical pretreatment of the hot-dip galvanised steel with the formation of a conversion coating had been carried out. Fig. 4 shows a BSE/BEC microscopic image of a cross section through the investigated coating system produced on the hot-dip galvanized coating. An acceleration voltage of 20 kV was applied. In the BSE/BEC imaging mode, the differences in the shades of grey reflect differences in the chemical composition of the different areas. Fig. 4 shows (from bottom to top) the steel substrate and the coatings: zinc, primer and topcoat. In the case analysed, no conversion coating was observed and the average thickness of the primer and topcoats was 69 μm and 149 μm.

2.3. Determination of the cause of general corrosion of zinc-coated bolts

The test objects in this case were bolts galvanised according to EN ISO 10684 [44], which had suffered general corrosion after only a few years of operation of the steel structure. Nearly the entire surface of the bolt heads, washers and nuts was covered with iron corrosion products, while zinc corrosion products and zinc coating residues were observed only locally. The review focused on the analysis of the chemical composition of the corrosion products of the zinc coating, as accelerated zinc corrosion is most often associated with chemical aggression of the environment. For this purpose, the EDS/EDX (energy dispersive spectroscopy / energy dispersive X-ray spectroscopy) method was used, which incorporates a scanning electron microscope coupled to an X-ray energy dispersive spectrometer.
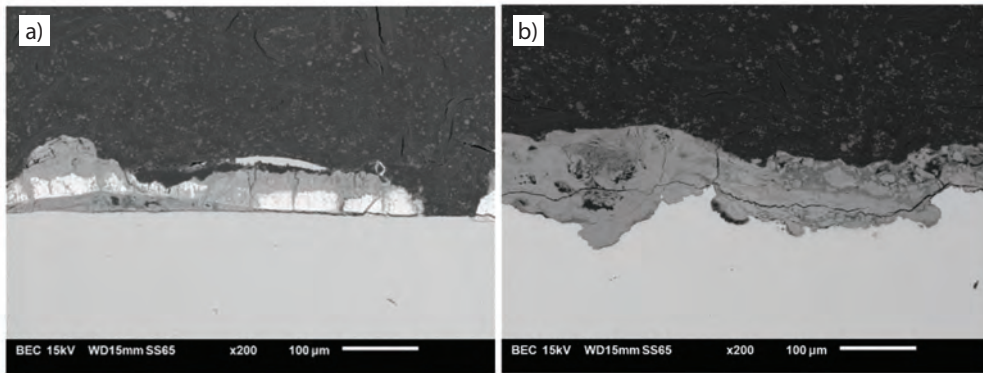
Fig. 5. Cross-section through the bolt head: a) in the area where the oxidised zinc coating remains (white colour) on the steel, b) in the area where the zinc coating has completely deteriorated after one year of operation

Rys. 5. Przekrój poprzeczny przez łeb śruby: a) w miejscu pozostałości utlenionej powłoki cynkowej (barwy białej) na stali, b) w obszarze, na którym powłoka cynkowa uległa całkowitej destrukcji po roku eksploatacji
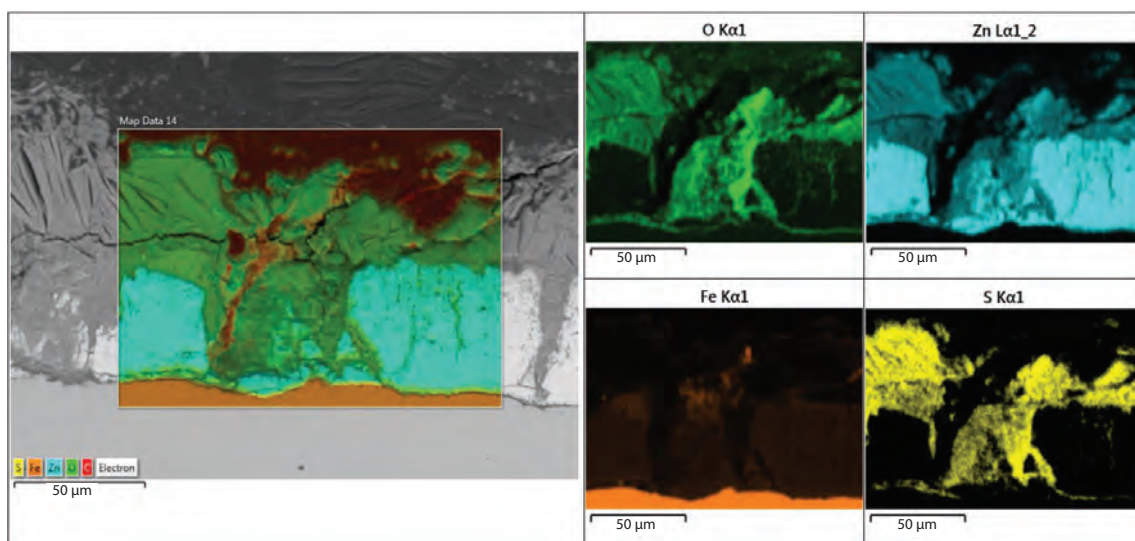

Fig. 6. Elemental distribution (excluding carbon) on the cross-section showing the remains of the zinc coating after one year of operation

Rys. 6. Rozkład pierwiastków (z pominięciem węgla) z uwidocznionymi na przekroju poprzecznym pozostałościami powłoki cynkowej po roku eksploatacji

Fig. 5 shows SEM-BSE/BEC micrographs of the area of the bolt where residual zinc coating is visible beneath the zinc corrosion products (white in colour) and the area where only iron corrosion products were observed (dark grey in colour). Steel is visible at the bottom of the micrographs in Fig. 5. The results of the surface chemical composition analysis on the cross-section of the remaining zinc coating are shown in Fig. 6. A cracked and oxidised zinc coating can be observed on the SEM-BSE/BEC image. As a result of the numerous cracks in the zinc coating, which can be seen mainly in the oxygen surface distribution (Fig. 6), the zinc coating is oxidised not only on the external side, but also on the side of the iron-rich intermetallic phase from the Fe-Zn system (γ-phase). Impurities in the form of sulphur compounds accumulate in the corrosion products of the zinc coating, which was also confirmed by a linear chemical composition analysis on a cross-section through the zinc coating with a lower degree of corrosion (Fig. 7). Linear chemical composition analysis is used to image compositional changes and fluctuations along the indicated line. Using Fig. 7 as an example, it can be observed that the highly oxidised surface of the zinc coating is the site of sulphur-containing impurities accumulation. Subsequently, this element is diffused deep into the zinc-rich δ-phase.

The results of the review indicated that the reason for the corrosion damage to the zinc coatings on the bolts was excessive aggression of the structure's operating environment in relation to the assumed durability of the corrosion protection system. The design of the corrosion protection system in the analysed case was based on an environmental corrosivity category of C4 according to PN-EN ISO 12944-2 [45], while in reality it was chemical aggression, related to the specific location of the structure (in close proximity to a fertiliser warehouse and storage areas for substances corrosively aggressive to zinc and steel, e.g. sulphates).

3. Conclusion

The analytical techniques presented in this article have been used in the course of conducting technical expert reviews to determine the causes of destruction of corrosion protection systems for steel structures.
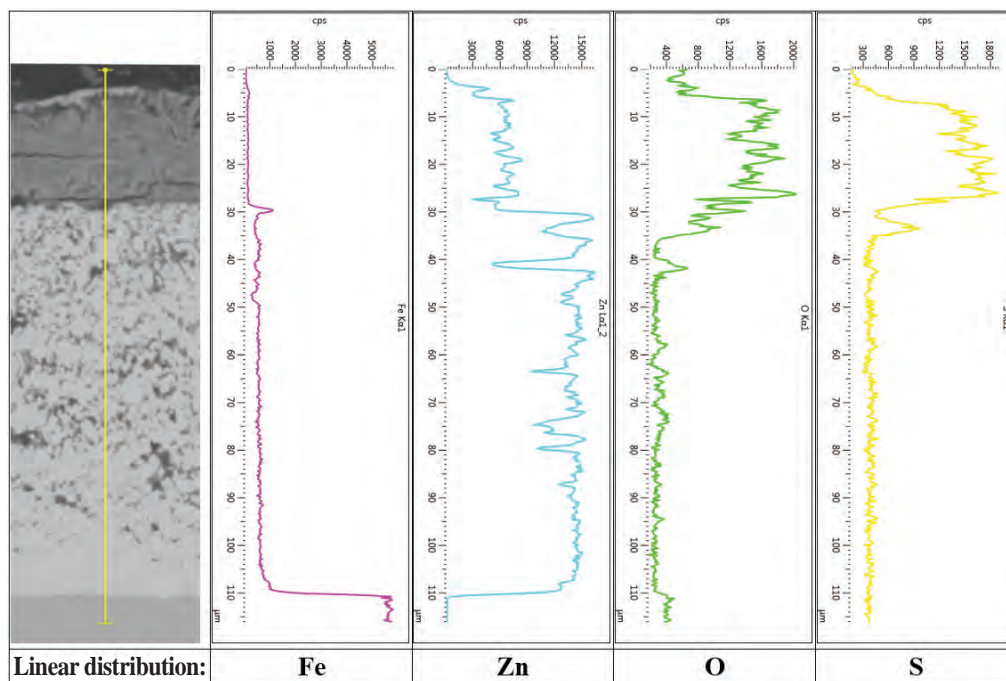
Fig. 7. Linear elemental distribution on the cross-section of the zinc coating after one year of operation

Rys. 7. Liniowy rozkład pierwiastków na przekroju poprzecznym powłoki cynkowej po roku eksploatacji

On the basis of the FTIR analysis results obtained, it was possible to determine why the topcoat on some parts of the steel structure had become chalked after only 18 months of UV exposure. The FTIR technique confirmed that an epoxy coating that did not comply with the corrosion protection specification had been applied.

In determining the cause of the poor adhesion of the powder coating to the hot-dip galvanised steel, scanning microscopy images confirmed the investor's suspicion that no chemical pretreatment of the surface to form a conversion coating had been carried out prior to painting. In addition, it was possible to locally assess the thickness of the zinc coating and the organic coating system in the duplex system. It was confirmed that the thickness of the zinc coating was in accordance with PN-EN ISO 1461 [46].

Imaging by scanning microscopy combined with a chemical composition analyser in the form of an X-ray energy dispersive spectrometer proved essential in determining the cause of the premature corrosion of the galvanised bolts, relative to their assumed durability. The presence of sulphur in the zinc corrosion products indicated chemical aggression of the environment and inadequate choice of fastener material in the structural design in relation to the corrosion environment.

CRediT authorship contribution statement
Izabela Kunce: Conceptualization, Formal analysis, Investigation, Resources, Visualization, Writing – original draft, Writing – review & editing.

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