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# Microscopic Analysis of Selected Materials Used for Hot Water Heating Using SEM

Ľuboš Marček (0009-0005-3699-0100), Ján Vavro, jr. (0009-0009-6787-9041), Jan Vavro (0009-0001-3704-4934)
Faculty of Industrial Technologies, Alexander Dubček University of Trenčín. Ivana Krasku 491/30, 020 01, Púchov. Slovak Republic. E-mail: lubos.marcek@tnuni.sk, jan.vavro.jr@tnuni.sk, jan.vavro@tnuni.sk

**The presented work deals with the microscopic analysis of metallographically prepared structures of selected metal materials, new and several years of operation of hot water heating system pipes. Microscopic examination was carried out using a scanning electron microscope (SEM). Prepared samples of seamless steel pipes were subjected to thorough microscopic examination from the outer surface to the inner areas in order to interpret the specific structure, including changes in the inner surfaces due to wear. The study of microstructures and surface layers of unused and operated metal materials from randomly selected locations creates an overall picture of their sensitivity to the aqueous medium to which they are exposed for a long time. The prediction of the failure of selected metal pipes will be based on a comprehensive approach to solving all dependent and independent variables of the operated heating system. One of them is appropriate maintenance (continuous inspection), which must ensure reliable and, above all, safe operation and thus the service life of the pipes in real conditions of use. The experiment showed that the microstructure and character of surfaces play a key role in the behavior of metallic materials in real conditions. The achieved results provide scientific knowledge in the field of using materials for hot water heating. They contribute to the ability to appropriately evaluate the material used, to design the material and its joining technology, for various types of constructions of piping systems of sanitary and technical installations in the field of technical equipment of buildings. It is clear from the results of the work that the materials will achieve the prescribed service life. However, there are many aspects that contribute to the process of corrosion degradation. The contribution of this work to the field of technical practice is the identification of these critical aspects that condition the emergence of degradation processes, the design of processes and parameters for the safe operation of hot water heating.**

**Keywords:** Analysis, Microscopic, Structure, Material wear, Pipes

## 1 Introduction

Nowadays, metallic materials play a crucial role in a wide range of industrial applications and construction. Their material properties, such as strength, resistance and thermal conductivity, make them irreplaceable in the production of various components and technical objects (TO). In this context, it is important to have a deeper understanding of metallic materials so that their potential can be effectively used to prevent unwanted phenomena. The external environment can cause chemical, mechanical, or physical changes in materials, which become initiators of future degradations. Since this work deals with metal pipes, it is important to pay attention to their microstructure. Changes to the internal surfaces of pipes as well as their wear and tear have a major impact on their functionality and lifetime. The nature of the microstructure of materials affects the overall properties and resistance to external influences. Superimposed by corrosion, wear can cause limit states and material degradation which affect safety, reliability and efficiency in specific applications. Solving this issue is essential for engineers, designers and scientists in the development and production of new products. The purpose of the introduced study is based on a detailed analytical research of metal materials used for the production of pipes with a focus on their microstructure. The internal surfaces of pipes as well as other hollow bodies in interaction with the flowing water medium are exposed to corrosion and further wear. Specifically, this work is dedicated to the microscopic examination for selected and prepared steel pipe samples, using an electron microscope (SEM). The aim of the investigation is to identify the selected microstructures and their purity (inclusions). Further, it is also focused on the study of new layers, for example, oxidized, corrosion areas from the surface of materials. Next, attention is also paid to important aspects, such as the effect of corrosion on subsequent wear, crack initiation and phase change in the microstructure of the material which can reduce the overall lifetime of metal components. The main areas of research can be expressed in the following points:

- The microstructure of selected materials and examination of the internal surfaces of metal pipes,

- The effect of corrosion on wear and change in the mechanical properties of the material,
- The electron microscopic analysis (SEM) for metallographically prepared material samples,
- The identification of inclusions and their evaluation in the given microstructure of the material.

## 2 Metal materials

Metallic materials are the basic building blocks of many hydraulic and industrial applications. They are characterized by their material properties which include strength, toughness, malleability, conductivity of heat and electricity, and many others. These materials are mostly selected due to their ability to withstand mechanical loads, while providing the reliability and long component lifetime [1].

### 2.1 Structure of metallic materials

Ferritic-pearlitic phases in the structure of steels represent one of the most common microstructures in construction and TO. With this structure, steels are also used for the production of seamless pipes intended for distribution of heat. Ferritic and pearlitic grains can be of different sizes with different proportions of these phases. They form a complex microstructure where, depending on the ferrite content, the material seems to be harder or softer, i.e. more or less resistant to wear. In addition to the ferritic-pearlitic microstructure, there are many other undesirable phases, such as carbides, sulfides and oxides. These phases worsen the material properties of steels. For this reason, the purity of metals is important and it is controlled as the first to exclude negative effects on the occurrence of future degradation processes in TO operation [2].

- By their nature, inclusions are strange, undesired particles in metal materials that can affect not only mechanical but also corrosion properties. The analysis and identification of inclusions is crucial for predicting the failure of steels, in various TO, operating under the real conditions,
- The distribution and size of the grains in the microstructure of the material has a significant effect on its mechanical properties. For example, when the inner surfaces of heating pipes contain inhomogeneity in the size of the ferritic-pearlitic grains, it is reflected in less resistance of the material to load and wear [2].

### 2.2 Properties of metallic materials

The properties of metallic materials are decisive for their use in specific hydraulic element applications. It is necessary to select the essential properties that affect the TO function according to their use. Therefore, from the aspect of the material parameters, it is about:

- The mechanical properties – strength, toughness, ductility, hardness,
- The physical properties – thermal conductivity, electrical conductivity, resistance to temperature changes, thermal stability,
- The resistance to chemical influences, corrosion, cavitation,
- The technological properties such as castability, malleability and weldability [1,2,3,4].

The required properties are used in the selection and design of metal materials for hydraulic elements (e.g. pipes) working in various environments.

## 3 Steel pipes for aqueous media and their internal surfaces

As a material, steel is one of the most used materials for the installation and production of traditional hot water heating pipes. It has the necessary and expected material properties that ensure good resistance to operating pressures. Pipes made of steel are sufficiently strong and durable, thus preventing the permeability of the medium as well as the given pipes are resistant to expansion changes and deviations from the specified geometry. The potential strength of steel and its ductility are based on the mass percentage of carbon. The higher percentage of carbon means the stronger and harder steel. On the other hand, it is less malleable. Relating to the material for pipes of heating system, the low carbon steel is used for the production of pipes because the steel with a high carbon content has a reduced melting point. Within the scope of standards, defining carbon steel, other elements can be used, e.g.: chromium, tungsten, manganese, cobalt, molybdenum as additional or alloying elements that can improve the material properties. For the assembly of heat-carrying medium distributions for hot-water heating, the following materials have been used still used: black steel pipes, carbon steel with external galvanization and copper [2,5,6].

The internal surfaces of steel pipes, which interact with the water medium, represent a critical aspect for their further functionality and service life. These surfaces are in direct contact with water of different purity and temperature which can affect the internal material surface of pipes. Their importance is based on:

- The material integrity – the internal surfaces of the pipes ensure or provide the mechanical strength and stability of metal hydraulic components,

- The impact on material properties – the nature of the microstructure and the occurrence of the undesirable phases (inclusions) affect resistance to load and degradation,
- The corrosive change of materials – both internal and external surfaces are susceptible to corrosion, which can lead to material degradation and a reduction in overall service and lifetime [2,5].

Steel pipes used for liquid media are exposed to various types of wear and temperature changes. The prediction of future degradations is based on the identification of:

- The corrosion layers – if the corrosion areas relating to the internal surfaces of the pipes are critical, they can lead to degradation of the base material and a reduction in service and lifetime. The identification of these layers is essential to prevent the spread of corrosion and the initiation of cracks that propagate into the inside layers of the pipe wall,
- The cavitation and its effects on the material – microscopic volumes of material are torn out by cavitation as a result of pressure changes in the microlocations of the water medium. The present corrosion with cavitation can significantly weaken the pipe walls and change their geometry. This phenomenon can have serious consequences for changing the mechanical properties of the material that can lead to component failure [5,7,8].

The researching and analyzing these processes for the inner surfaces of metal materials (pipes) can be the crucial factors for better understanding of their reaction or response to real operating conditions. As long as all aspects that can affect the occurrence of material failure are mapped, it is possible to develop measures to optimize their performance and in-service conditions as well as lifetime.

## 4 Wear of metal pipes

Wear of metal materials can occur from various environmental influences and it depends on the specific application and liquid environment. The typical main wear mechanisms of pipes for aqueous media are:

- The abrasion – surface wear due to friction with other materials or particles in the internal (liquid) environment,
- The adhesive wear – friction between liquid versus metal surfaces can cause a change in profile (reduction of flow diameters) and surface geometry,
- The corrosion wear – change of the surface microstructure to brittle corrosion products (oxides) which are released and exfoliated into the liquid medium, thus becoming abrasive particles,
- The fatigue wear – gradual weakening of the material as a result of repeated loading with liquid and changes in temperature and pressure,
- The cavitation wear which is caused by the change of local pressures in different micro-volumes of the liquid, resulting in the implosion of bubbles that tear it from the surface due to its dynamic impact on the material and thus it becomes an abrasive particle [2,5,9].

### 4.1 Effect of corrosion on wear

All purposely produced materials are under slower or faster erosion when they are in contact with the external environment, and it stands for process of physico-chemical destruction of materials due to the influence of the environment that is the fundamental aspect and definition of corrosion [10,11]. Pipes are commonly a very critical part within the individual industrial application, including in the construction industry, whether they are for fire and potable water distribution, technical water or heating. When designing pipeline systems, it is necessary to pay attention to the increased requirements for safety and the selection of suitable material. The material for pipe systems should meet a number of requirements, involving required strength, technological aspects as well as resistance to corrosion in the given environment. The susceptibility of pipelines to corrosion depends on many indicators and also on the type of construction of the pipeline system [10,11]. The degradation corrosion process in a liquid medium can be described as an electrochemical process which is superimposed by other agents, such as impurities, microorganisms, chemical compounds (salts, hydroxides, acids, organic substances) and many others. Corrosion in water is influenced by dissolved oxygen, pH and water temperature [12,13].

If corrosion occurs in the pipes, corrosion fumes are produced and abrasive particles occur and it leads to the acceleration of the entire corrosion process. Corrosion products can destroy protective layers and increase friction, thus accelerating the entire wear process. The combination of corrosion and wear can lead to the formation of cracks, rupture of the material, resulting in the overall deterioration of the material properties [10]. The interaction of metal materials with the external environment is a critical aspect affecting their service and lifetime. The factors, such as temperature, humidity, environmental chemistry and pressure can affect the rate of corrosion and wear. The long-term impact of these deteriorative factors can lead to a gradual weakening of material properties that affect the ability to withstand loads and preserve integrity [12,14].

## 5 Tests of materials

### 5.1 Microscopic evaluation

Microscopic evaluation is commonly used for controlling the state of the materials structure. It is carried out using light and electron microscopes. For microscopic control and evaluation, the sample is taken from the places where the largest and smallest structural heterogeneity is expected. In order to find out the causes of fractures or component failures, the samples from a defective pipe area are cut and subjected to the observation and in addition, for comparison, the sample from an undamaged pipe area is also prepared and observed. For longitudinal sections, the observation involves:

- The morphology and arrangement of non-metallic inclusions,
- The degree of plastic deformation of the individual structural components of the metal base material,
- The occurrence of linearity,
- The effect of heat treatment on the microstructure.

Transverse sections are used to observe:

- The distribution of non-metallic inclusions in the entire cross-section,
- The depth and uniformity of surface or corrosion layers,
- The penetration depths of surface defects [15].

## 6 Experiment – microscopic analysis

Microscopic analysis was performed for four materials which were cut from different types of pipes. For better evaluation, the cuts from a pipe, which has not been in service yet, were used as a standard o reference sample. The other samples of pipe were from service where there was the water medium flowing through them for a certain period of time. Individual parts of the materials were cut and metallographically prepared in order to study the internal and external surfaces of the pipe (designated as sample 1, 2, 3 and 4). All samples (Fig. 1 and 2) for microscopic analysis were prepared as transverse sections. The samples were cleaned in isopropyl alcohol in an ultrasonic cleaner for 20 minutes [5].

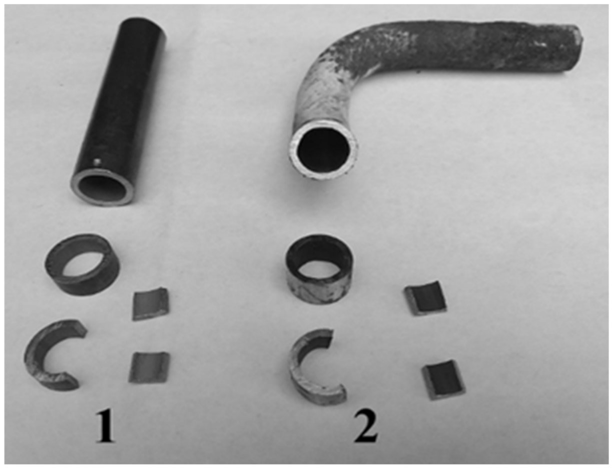



***Fig. 1*** *Samples from selected areas of metal pipes [5]*

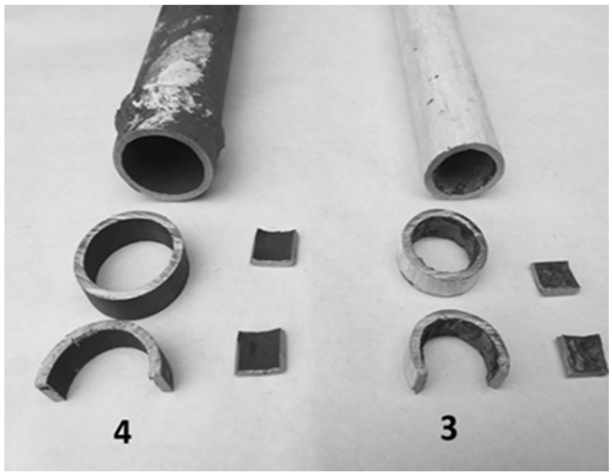



***Fig. 2*** *Samples from selected areas of metal pipes [5]*

The cleaned samples were examined with an electron microscope – VEGA 2 TESCAN. Before the microstructure evaluation, the samples were etched. Each sample was examined in detail from the external surface to the internal surface and attention was mainly paid to the nature of the material microstructure as well as purity and the chemical composition of inclusions, corrosion products and internal surface deposits.

The first observation and analysis was performed for sample No. 1 (non in any service – seamless steel pipe DN 15) from material 11 353 (according to STN 42 0250 – for threading and welding). The tube was not protected with any painting. At high resolution, the evaluated cut of the pipe revealed the characteristic non-uniformity of the material and there was the occurrence of oxides, which form the surface of the pipe (Fig. 3). A previous visual inspection of the outer surface indicated a large roughness and some non-uniformity. A detailed examination of the surface (at 2000x zoom) highlighted its broken integrity which could be attributed to the process of forming technology (Fig. 4). The internal surface did not show the occurrence of the corrosion layers or deposits due to the fact that it was a new pipe – it was not used in service before. The microstructure was identified as ferritic-pearlitic structure. The composition of the steel was controlled in the site which was tightly close to the internal surface (Fig. 5). Chemical analysis confirmed that from the qualitative aspect, it is common plain steel that had not been micro-alloyed or surface treated [5].

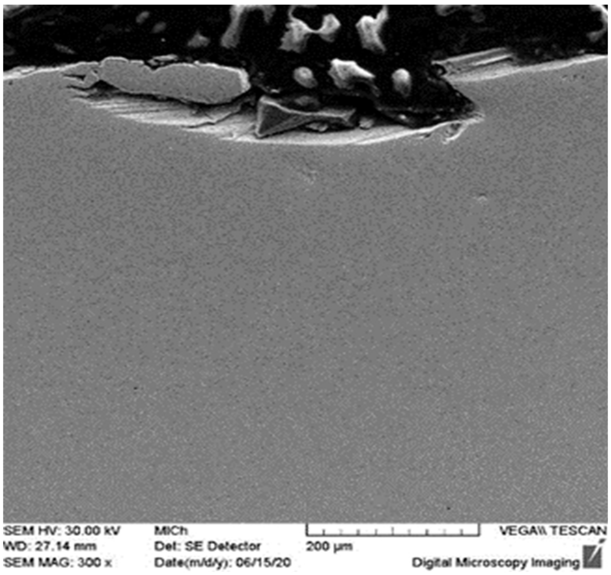



***Fig. 3*** *Detail of the non-uniformity on the external surface (300x zoom) sample No. 1 [5]*

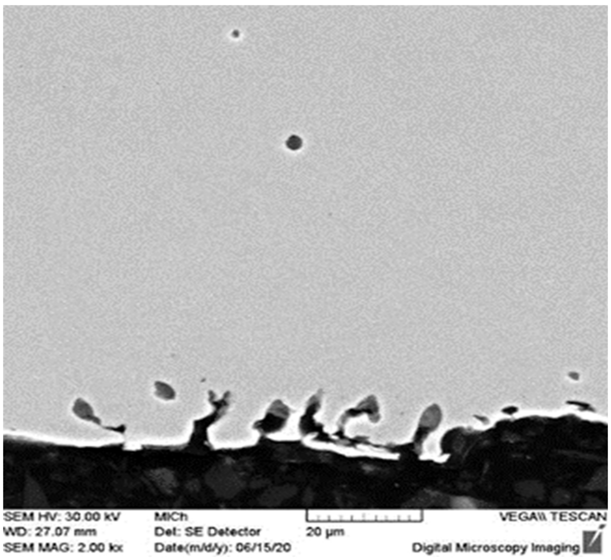
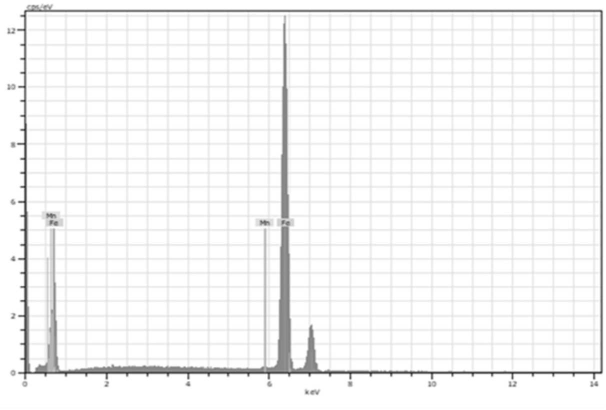



***Fig. 4*** *Internal surface with inclusion (2000x zoom) sample No. 1 [5]*

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                Spectrum:  2

El AN  Series  unn. C norm. C Atom. C Error (1 Sigma)
               [wt.%]  [wt.%]  [at.%]           [wt.%]
-----------------------------------------------------
Mn 25 K-series   0.00    0.00    0.00             0.00
Fe 26 K-series  97.34  100.00  100.00             2.49
-----------------------------------------------------
         Total:  97.34  100.00  100.00
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***Fig. 5*** *Results of chemical analysis for the edge of the internal surface – inclusions: m 2 – sample No.1 [5]*

Sample No. 2 is a seamless steel pipe (DN 15) made of 11 353 material class, according to STN 42 0250 (for threading and welding). The pipe was used for a hot water heating system with a temperature gradient of 70/55 °C for about 20 years. Microscopic examination identified the corrosion of the external surface of the pipe under the paint (Fig. 6). Corrosion propagation or spreading was observed along the grain boundaries towards the deeper interior (internal site) of the material.

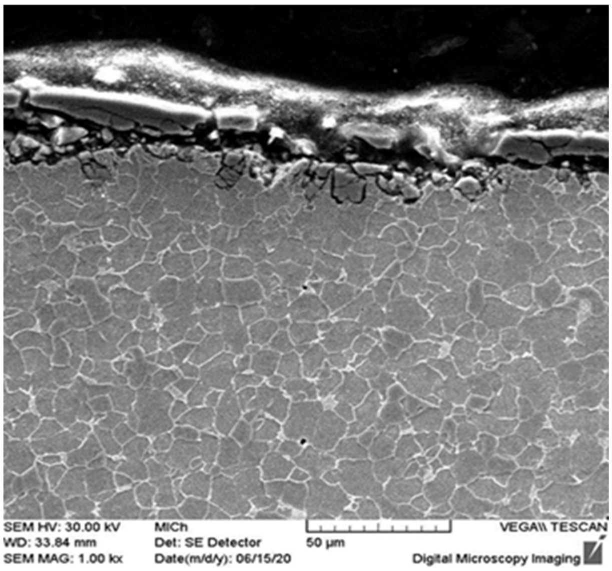



***Fig. 6*** *Detail of external surface, structure, corrosion process (1000x zoom) sample No. 2 [5]*

This is intercrystalline corrosion, which manifests itself in the fact that the material is inadequately disturbed. Grains are released from the surface, making the pipe material more sensitive to the external environment. Since it is a ferritic-pearlitic structure, the shape, size and distribution of the grains will be essential. Just below the outer surface were ferrite grains depleted in carbon. This decarburized subsurface area has different material properties than the base material and therefore becomes more sensitive to the formation of corrosion. The inner surface of the pipe (Fig. 7) is typical in that there are cracks towards the material, peeling of the base material and deposits. The detail of the microstructure in the given areas and the inclusions are shown at a magnification of 1000x in Fig. 7. It is undesirable if pieces of the base material and deposits are released and washed away by the flowing medium. In these cases, wear due to corrosion and cavitation is superimposed on abrasive wear, which is unacceptable for these pipes. Pipe degradation can accelerate, which is also reflected in thinning of the wall thicknesses (change in geometry), which can even lead to perforation of the material and leakage of the aqueous medium.

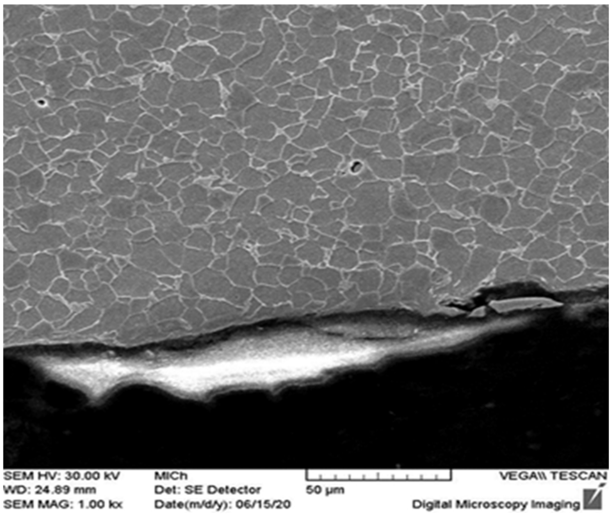
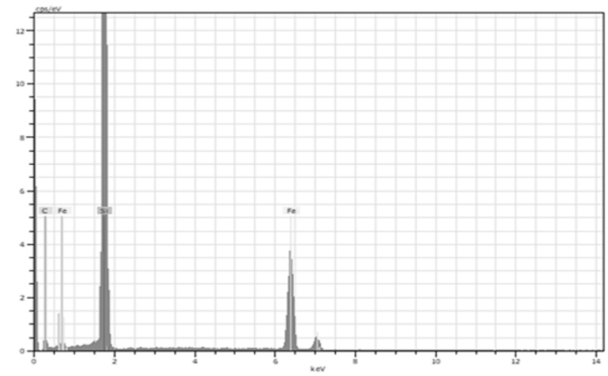



***Fig. 7*** *Detail of internal surface, structure, inclusions, corrosion deposits (1000x zoom) sample No. 2 [5]*



Spectrum: 1

| El | AN | Series | unn. C [wt.%] | norm. C [wt.%] | Atom. C [at.%] | Error (1 Sigma) [wt.%] |
|---|---|---|---|---|---|---|
| C | 6 | K-series | 34.48 | 27.19 | 50.57 | 6.28 |
| Si | 14 | K-series | 65.11 | 51.35 | 40.84 | 2.93 |
| Fe | 26 | K-series | 27.21 | 21.46 | 8.58 | 0.73 |
| | | Total: | 126.79 | 100.00 | 100.00 | |

***Fig. 8*** *Results of chemical analysis – sample No. 2 [5]*

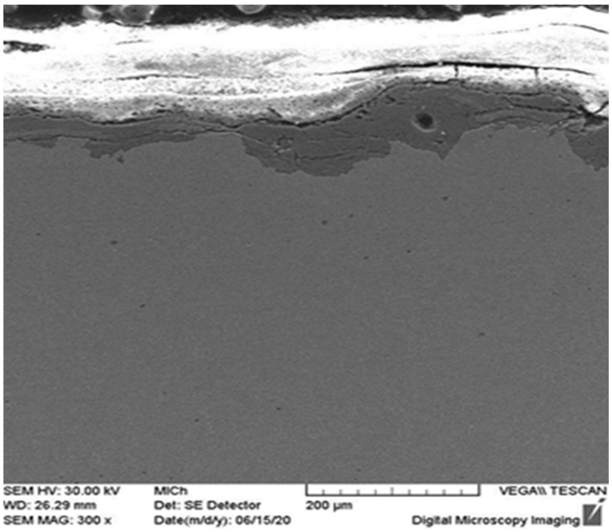



***Fig. 9*** *Detail of external pipe surface (300x zoom) sample No. 3 [5]*

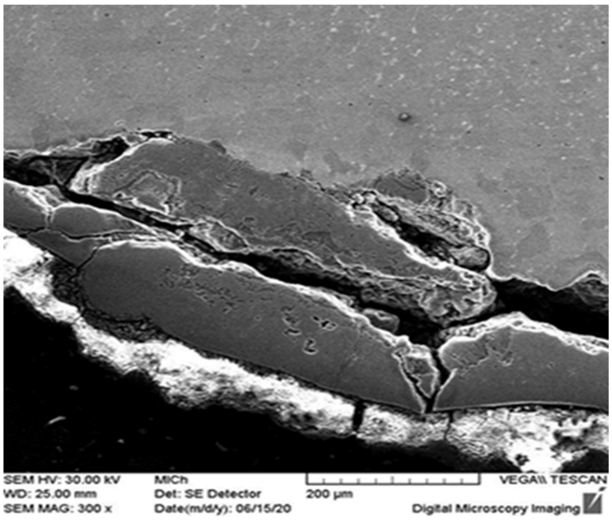


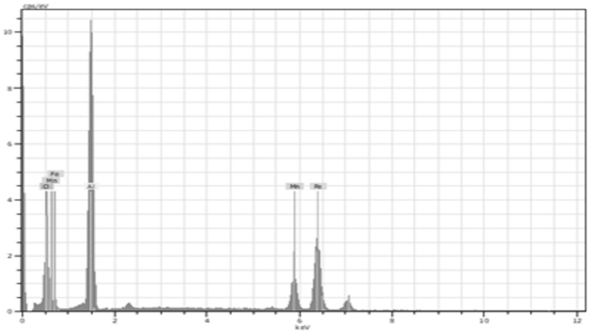

***Fig. 10*** *Detail of internal pipe surface (300x zoom) sample No. 3 [5]*

Chemical analysis Fig. 8 confirmed that this is a material that was not alloyed [5].

The sample No. 3 was obtained from a seamless steel pipe (DN 20) and metallographically prepared for observation. This pipe was in service for the longest time in a hot water heating system with a temperature gradient of 70/55 °C. The lifetime of this pipe was declared to be 48 years. Part of the pipe was cut from the rising pipeline in the interior. The surface of the pipe was protected with the basic and covering paint. These pipes are commonly made of weldable 11 353 material class, according to STN 42 0250 (for threading). The surfaces from the external pipe side (Fig. 9) and the internal pipe side (Fig. 10) exhibit quite noticeable non-uniformity. It is visible that layers or deposits of corrosion products are on the internal as well as external pipe surface. In addition, the internal surface contains deposits that, along with oxides, create significant stress states on the surface of the material resulting in the appearance of cracks and discontinuities. If such a layer (oxides plus deposits) peels off due to the movement of water, abrasive wear occurs in the given area. This steel is ferritic-pearlitic with a relatively high occurrence of inclusions based on sulfides and aluminates and it was also confirmed with the results of the chemical analysis (Fig. 11 a, b) [5].

The sample No. 4 is made of seamless steel pipe (DN 25) and the given pipe was in service for 20 years in a hot water heating system with a temperature gradient of 70/55 °C. The sample was cut from a horizontal pipeline in a hot water pipeline channel under the ground with and there was also the thermal insulation used (mineral wool with glass fibers and wrapped in aluminum foil) to protect and to eliminate or reduce the heat loss. Relating to pipe, the declared material is 11 353 material class, according to STN 42 0250 (for threading and welding). The non-uniform oxidation layers are visible on the surface of the pipe and it stands for deformation of geometry. The ripple effect is visible for the overall external surface of the material. In addition, the given surface was ruptured or damaged. The given external surface of the material for the pipe was attacked by uniform intercrystalline corrosion with the different depths (Fig. 12). Both ferrite and pearlite grains are uniform with the occurrence of a low number of inclusions in the microstructure. The internal surface of the given investigated pipe has specific noticeable a corrosion layer, which penetrates into the material and by this way, there are the changes in properties. Furthermore, a decarburized layer of the base (substrate) material was observed under the corrosion deposit (Fig. 13) [5].

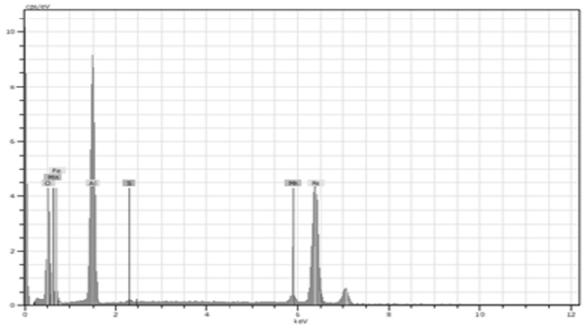
a)




Spectrum: 2

| El | AN | Series | unn. C [wt.%] | norm. C [wt.%] | Atom. C [at.%] | Error (1 Sigma) [wt.%] |
|---|---|---|---|---|---|---|
| O | 8 | K-series | 32.36 | 31.40 | 50.62 | 4.30 |
| Al | 13 | K-series | 36.60 | 35.51 | 33.94 | 1.87 |
| S | 16 | K-series | 0.46 | 0.44 | 0.36 | 0.05 |
| Mn | 25 | K-series | 1.48 | 1.44 | 0.67 | 0.07 |
| Fe | 26 | K-series | 32.16 | 31.21 | 14.41 | 0.85 |
| | | Total: | 103.06 | 100.00 | 100.00 | |

b)




Spectrum: 1

| El | AN | Series | unn. C [wt.%] | norm. C [wt.%] | Atom. C [at.%] | Error (1 Sigma) [wt.%] |
|---|---|---|---|---|---|---|
| O | 8 | K-series | 37.99 | 35.11 | 54.03 | 5.15 |
| Al | 13 | K-series | 39.71 | 36.70 | 33.49 | 2.03 |
| Mn | 25 | K-series | 8.68 | 8.03 | 3.60 | 0.27 |
| Fe | 26 | K-series | 21.82 | 20.16 | 8.89 | 0.60 |
| | | Total: | 108.20 | 100.00 | 100.00 | |

***Fig. 11*** *a) Results of chemical analysis for inclusions: m1 – sample No. 3 [5]; b) b Results of chemical analysis for inclusions: m2 – sample No. 3 [5]*

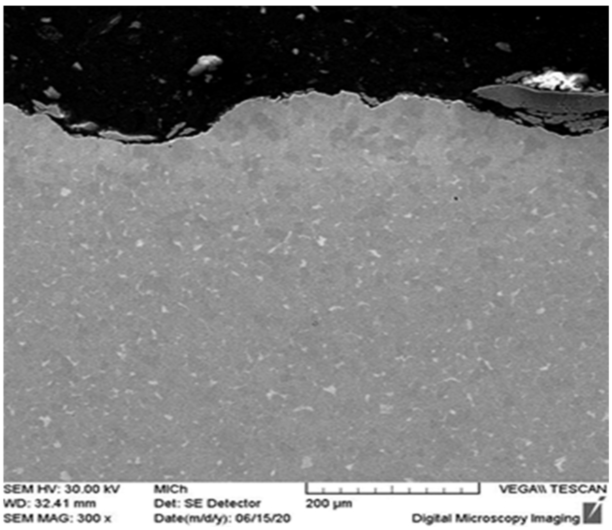



***Fig. 12*** *Detail of external pipe surface (300x zoom) sample No. 4 [5]*

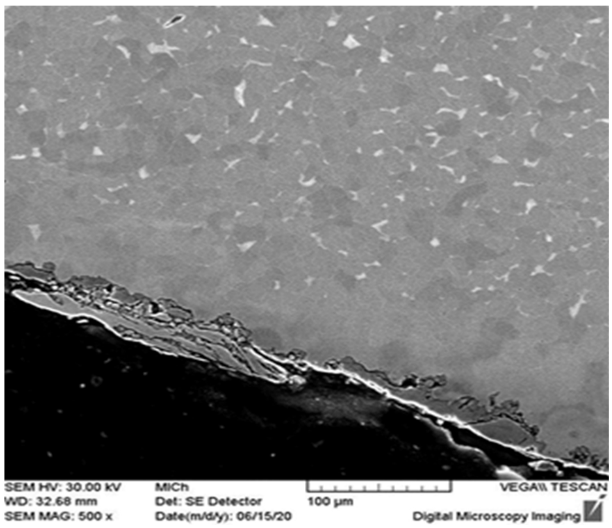



***Fig. 13*** *Detail of internal pipe surface (500x zoom) sample No. 4 [5]*

## 7 Final evaluation and recommendation

Using SEM, the microscopic analysis provided important information about the structure, purity and wear of the metal materials from which the examined pipes for the aqueous medium were made. This information is crucial for their evaluation and prediction of degradation that shortens the lifetime of pipes. The results from evaluation of samples for seamless steel pipes confirmed that all the process of corrosion was mainly on the surfaces and on the internal surfaces of pipes in service, there was noticeable deposits from the aqueous flowing medium. The surface (external and internal) of sample, designated as No. 1 (pipe which was not in service before), had a quite high roughness due to the process of forming technology during production. The sample designated as No. 2, showed intercrystalline corrosion, cracks and material peeling from the internal surface after 20 years of use in a hot water heating system. The oldest pipes – pipes used in a hot water heating system for 48 years and more (samples designated as No. 3 and No. 4) had a decarburized subsurface area and therer was the occurrence of inclusions based on sulfides and aluminates on the internal side of surface. The knowledge obtained from the research of the materials for pipes for the water medium provides valuable information which is necessary for the design, production and maintenance of metal components in the hot water heating system. Based on the knowledge gained, we recommend:

- The regular inspection and maintenance of metal hydraulic elements, especially those that interact with the water medium,
- The evaluation of currently used materials for the production of water pipes with regard to their resistance to corrosion and wear, depending on the specific application,
- The implementation of appropriate coatings/paints and protective insulation to reduce the risk of atmospheric corrosion and mechanical wear,
- The monitoring the development of wear over time,
- The regular control of the water medium, thermal conductivity values, occurrence of chemical elements and water pH.

The obtained results also show that seamless steel pipes can reach the prescribed service life or lifetime of 50 years. An important factor affecting their service life is the flowing medium and its temperature, hardness, conductivity and pH. The attention should also be paid to pipe placement through ceilings, walls of building structures and assembly channels or shafts, where atmospheric and bacterial corrosion occurs very often. Based on the results of microscopic examination, we can conclude that it is possible to predict the service life or lifetime of pipes (pipes) on the basis of the elimination of manufacturing and assembly errors. The results and knowledge gained from the work can be used in the areas of technical practice, scientific research, but also pedagogical activities.

In the field of technical practice, we can use the results and knowledge of the research to design the type of material for the reconstruction of the heating system, to identify critical aspects that determine the occurrence of subsequent degradation processes, to design processes and parameters for the safe operation of hot water heating.

In the field of scientific research, we can use the results and knowledge to confirm the service life of steel pipes when exposed to the surrounding environment, to design a procedure for predicting the failure of metal hot water pipes based on the knowledge gained from the operated heating system, and to continue research in comparing the use of materials in comparison with polymers and composites.

In the field of pedagogical activity, we can use the work as a tool for clarifying concepts, processes and parameters for workers who work in the field of sanitary and technical installations, and to use the results of the work as a teaching aid (textbook) for teachers of specialized subjects, masters of vocational education, and in theoretical and practical teaching of secondary school students with a specialization in technical building equipment.

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