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CORROSION EFFECTS ON PIPES MADE OF Cr-Mo STEEL FROM CENTRAL HEATING SYSTEMS

Vasile IORDACHESCU[1,*], Ion Mihai VASILE[2], Ionelia VOICULESCU[3]

[1)] Eng., PhD Student, Quality Engineering and Industrial Technology Dep., National University of Science and Technology POLITEHNICA Bucharest, Romania

[2)] Lecturer, PhD, Quality Engineering and Industrial Technologies Dep., National University of Science and Technology POLITEHNICA Bucharest, Romania

[3)] Prof., PhD, Quality Engineering and Industrial Technologies Dep., National University of Science and Technology POLITEHNICA Bucharest, Romania

Abstract: *The paper refers to the analysis of the factors contributing to the occurrence of corrosion, one of the main degradation mechanisms affecting the reliability and service life of Cr-Mo steel pipes used in thermal energy production facilities. These steels, appreciated for their resistance to high temperatures and good creep behavior, are often exposed to aggressive environments characterized by high temperature (160-450°C) and pressure (140-200 bar), as well as by the presence of oxygen, water vapor and corrosive compounds, such as sulfates and chlorides. In most cases the combined action of these factors leads to the formation of oxide layers, the occurrence of pitting corrosion and the thinning of the pipe walls, which can compromise structural integrity and operational safety. The paper investigates the aspect and amplitude of corrosive attacks observed in Cr-Mo steel pipes in service, under various working conditions. Pipe samples taken from decommissioned district heating installations, which had operated for long periods of time ranging from 27 to 41 years, were examined in cross-section, measuring the thicknesses of the oxidized layers and the microhardness. It was found that the thickness of the oxide crusts depends on the alloy class of the steels, the nature of the working environment and the operating temperature.*

Key words: *corrosion, heat-resistant steels, microstructure, microhardness.*

1. INTRODUCTION [1]

The proper functioning of semi-refractory steels intended for use at high temperatures (200–650 °C) depends on their mechanical and microstructural characteristics, which are influenced by the nature of the working environment, the service life, the material's ability to form protective surface layers, resistance to hydrogen diffusion, etc. [1–3].

The main structural instability phenomena that lead, over time, to the deterioration of strength and ductility characteristics are [4, 5]:

a) graphitization of carbon existing in the metallographic constituents (the Fe-Fe3C equilibrium diagram is a metastable diagram in relation to the stable Fe-C diagram, highlighting, at very long periods of maintenance at high temperatures, the possibility of decomposition of cementite into free carbon). This graphitization is achieved by diffusion of carbon, its accumulation in the form of graphite nests, which results in a drastic decrease in strength and plasticity characteristics;
b) spheroidization and dimensional increase of Mo, CrMo, CrMoWV carbides, depending on the alloying base of the steel; during operation over long periods, spheroidization and disintegration of pearlite in the microstructure occurs, with cementite lamellae transforming into spherical carbides, of the M23C6 and M7C3 type [6];
c) the diffusion of alloying elements from inside the grains to the grain boundary, with the formation of intermetallic compounds;
d) accumulation of impurities (P, Sb, Sn, Ar) in the intergranular layer which causes loss of microstructural integrity. This requires maintaining the level of these chemical elements below 0.01% [7].

Alloying with Cr contributes to strengthening the metal matrix by forming solid solutions, as well as increasing resistance to corrosion and high temperatures. Cr promotes the formation and stabilization of mixed carbides, if present in low concentrations (≈ 0.5%Cr) [1, 8]. For medium concentrations (< 5%Cr) or higher (> 9%Cr), it increases resistance to oxidation in air during exposure at high temperatures. Too high chromium concentrations can generate excessive hardening and cracking in the heat-affected zones of the welds.

In commercial Cr-Mo-steels containing 0.5% to 1% Mo, the increase in Cr content is more significant in terms of hot mechanical characteristics, for the chromium concentration values between 0–2.25% Cr. Under creep conditions, the chromium concentration that ensures maximum resistance at high temperatures is about 2.25% Cr. Based on these considerations, the compositional recipes of the most widely used semi-

* Corresponding author: Splaiul Independenței 313, 060042, Bucharest, Romania,
E-mail address: *ing_valy@yahoo.com* (V. Iordachescu).

refractorysteels alloyed with Mo or Cr–Mo were developed, the most representative being those with a chemical composition of 0.5% Mo and 1% Cr or 0.1% Mo and 2.25% Cr [9–11].

Molybdenum and chromium are alphagenic alloying elements that stabilize the ferritic domain and tend to reduce the austenitic domain. Iron and chromium form, at high temperatures, an isomorphic series of solid solutions with a BCC. (Body Centered Cubic) crystal lattice. The heat resistance of steels alloyed with Cr and Mo is determined mainly by the Mo content, which is the main element responsible for increasing the stability during tempering, by reducing the coalescence tendency of carbides and favouring their fine dispersion in the low-alloyed metallic matrix. Finely dispersed carbides exhibit a blocking effect on grain boundaries and limit the sliding effects on the surfaces of ferrite crystals [12]. Steels alloyed with Mo and Cr have better strength and impact toughness than steels alloyed with Mo and a more uniform hardness distribution [13].

Another potential factor influencing the cracking of Cr-Mo steels is the tendency for accelerated diffusion and accumulation of hydrogen protons at the crack tip, influencing their propagation mode under thermo-mechanical fatigue conditions [14, 15]

Superficial oxidation and the occurrence of chemical corrosion are the most common phenomena in industrial applications [16–18]. Although their effects seem to have little significance on the mechanical characteristics, under certain stress conditions it is possible to perforate or crack the metal wall, even for very low values of material loss and at low corrosion rates.

The important influencing factors in this case are the concentration of dissolved oxygen, the temperature and pH values. This damage mode occurs in heat exchanger tubes and in steam turbine blades. Research has shown that the presence of 2% Mn in steels can reduce the driving force for the decomposition of FeO through the eutectoid reaction [19].

In extreme cases, narrow and extremely long pitting zones can generate, under sufficiently high mechanical stress gradients, ruptures of the thin oxide film on the metal surface, leading to the development of cavities with openings between 1 and 5mm, called crevices. To limit the harmful effects of corrosion, systems are applied to retain oxygen from the working environment or to protect exposed external surfaces [20].

The main phases that occur in oxidation crusts on corroded surfaces contain the phases of hematite, magnetite and chromite, which have rhombohedral cubic, octahedral and spinel crystal structures, and hardnesses of approximately 6.2, 4.5 and 8.1 GPa, respectively. Increasing the Cr content in steel makes the thickness of the oxide layer thinner and the thickness of the chromite layer increase slightly over time [17].

The study involved research on samples taken from pipes used in industrial thermal installations operating in a mixed steam-water regime. Before taking cross-sections from the pipes, a visual examination of the corroded surfaces was performed.

For each sample, the oxide layer, the depth of the areas affected by corrosion, as well as the distribution of the predominant chemical elements (Fe, O, S, Cl) were analyzed. Research has shown that the heat-resistant steels used for the manufacture of district heating pipes selected in this study have specific characteristics regarding their behavior under stress conditions characterized by medium or high temperatures and medium or low corrosive environments (water, superheated steam, etc.).

In samples exploited for long periods, the corrosive attack of the working environment can be observed through the formation of corrosion caverns that selectively attacked the crystalline grains and the formation of crusts made up of oxides.

2. EXPERIMENTAL PROCEDURE

2.1. Alloys analyse

P265GH is a non-alloy steel for metal structures working under pressure, classified according to SREN10216. This steel has good plasticity, medium hardness, good cold bending behaviour and good weldability, and can be used even at medium temperatures. Carbon steel loses its resistance to flow, rupture and fatigue starting at 345°C. Steel is prone to stress relief cracking and cold cracking after welding. Its chemical composition contains (wt.%): C 0.20; Si 0.40; Mn 1.4; Cr 0.3; Mo 0.08; V 0.02; Ti 0.04; Ni 0.30.

16Mo3 is a high-quality chromium molybdenum steel used for the manufacture of vessels and related piping operating under high pressures and temperatures. The material is used as a weldable steel in the manufacture of industrial boilers and steel pressure vessels for the oil, gas and chemical industries. Due to the chromium and molybdenum content of the materials, 16Mo3 has excellent heat resistance and corrosion resistance characteristics. Its chemical composition contains (wt.%): C 0.20; Si 0.35; Mn 0.7; Cr 0.3; Mo 0.35; Al 0.02; Cu 0.30; Ni 0.30. This steel has a slight tendency to form hot cracks and is prone to stress relief cracking. In parts exploited for a long time, SEM microscopy studies performed in phase contrast have highlighted the presence of precipitates that form chains and bands at the boundaries of primary austenite grains, mostly linked to microcracks already oxidized [2].

12CrMoV3 is a low-alloy heat-resistant steel used as the main material for steam loop pipes operating at high temperatures and pressures. However, thermal aging (long-term thermal effect) greatly degrades the performance of 12CrMoV-3 steel. The microstructure of 12CrMoV-3 steel continuously changes during thermal aging, resulting in increased hardness and brittleness, thus affecting the service life and safety of the equipment. Its chemical composition contains (wt.%): C 0.15; Si 0.37; Mn 0.7; Cr 1.2; Mo 0.35; V 0.3; Al 0.045.

Precipitation and growth of carbides along grain boundaries are the main causes of degradation of the structure and properties of the material. Precipitation phenomena cause a decrease in the content of alloying elements (Cr, Mo) in the matrix and their depletion, which substantially reduces the strength of the material at working temperature in the case of 12Cr1MoV steel.

As a result of prolonged maintenance at working temperature, a series of microstructure changes may occur (spheroidization of precipitate-type phases;

diffusion of alloying elements Cr, Mo with the formation of carbides on grain boundaries that determine the weakening of mechanical strength; diffusion towards the grain boundary of components such as Sn, Sb, As, S, P, which determine embrittlement and crack formation, decarburization in the presence of hydrogen, etc.).

Slow creep deformation, which can produce stresses or even ruptures, breaks, perforations of the tubing wall, because of the effects of precipitation and formation of carbides with depletion of the base matrix.

Cold cracking in the presence of hydrogen, embrittled structures and residual mechanical stress states – requires preheating and/or post-welding heat treatments of the stress relief and dehydrogenation type. During post-welding cooling of these steels, hard martensite-type structures may appear, which are prone to cold cracking.

Oxidation resistance depends largely on the ability of the material to form its own protective oxide crust. During oxidation, unalloyed carbon steels form a crust composed of one or more layers of oxides such as wustite, hematite and magnetite, the nature of these crusts depending on the specific oxidation conditions. Alloying with Cr causes a change in the thickness and nature of the steel's own oxide crust, favouring the formation of an iron-chromium spinel in low-alloyed ferritic steels with up to 3% Cr [20].

2.2 Sampling procedure

In this study, pipe samples were analysed that were taken from disused pipes that had been used for long periods in district heating systems (Fig. 1).

Fig. 1. The aspect of pipes extracted from the district heating system after different periods of operation at internal pressure and temperature.

Table 1

Operating conditions of analysed materials

Sample code	Dimensions, mm	Steel Trademark	Working period, years	Temperature, °C	Pressure, bar	Working environment
1S	Ø28 x 5	12CrMoV-3	41	350	140	water-steam emulsion
2S	Ø36 x 4	12CrMoV-3	41	350	140	
3S	Ø32 x 3.3	P265GH	33	250	200	boiler feed water
4S	Ø60 x 6	P265GH	33	250	200	
1G	Ø42 x 5	12CrMoV-3	26	160	140	water
1P	Ø35 x 3.4	16Mo3	27	450	140/200	water-steam emulsion
2P	Ø60 x 4	16Mo3	27	450	140/200	
4P	Ø35 x 3.4	16Mo3	1 year	450	140/201	water-steam emulsion

To analyse the effects of degradation of pipes exposed to corrosive working environments (water, superheated steam), pipe sections with various thicknesses and diameters, made of steels belonging to different alloy classes, were extracted. The main operating conditions of the analysed materials are presented in Table 1.

The samples were cut both longitudinal and in cross sections, using the IsoMet 4000 precision cutting machine (Buehler, Düsseldorf, Germany) were embedded in epoxy resin, ground with SiC papers, and mirror-polished with 0.3 µm alumina suspension according to ASTM E3-11 (2017) [21].

After polishing the surfaces, the samples were not attacked with a chemical reagent, to correctly assess the extent of the corrosive effects and to measure the thickness of the layer affected by oxidation/corrosion. Optical microscopy (Olympus GX51, Tokyo, Japan, equipped with AnalySIS program for images processing) was used to characterize the alloys. Using the AnalySIS program, the thicknesses of the corroded layers of the analysed samples were measured.

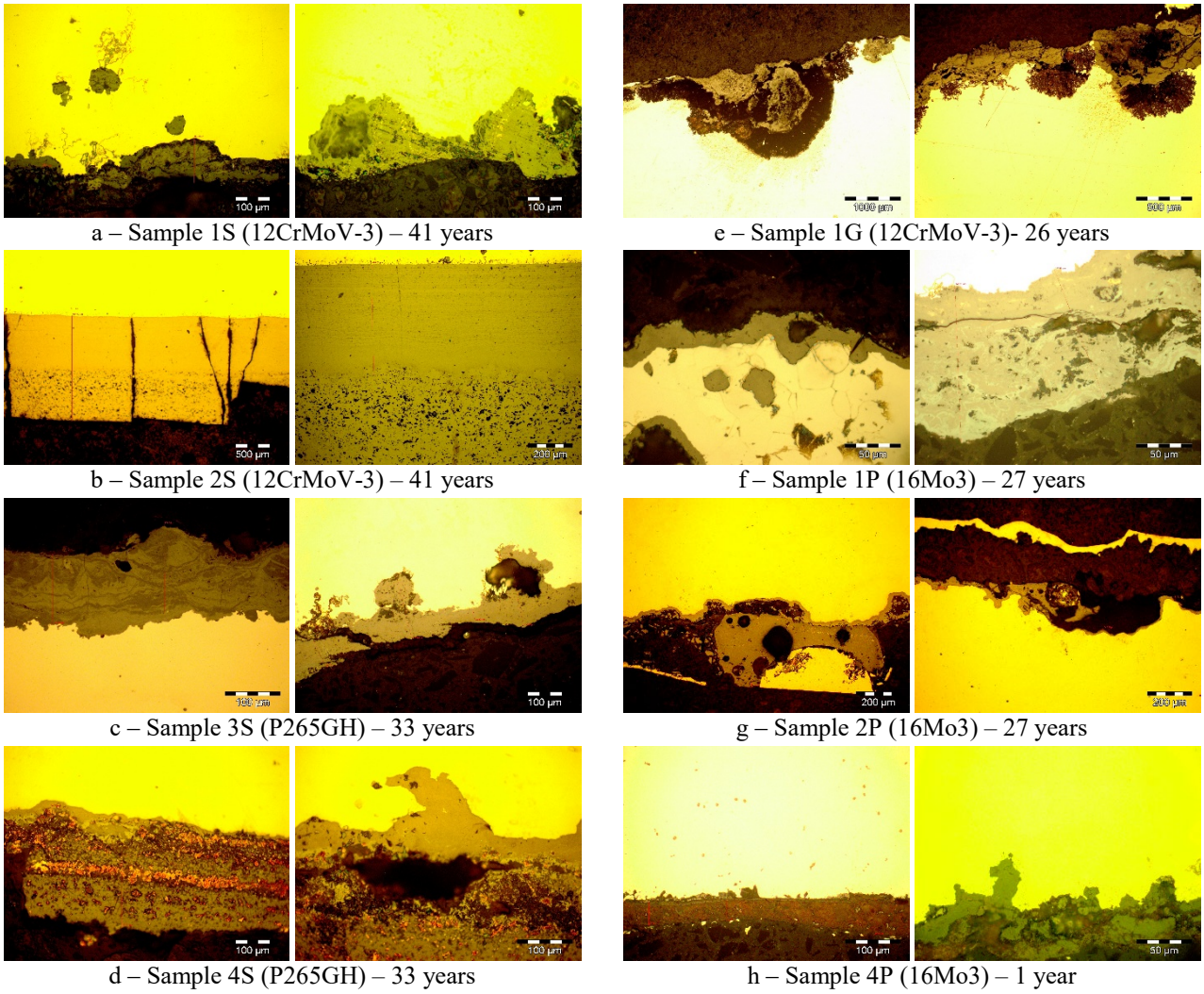
Microhardness tests were carried out using a Shimadzu HMV-2 tester (according to ISO 6507/ISO 4545/ASTM E384 [22]) using a 500 gf (HV0.5) load. Ten indentations were performed for each alloy, then average values, standard deviation and variation coefficient were calculated.

3. RESULTS AND DISCUSSION

3.1. Microstructure

The investigations were carried out on samples taken from pipes used in industrial thermal installations, operated in mixed steam-water regime. The cross-sectional appearance of the surface layers of the heat-resistant steel pipes analysed in this work is shown in Fig. 2.

One of the effects that occur in steels maintained for a long time at high temperatures is decarbonization. Decarbonization is a phenomenon that manifests itself by reducing the carbon content at the metal surface level



a – Sample 1S (12CrMoV-3) – 41 years

e – Sample 1G (12CrMoV-3)- 26 years

b – Sample 2S (12CrMoV-3) – 41 years

f – Sample 1P (16Mo3) – 27 years

c – Sample 3S (P265GH) – 33 years

g – Sample 2P (16Mo3) – 27 years

d – Sample 4S (P265GH) – 33 years

h – Sample 4P (16Mo3) – 1 year

Fig. 2. Cross-sectional aspect of the exposed surfaces after different periods of operation in water or steam-water emulsion environments.

that is exposed to the cumulative action of the temperature and pressure of the working environment. Essentially, decarbonization occurs because, at over 220 °C, the carbon dissolved in the ferrite solid solution or in pearlite reacts with H_2 or O, forming CH_4 or CO_2. Within this degradation process, O and H diffusion towards the interior of the metallic material occurs, with a predilection for the grain boundaries, and C diffusion towards areas rich in O and H, when the decarbonization rate is higher than the oxidation rate [23–25].

If the metal oxidation rate is high, the decarbonization effect is slow, because the oxidized layer acts as a barrier against the penetration of H and O from the working environment. This phenomenon is especially found in steels highly alloyed with Cr and Ni. In the presence of weakly oxidizing working environments, the decarbonization layer may be deeper, being developed because of a selective attack process of the areas of steel less alloyed with Cr and Mo.

Thus, in all the parts analysed in this study, on the surfaces in contact with the working environment (steam, water and steam emulsion) at temperatures between 160 and 450 °C, areas affected by corrosion appeared, consisting of oxide layers of different thicknesses, portions with selective attack on grain boundaries or extended craters in areas depleted in Cr and Mo.

In Figure 2,*a*, a tick crust (of 80 µm thickness on longitudinal and 200 µm on cross section) has been appeared, consisting of corrosion products and metal oxides, selective attac and corrosion path extended to other crystalline grains. In Fig. 2,*b*, a thick cracked crust (of 935 µm thickness in the longitudinal direction) can be observed and a 490 µm thick band where cementite dissolution occurred.

In Fig. 2,*c*, a thick and continuous crust (172 µm thick in the longitudinal direction) has been formed. Also, selective attack on grains is accompanied by the formation of caverns, extending to a distance of 145 µm below the exposed metal surface.

In Fig. 2,*d*, a thick and broken rust crust (202 µm thick in the longitudinal direction) can be observed and corrosion cavern with an opening of 200 µm placed under the zone of selective attack extended towards the grains.

In Fig. 2,*e*, in the areas where a selective attack developed on the crystalline grains destabilized by the diffusion of alloying elements (Cr, Mo), corrosion craters (600 µm deep) were formed.

In Fig. 2,*f*, a thick crust (of 140 µm thickness) made up of corrosion products and metal oxides, which encloses unattached crystalline grains occurred, while in Fig. 2,*g* similar crust (of 392 µm thickness) made up of corrosion products and metal oxides was formed, which encloses unattached crystalline grains can be seen.

Finaly, in Fig. 2,*h* the corrosion process begins by forming an uniform crust on the steel surface, which fragments and cracks during alternating working cycles. The selective attack on grains located at the exposed surface can be observed. In this case, the crust thickness has only 64 µm, because of short period of maintaining at 450 °C in working environment.

3.2. Microhardness measurements

Microhardness measurements were performed on the polished surfaces of the samples that were embedded in epoxy resin. The average values, the standard deviation and the coefficients of variation measured for each sample, are presented in Table 2.

As can be seen from the data presented in Table 2, there are variations in microhardness between alloy classes or between samples located in the same alloy class. Thus, samples 1S and 2S belonging to the 12CrMoV-3 brand, although they were exploited under the same conditions, for similar periods of time, have slightly different hardnesses. This difference may be due to the location of the measurement area in relation to the surface affected by corrosion.

As expected, the lowest average hardness value was measured for samples 3S and 4S, made of P265 GH steel, which have the lowest alloying level among all the analysed samples. The highest hardness values were measured for samples made of 12CrMoV-3 steel, then for those made of 16Mo3 steel, due to alloying with Cr, V and Mo.

4. CONCLUSIONS

The heat-resistant steels used for the manufacture of district heating pipes selected in this study have specific characteristics regarding their behaviour under stress conditions characterized by medium or high temperatures and medium or low corrosive environments (water, superheated steam, etc.).

Steels alloyed with Mo and Cr have higher corrosion resistance and higher creep resistance compared to unalloyed steel grade P265GH. In the crust formed during steam oxidation of low Cr and Mo alloyed steels, distinct regions appear. On the outside, a thick, porous layer is observed, beneath which there is a thinner and very adherent oxide layer, which develops mainly along the grain boundaries (sample 1S and 2S). Depending on the alloying level with Cr and Mo, the corrosive attack can manifest itself uniformly or selectively. The oxide crust is much thinner on the 12CrMoV-3 steel samples than on the other steels analysed. A less beneficial aspect is the selective attack on sensitized areas, with a depth of approximately 600 microns, although the operating period was shorter (26 years) and the temperature was only 160 °C.

In the case of low-alloy steels of type P265GH (samples 3S and 4S), the oxide crust is much more porous, allowing the development of craters at the interface with the metal material unaffected by corrosion. The thickness of the crust formed on the sample that operated for about 33 years at 250 °C in a boiler feed water environment reached about 200 microns.

In the case of 16Mo3 steel samples, the appearance of the oxide crust has distinct characteristics. It is a non-uniform mixture of corrosion products and portions of unaffected metal grains, after about 27 years of working in water steam emulsion (Fig. 2,*f* and *g*). The thickness of the oxide crust formed on this steel is the largest of the samples analysed, between 140 µm and 392 µm.

The hardness of the steels analysed in the paper depends on the alloying level and the working regime, with P265GH steel samples having the lowest hardness values and 12CrMoV-3 steel the highest values. The reduction in hardness may be caused by the decarbonization of surfaces in contact with the working environment at temperatures within the range of use of the analysed samples, namely 160–450 °C.

Table 2

Microhardness values for the analysed samples

Sample code	Average HV0.5	Standard deviation	Coefficient of variation
1S	207	2.65	1.28
2S	194	3.06	1.87
3S	161	1.00	0.55
4S	146	2.08	1.42
1G	178	1.73	0.97
1P	184	1.53	0.83
2P	172	1.53	0.89
4P	168	2.08	1.04

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