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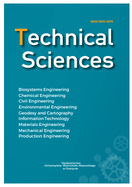

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CHARACTERIZATION OF DEPOSIT LAYERS FORMED ON GREY CAST IRON

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Abstract

This paper presents the results of diagnostic examinations conducted on the water pipe made of grey cast iron that were operated in conditions for 260 thousand hours. The tube was exposed in a soil. The phase composition of the deposit layers formed on both sides of the tube walls (outside and inside) was examined using optical and confocal microscopy and X-ray diffraction. The obtained XRD test results showed that mainly α-FeOOH) and γ-FeOOH is formed on the inside of the pipe wall. In turn, on the outside, in addition to the above-mentioned compounds, also SiO_2 is formed. The results of these examinations have shown a higher degradation of pipe on the outer side. The arithmetic mean deviation of the profile from the mean line was 6.23 µm and 8.07 µm for the inner and outer side of the pipe wall, respectively. The work demonstrates the usefulness of material science, especially X-ray structure, topography and surface studies, in characterizing the degradation processes of layers formed on gray cast iron exploited in the water supply industry.

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Introduction

Water pipes are made of: asbestos-cement (JONES et al. 2021), steel (JONES et al. 2021), cast iron (JONES et al. 2021), polymer materials (JONES et al. 2021, VERTOVA et al. 2019), composites, concrete and reinforced concrete, and copper (VERTOVA et al. 2019). There are also lead or lead lined pipes in water distribution systems (GERKE et al. 2009). Various types of corrosion contamination are formed in metallic pipes during the flow of drinking water (YANG et al. 012). The process of formation and physicochemical characteristics of corrosion scale in drinking water supply pipes depend, among others, on the material of the pipes, the quality of the water with which the material has come into contact and the hydraulic conditions. Corrosive scales formed on the surfaces of ferrous pipes are an important affect the water quality of water in drinking water distribution systems, as they release contaminants and cause water discoloration (LI et al. 2018). Increasingly, ClO_2 is used to disinfect drinking water (VERTOVA et al. 2019), which research shows is harmful by contributing to aggravating corrosion, especially of copper pipes. VERTOVA et al. (2019) in they conducted research on the influence of ClO_2 on the inner surface of pipes. The tests were carried out on both metal pipes (copper, galvanized steel) and polymer pipes. The conducted research showed that both metal and polymer pipes showed a strong degradation after only a short contact with ClO_2. RAJAKOVIC-OGNJANOVIC and GRGUR (2011) conducted research on steel pipes, concluding that, depending on the water composition, the corrosive layers represent various iron compounds, the consequence of which is the degradation and destruction of pipe materials. The water quality influences the release of iron from corroded surfaces. With the presence of oxidants in the water, the iron that is released as divalent turns into a trivalent form. Corrosion of iron in an aqueous environment can be uniform, develop evenly or locally over the entire metal surface. Homogeneous corrosion is manifested by uniform corrosion layers of similar composition, local corrosion gives different types of corrosion products, differing in composition and proportions of components. According to the researchers (CHEN, CHEN 2022), the formation of scale in the water pipeline significantly affects the chromaticity and turbidity of the water. Due to large changes in the direction and velocity of flow, as well as with fluctuations in water pressure in the pipes, the phenomenon of iron release occurs (CHEN, CHEN 2022). This iron destroys the relatively stable structure of the scale, which in turn leads to deepening of the corrosion in the pipe. Along with the operating time of the water supply elements, the scale formed in the pipes continues to thicken. The consequence of this is the reduction of the cross-section of the flowing water and thus the limitation of its flow (CHEN, CHEN 2022). Cast iron pipes are widely used in water distribution systems due to their low cost, availability, ease of manufacture and adequate corrosion resistance (SHANKAR et al. 2020). The outer surfaces of cast iron water networks buried in the soil for several dozen years are usually covered with a corrosive layer characterized by a rough, undulating topography (MELCHERS 2017). Cast iron pipes are widely used in water distribution systems around the world (QI et al. 2016). Cast iron has good corrosion resistance in the atmosphere, and lower corrosion resistance in water and soil (QI et al. 2016). Corrosion on the inside of the pipe wall in these pipes is the most common problem in the water distribution system. Corrosion on the inside of the pipe wall is responsible for the reduction of water transfer capacity and deterioration of the quality of drinking water (QI et al. 2016). Corrosion of this wall may lead to an increase in iron concentration, and thus to the rapid decomposition of disinfectant residues or iron particle suspensions, which causes turbidity and gives the tap water a yellow or brown color (QI et al. 2016). Water pipes should meet specific requirements, such as appropriate durability, strength, resistance to tap water and ground water, or complete tightness. The durability of water pipes is closely related to their corrosion resistance. The work focuses on the gray cast iron material used in water pipes used several decades ago. Currently, some of these pipes are still used in waterworks. By carrying out a series of material science tests, it is possible to determine to a large extent their suitability for further exploitation.

The aim of the paper was to present the development of pitting corrosion of both internal and external surfaces of water pipes buried in the ground.

Material and Methods

Samples with dimensions of 10 mm × 10 mm were taken from the pipe. The specimens were made transversely to the material axis. The preparation of the metallographic specimen consisted of grinding and polishing. Selected samples were etched in five percent nitric acid. The research material was a water pipe after operating for 260,000 hours. The test was performed on a cast iron socket pipe with a nominal diameter of 50mm. The structure tests were carried out on the entire cross-section of the pipe wall. The microstructure examinations comprised microscopic examinations using an optical microscope Olympus GX41.

The deposit layer was studied on a surface at the inner and outer surface of the tube wall. Surface research included:

– confocal microscope Lext;

– analyses of the phase composition using X-ray diffraction (XRD). The XRD experiments were carried out on a Bragg-Brentano diffractometer 3003T/T from SEIFERT. In order to reduce the fluorescence radiation of iron, the CoK radiation (1.79026 Å) was used. The X-ray tube was operated at 40 kV and 30 mA. The XRD patterns were collected in the 2 theta range between 15 and 120 with an angular step of 0.1;

– stereometric measurements of the surface were carried out using the LEXT confocal microscope, designed for 2D and 3D surface examinations. The arithmetic mean deviation of the profile from the mean line (*Ra*) and the roughness height were determined according to ten profile points (*Rz*). Moreover, the research allowed to determine the waviness height (*Wz*) and the arithmetic mean of the waviness profile ordinates (*Wa*).

Results and Discussion

The structure of the tested material was different along the pipe cross-section. Directly from the inside and outside, a ferritic structure was observed with a large amount of sulphides both inside and at the grain boundaries. An example of the structure is shown in Figure 1. Going deeper into the pipe wall, the ferritic-pearlitic structure of gray cast iron was visible, with few precipitations of graphite

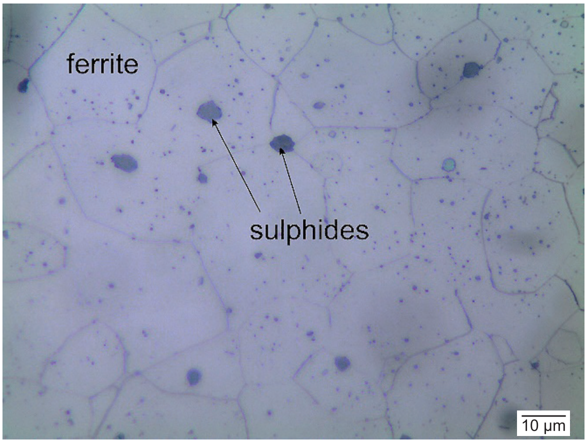

Fig. 1. Structure directly from the side of the pipe wall, magnification 1000×

and numerous precipitations of sulphides (Fig. 2). Graphite precipitates occur in a branched form, characteristic of distorted graphite. This graphite is in the form of curved streaks that are branched in different directions. These strands touch each other at different points. In grey cast iron, excess carbon beyond the solubility limit in the austenite phase precipitates as flake graphite (Shankar et al. 2020). Sulfur in cast iron occurs in the form of iron and manganese sulphides (FeMn)S (Stefan et al. 2021).

The microscopic observations carried out on the transverse sections showed the presence of deposits on both sides of the pipe wall (Fig. 3). The thickness of the layer on both sides was varied. At its widest point, the

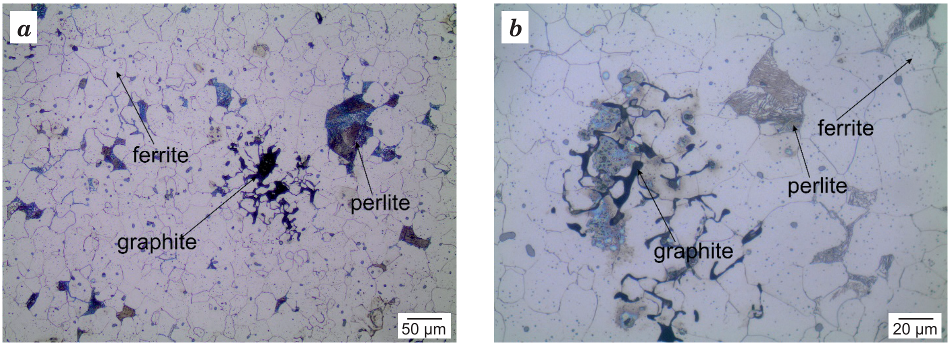

Fig. 2. Structure at the center of the pipe wall section. Visible ferrite, perlite, graphite and sulfides: *a* – magnification 200×, *b* – magnification 500×

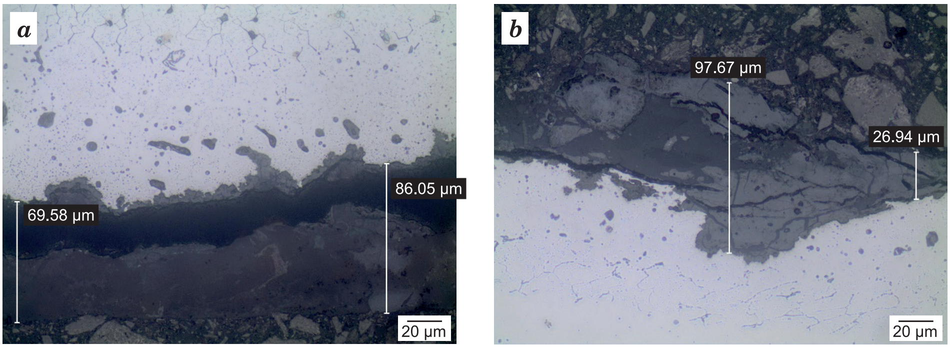

Fig. 3. Cross-section of the sediment layer:
a – inner side of the pipe wall, *b* – outer side of the pipe wall

layer had a thickness of 86.05 μm and 97.67 μm for the inner and outer sides of the pipe wall, respectively. In both cases, pitting corrosion is locally visible at the contact of the sediment layer with the substrate. On the inside, a gap was observed between the native material and the layer. On the other hand, observations of the outer side revealed numerous cracks in the sediment layer. These cracks take on a multidirectional character. In the paper (YANG et al. 2012), sections of cast iron pipes after 20-25 years of operation were examined. The researchers found mounds, so-called corrosion nodules, and thick continuous scales on the highly internally corroded pipe surfaces. WICHOWSKI et al. (2021) did not observe pitting corrosion in cast iron pipes after 30 years of operation. In addition, no reduction in wall thickness and wall degradation were observed, which indicates a slight damage to the pipes and a possible extension of their service life. Researchers have shown (WICHOWSKI et al. 2021) that the structure and chemical composition of the accumulated sediments are different in both layers: the outer and inner layers. The chemical composition of the accumulated sediments, both in the outer and inner layers, is dominated by iron oxides and hydroxides (WICHOWSKI et al. 2021). The inner layer is characterized by a higher calcium content (WICHOWSKI et al. 2021). According to the literature, significant accumulation of sediments in water pipes may lead to deterioration of water quality, i.e. red water effect (WICHOWSKI et al. 2021). According to GUO et al. (2020) ductile iron has the lowest corrosion rate and the most stable scale compared to gray iron and carbon steel. In ductile iron, both the outer and inner scales are dense enough to inhibit the corrosion process.

The obtained XRD test results showed that mainly goethite (α-FeOOH) with a small amount of lepidocrocite (γ-FeOOH) is formed on the inside of the pipe wall. In turn, on the outside, in addition to the above-mentioned compounds, also quartz (SiO_2) is formed. The results of studies by YANG et al. (2012) showed that magnetite (Fe_3O_4) and α-FeOOH were the main components formed on the inside of the pipe wall. The presence of Goethite on cast iron pipes was also observed by WICHOWSKI et al. (2021). In addition to the above-mentioned deposits, researchers (YANG et al. 2012) also observed, although in smaller amounts, such as: lepidocrocite, siderite ($FeCO_3$), green rust ($Fe_6(OH)_{12}CO_3$), hematite (Fe_2O_3), akaganeite (β-FeOOH), iron carbonate hydroxide hydrate ($Fe_6(OH)_{12}$-$CO_3 \cdot 2H_2O$), honessite (Ni_6Fe_{23}+$(SO_4)(OH)_{16} \cdot 4H_2O$) and pyrite ($FeS_2$). The presence of γ-FeOOH was also confirmed in (WICHOWSKI et al. 2021). On several of the dozens of samples, YANG et al. (2012) also revealed non-iron-based phases, such as: SiO_2, calcite ($CaCO_3$), albite ($(Na,Ca)Al(Si,Al)_3O_8$), microcline ($K(AlSi_3)O_8$), gypsum ($CaSO_4 \cdot 2H_2O$). The presence of $CaCO_3$, Fe_3O_4, α-FeOOH, SiO_2 was also confirmed in the paper (LI et al. 2018). Also detected: tenorite (CuO), manganese oxide (MnO), zincite (ZnO) and hydrozincite ($Zn_5(CO_3)_2(OH)_6$). Short-term laboratory tests (6 months) (GUO et al. 2020) confirmed the formation of an effective anti-corrosion scale layer consisting of α-FeOOH and $CaCO_3$.

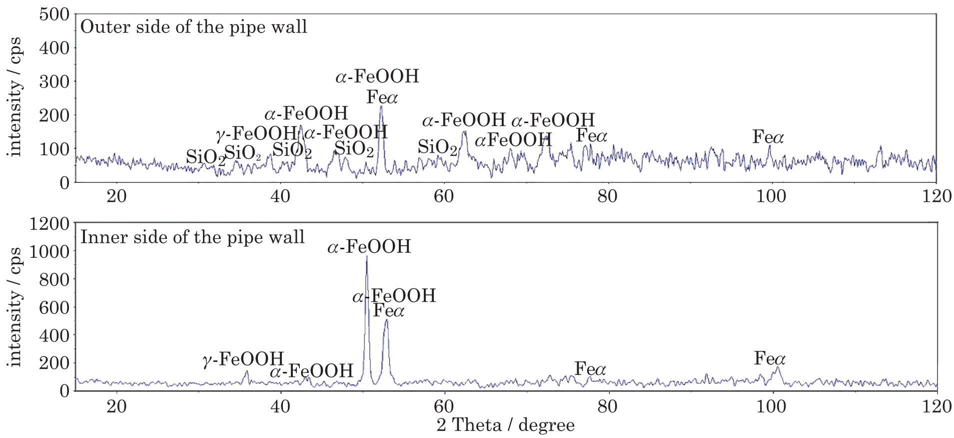

Fig. 4. Diffraction pattern of the outer and inner side of the pipe

Fe, Ca, Mn, Zn and Al are the predominant metallic impurities that are identified in the sediments in the water pipes (TIAN et al. 2022).

The obtained results of the surface topography showed that on the outer side of the pipe wall, the sediment layer has a more developed surface (Figs. 5, 6, Tab. 1). The arithmetic mean profile deviation from the mean line was less than two units higher for the outer side of the pipe wall. A similar growth character was

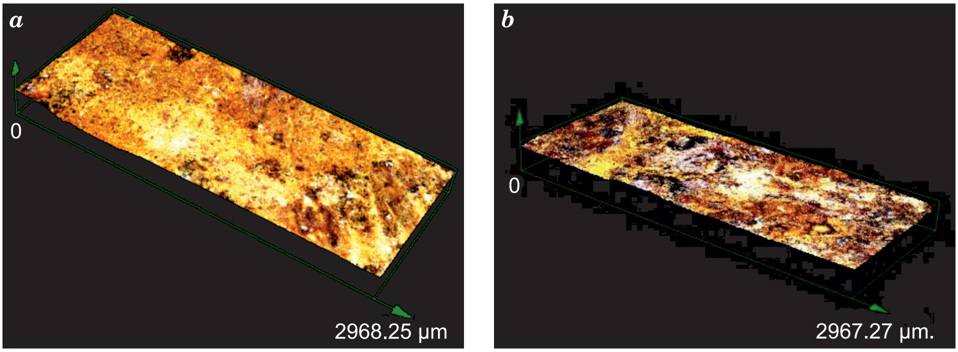

Fig. 5. 3D view, confocal microscope:
a – inner side of the pipe wall, *b* – outer side of the pipe wall

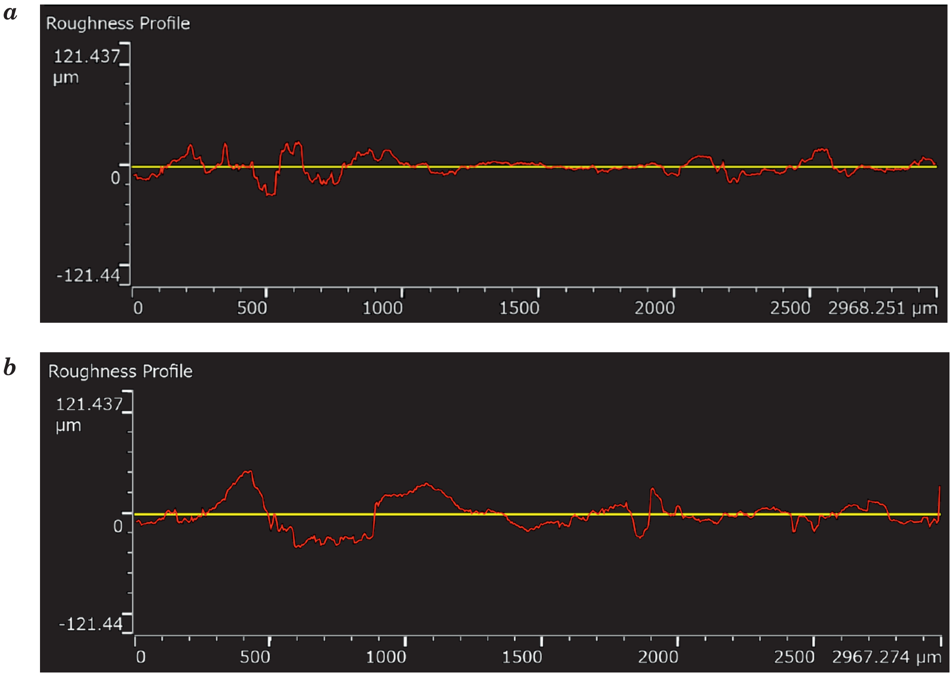

Fig. 6. Roughness profile: *a* – inner side of the pipe wall, *b* – outer side of the pipe wall

Table 1

Roughness test results

Specification	*Ra* [µm]	*Rz* [µm]	*Wa* [µm]	*Wz* [µm]
Inner side of the pipe wall	6.23	41.55	9.44	37.01
Outer side of the pipe wall	8.07	47.00	10.70	50.70

observed for the *Rz* parameter, only by 6.5 units. A slightly smaller increase was observed for the arithmetic mean of the waviness profile ordinates (an increase by 1.26 units). The greatest increase was related to the *Wz* parameter and amounted to as much as 13.96 µm. As shown in the literature, digital microscopes can be used for empirical roughness measurements of the formed sediments on water pipes (WICHOWSKI et al. 2021). Observations of the surface topography of water pipes are very important due to the progressive development of their corrosion. ASADI and MELCHERS (2018) presented the observations of the surface topography of cast iron pipes exposed for 34 to 129 years in clay soils. For this pipes, pitting was inferred to progress in a step-wise manner, with limited, staged increases in pit depth and lateral amalgamation of closely neighbouring pits to form plateaus from which new pitting occurs, in a manner generally similar to that reported for steels.

Summary

Based on the microstructure investigations, X-ray diffraction and topography roughness the following statements and conclusions can be formulated:

1) The structure of the water pipe was graphite distorted in the ferritic--pearlitic matrix. Numerous precipitations of sulphides were also observed. Directly from the medium side, the structure was made of ferrite with numerous precipitates of sulphides.

2) Pitting corrosion was observed both on the outside and inside of the pipe wall.

3) The inner layer of sediments was composed of α-FeOOH and γ-FeOOH, while the outer layer was made up of α-FeOOH, γ-FeOOH and SiO_2.

4) The deposit layers formed on the outside of the pipe wall experienced higher degradation than the deposit layer formed on the inside.

5) Roughness test results showed higher parameters for the external side (Ra = 8.07 µm, Rz = 47.00 µm, Wa = 10.70 µm, Wz = 50.70 µm) than on the inside (Ra = 6.23 µm, Rz = 41.55 µm, Wa = 9.44 µm, Wz = 37.01 µm.

6) Summarizing, the study tried to demonstrate that material science (structural), especially X-ray, and methods related to surface topography are important tools in the characterization of sedimentary layer structures after long-term

exploitation. Destruction of the top layer occurred as a result of emergence and separation sediment layers. In the future, it will be very important to combine structural and mechanical properties on this type of elements.

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