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XRD Analysis of Carbide Phase in Heat Resistant Steels

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In this article we have demonstrated the possibility to perform a quick and rather complete estimation of carbide phase evolution in power plant steels using X-ray diffraction. The distribution of alloying elements in the pipeline steels after long-time operation was examined. This important factor of steel's obsolescence and properties change was measured by identifying carbide phases from the XRD powder patterns. The accomplished XRD analysis of the carbide phases allows to state that only steel 12X1MΦ exploited for $1.42 \cdot 10^5$ hours and partly steel 12X1MΦ exploited for $1.51 \cdot 10^5$ hours meet the requirements of exploitation structure. However for the rest steels, the regenerative heat treatment should be projected. Additionally we have shown the possibility to do the regenerative heat treatment for our examined steels and the changes of carbide phases in these steles have been analyzed.

Keywords: XRD, power plant, heat resistant steel, creep, ferrite, pearlite, alloy carbide, cementite, regenerative heat treatment, electrochemical etching.

1. INTRODUCTION

Thermal power stations equipment operating resource depends on the changes of the metal microstructure that appear during the creep process. Therefore, when evaluating operating resource of power plant equipment components and predicting tools to extend it, the long-term periodical analysis of the permanent deformations of steels is performed [1].

Deformation and breakage of heat resistant steel often happens within the boundaries of the grains. Steel resistance for the heat is increased alloying the solid solution. A special alloy structure is formed where small carbide grains are inserted into the base solid alloy and positioned along the boundaries of the matrix grains. Intermetallic phase coherently tied with the matrix is also formed here. Dislocations are retarded when they confront those phases. The smaller are the phases and the bigger is the amount of them – the more intense is the retarding and the bigger is the resistance to the creep [2, 3].

The important factor of ferritic steels obsolescence and properties change is alloying elements diffusion from ferrite into the boundaries of matrix grains and primary carbide phase-cementite transformation and sequence of secondary coarse-grained carbide phases of more thermodynamically stable carbides rich in Mo, V and Cr precipitation [4]. These microstructural effects have been widely measured by optical microscopy [1], scanning electron microscopy (SEM) [5 – 7], transmission electron microscopy (TEM) [6 – 12], Auger electron spectroscopy (AES) [7, 11], X-ray diffraction (XRD) [9, 10, 12, 13] and energy dispersive spectroscopy (EDS) [5 – 7, 11].

Steam pipelines and boilers of Lithuanian thermal plants are exploited at 550 °C temperature for $2 \cdot 10^5$ hours and more and the steam pressure can reach to 15 MPa.

The initial structure of those pipelines steels 12X1MΦ (12Cr1MoV) and 15X1M1Φ (15Cr1Mo1V) consists of ferrite, pearlite and fine-grained carbide-cementite [1]. Eventually, high temperature and permanent pressure causes the diffusion of the alloying elements (Mo, V, Cr) from the ferrite matrix, alloying elements and cementite start to form coarse-grained special carbides along the boundaries of ferrite grains. Due to this reason the stability of the dislocation structure decreases (the density of dislocation decreases, the number of vacancies increases), 0.05 μm – 0.20 μm size micropores appear along the grains boundaries which further join and microcracks appear which start the cracking of the metal [1, 3].

The optical metallographic examination of carbides coarsening of steam line metal 12X1MΦ after various operation times has been shown in article [1].

Most of the analysis of steels microstructure was carried out using TEM [6 – 12], but it proved difficult to obtain thin foil specimens containing the large primary carbides and their surrounding regions. These areas were therefore analyzed in a SEM fitted with EDX [5 – 7].

The aim of this study is to test the XRD technique as a simple and quick but fairly complete and accurate carbide phase analysis of power plant steels. It is important for diagnostic of steel and making conclusions about suitability for further exploitation of equipment. The XRD examination of carbide phase transformation, precipitation and distribution has been carried out on two power plant steels.

2. EXPERIMENTAL

For our research we have used heat resistant steels taken from SPAB "Kaunas energija" branch Kaunas power plant. Steels were exploited at 550 °C temperature, when pressure was 14 MPa and operation time: a) $1.42 \cdot 10^5$ h for 12X1MΦ and $1.51 \cdot 10^5$ h for 12X1MΦ steam pipeline steel b) $1.59 \cdot 10^5$ h for 15X1M1Φ main steam collector boiler steel and $1.59 \cdot 10^5$ h for 15XM1Φ welding seam steel. Table 1 shows the chemical compositions of the alloys used.

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Table 1. Concentrations in wt% of the major alloying elements in 12X1M1Φ and 15XM1Φ steels

Steel	C	Cr	Mo	V	Mn	Si
12X1MΦ	0.15	1.2	0.35	0.3	0.7	0.37
15X1M1Φ	0.16	1.4	1.1	0.25	0.7	0.37

The XRD analysis was performed using diffractometer DRON-UM2. Diffraction patterns were recorded at 30 kV and 20 mA in 1 °/s detector's movement speed, intensity was measured every $2\theta = 0.02°$. Flat diffracted beam pyrolitic graphite monochromator was used to remove fluorescent X-rays. It becomes very relevant when analyzing iron compounds with CuK_α radiation. Noise that emerges because of the fluorescence (without monochromator) would be so large that only the most intensive diffraction maximums (e.g. from ferrite) would appear in the diffraction pattern.

The diffraction patterns were recorded automatically by a data acquisition system. The peaks obtained were identified with those available in PDF-2 data base [14].

The XRD analysis results of the heat resistant steels were compared to the experiments of metallographic microstructure, which were carried out by optical microscope "Olympus" with video camera Sony DXC-151 and NIH Image 1.61 computer program.

To accelerate the carbide coarsening the 15X1M1Φ steel samples were additionally heat treated for five days at 675 °C temperature, after that they were slowly cooled in the furnace. Other samples of the same steel were heat treated for 0.5 hour at 1000 °C temperature, some of them were cooled slowly, other were water – cooled to the room temperature and heat treated for 5 h at 400, 500, 600 and 700 °C and after that they were cooled quickly in the air to the room temperature.

У8 steel was used for choosing the etching technology.

Mechanical grinding was done in Buechler Ecomet II device. Polishing was done with polishing fluid Buehler Micropolish II 0.05 μm (γ-Al_2O_3 – water suspension).

In order to highlight the metal microstructure, the polished surface was etched by nital solution (4 % azotic acid solution in ethanol) – from 5 s to 2 min. and also the polished surface was etched by Marble (4 g $CuSO_4$ dissolved in 20 ml H_2O and 20 ml concentrated HCl added) solution from 15 s to 5 min.

First of all XRD patterns were recorded of the samples that were used in the optical microscopic analysis. Then, the same samples were chemically etched in Marble solution for 5 min. – 30 min. and then again their XRD patterns were recorded. Later various carbides were extracted by a selective electrochemical dissolution technique using the electrolytes: 7 % KCl + 0.5 % citric acid, 7 % NaCl + + 0.5 % citric acid and 5 % HCl solution, cathode was stainless steel, anode was sample, voltage 3 V – 5 V, current 0.3 A – 0.5 A. Electrochemical etching lasted from 15 s to 2 hours. Samples taken from the etching solution were washed by warm water's weak flush in order not to wash down the extracted carbide residue. Then they were dried in a hot air stream and their XRD patterns were recorded.

3. RESULTS AND DISCUSSIONS

The microstructure (Fig. 1) of heat resistant steels 12X1MΦ and 15XM1Φ looks like the one of the steel 12X1MΦ that is presented in the work [1]. Here also carbide "chains" (dark thicker fields) – coagulated carbides are clearly seen along the boundaries of the grains. Different is only the welding seam steel (Fig. 1, e), which is fine-grained because of different thermal impact, but still relatively many carbide zones are seen.

The clearest carbide chains are in the microstructure of the steel 12X1MΦ, which was exploited for $1.51 \cdot 10^5$ h (Fig. 1, b). Although this is not the longest service time, but probably conditions of exploitation were severer.

It was expected that in the microstructure of steel 15X1M1Φ ($1.59 \cdot 10^5$ h) that was heat treated for 5 days at 670 °C temperature and slowly cooled, would have clearly seen carbide "chains" (Fig. 1, f), but there were no differences from the microstructure of steels that were only in exploitation. This means that during the exploitation all carbon was bound to carbide precipitates.

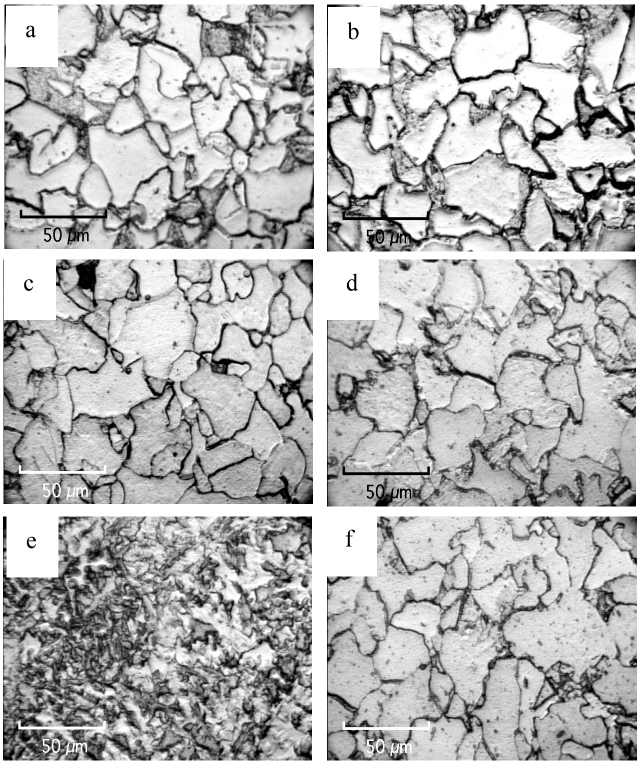

Fig. 1. Optical microstructure of the steel etched by 4 % nital solution for 2 min. – 3 min.: a – 12X1MΦ ($1.42 \cdot 10^5$ h), b – 12X1MΦ ($1.51 \cdot 10^5$ h), c – 15X1M1Φ ($1.59 \cdot 10^5$ h) kept for 0.5 h at 1000 °C temperature and slowly cooled, d – 15X1M1Φ ($1.59 \cdot 10^5$ h), e – seam steel, f – 15X1M1Φ ($1.59 \cdot 10^5$ h) kept for 5 days at 670 °C temperature and slowly cooled. In brackets – service time of the steel

In the steel 15X1M1Φ ($1.59 \cdot 10^5$ h) (Fig. 1, c), which was heat treated for 0.5 h at 1000 °C temperature and slowly cooled, carbide zones are also clearly seen. Probably, the time when the steel was kept at the certain temperature was too short and the carbide phases did not melt; or the cooling was too slow, so carbide phase formed newly.

X-ray diffraction patterns (Fig. 2) of the samples, that were used in microscopic analysis, are quite similar, and all visible very sharp diffraction peaks, that have interplanar spacings $d_{(100)} = 0.2027$ nm; $d_{(200)} = 0.14336$ nm and $d_{(211)} = 0.1171$ nm, belong to ferrite (α-Fe) cubic lattice. There are no other diffraction peaks, also typical to carbide phase. This confirmed the literature data that carbide phase in the steel could be identifiable by XRD only if there are 5 or more percents of it.

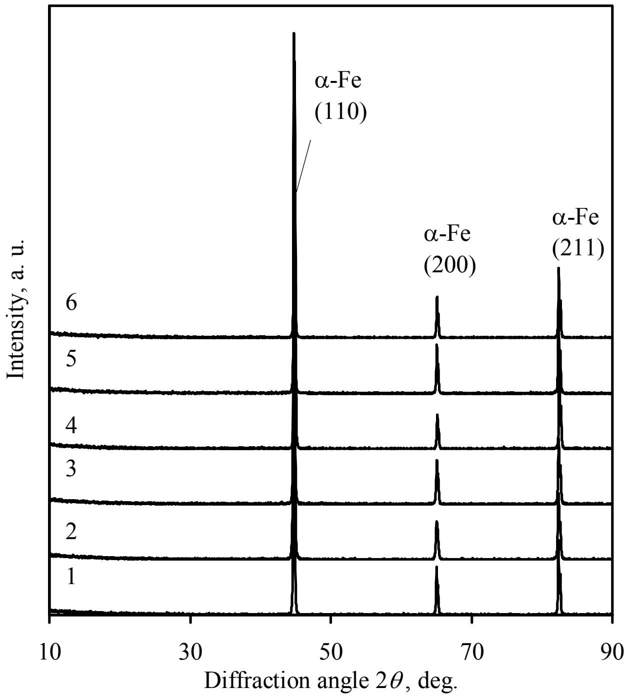

Fig. 2. X-ray diffraction patterns of the steel etched by 4 % nital solution for 2 min.– 3 min.: 1 – 12Х1МФ ($1.51\cdot10^5$ h), 2 – seam steel 15Х1М1Ф ($1.59\cdot10^5$ h), 3 – 12Х1МФ ($1.42\cdot10^5$ h), 4 – 15Х1М1Ф ($1.59\cdot10^5$ h), 5 – 15Х1М1Ф ($1.59\cdot10^5$ h) kept for 0.5 h at 1000 °C temperature and slowly cooled, 6 – 15Х1М1Ф ($1.59\cdot10^5$ h) kept for 5 days at 670 °C temperature and slowly cooled. In brackets – service time of the steel

Therefore, in order to highlight the carbide phase, we tried various chemical and electrochemical etching methods.

Etching the heat resistant steel in Marble solution was not effective to show up the carbide phase. After etching the steel 15Х1М1Ф (exploited for $1.59\cdot10^5$ h) for a short time (2 min. – 3 min.) only traces of carbide phase can be seen in XRD patterns (Fig. 3, curves 1 and 2), probably it is molybdenum carbide $M_6Mo_6C_2$, where M can be any alloying element Cr, V, M, and Fe or combination of them.

Etching for a longer time (to 15 min.) there was no increase of the carbide phase diffraction peaks, and the surface of the sample encrusted with copper (Fig. 3, curve 6).

Electrochemical polishing for 30 minutes in 5 % oxalic acid solution was not effective as well. No traces of carbide phase diffraction peaks are seen in X-ray diffraction pattern (Fig. 3, curve 5).

More effective appeared to be etching in 5 % NaCl solution (Fig. 3, curve 3) – here we can more clearly identify molybdenum carbide.

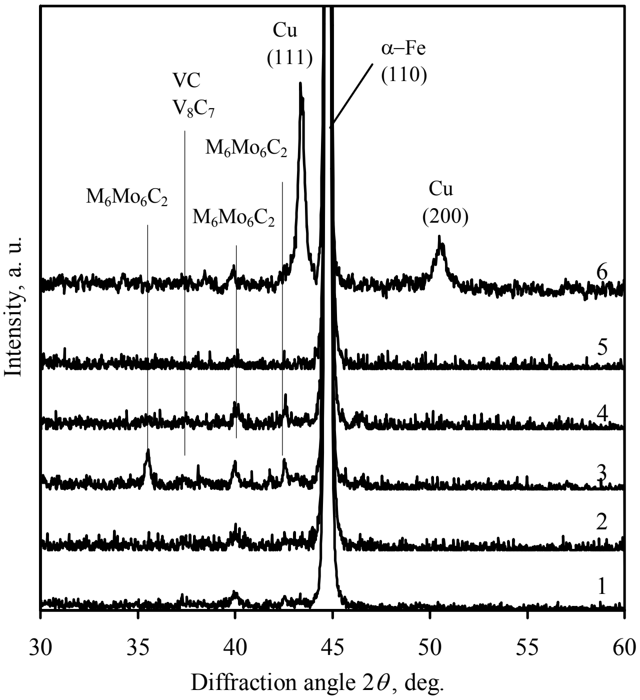

Fig. 3. X-ray diffraction patterns of the steel 15Х1М1Ф (exploited for $1.59\cdot10^5$ h): 1 – the surface of the sample is polished 0.02 mm and etched for 2 min. in Marble solution, 2 – polished 0.05 mm layer and etched for 3 min. in Marble solution, 3 – the surface processed like the 2nd sample and etched electrochemically for 10 min. in 7 % NaCl + 0.5 % citric acid solution, 4 – the surface processed like the 3rd sample and etched for 30 min. in nital solution, 5 – the surface polished mechanically and for 30 min. polished electrochemically in 5 % oxalic acid solution, 6 – the surface polished mechanically and for 15 min. etched in Marble solution

To set down a more effective etching regime we chose the steel У8. Carbide Fe_3C – cementite is typical for this steel, and the amount of Fe_3C in this steel is up 12 %, i.e. more than is needed to identify this phase by XRD.

As electrochemical method of etching appeared to be the most effective, so, after trying three etching solutions: 7 % KCl + 0.5 % citric acid solution, 7 % NaCl + 0.5 % citric acid solution and 5 % HCl solution, we chose the latter, because by effectiveness it is as good as the recommended [15] 7 % KCl + 0.5 % citric acid solution and also it is cheaper and easier to prepare.

In diffraction patterns of the steel У8 we can clearly identify Fe_3C peaks (Fig. 4, curve 4) after etching 90 s in 5 % HCl solution, and after 60 min. etching ferrite diffraction maximum completely disappears (Fig. 4, curve 8 and Fig. 5, curve 1).

Fig. 5 shows the enlarged diffraction pattern of the steel У8 that was etched electrochemically for 1 hour in 5 % HCl solution (Fig. 5, curve 1), this pattern has almost no differences from the pattern of Fe_3C standard, taken from PDF – 2 data base (Fig. 5, curve 2). So, for this particular steel electrochemical etching, which highlights carbide phase, is very effective.

By applying previously mentioned electrochemical method of etching to highlight the carbide phase of heat resistant steels we got the results that are shown in Fig. 6.

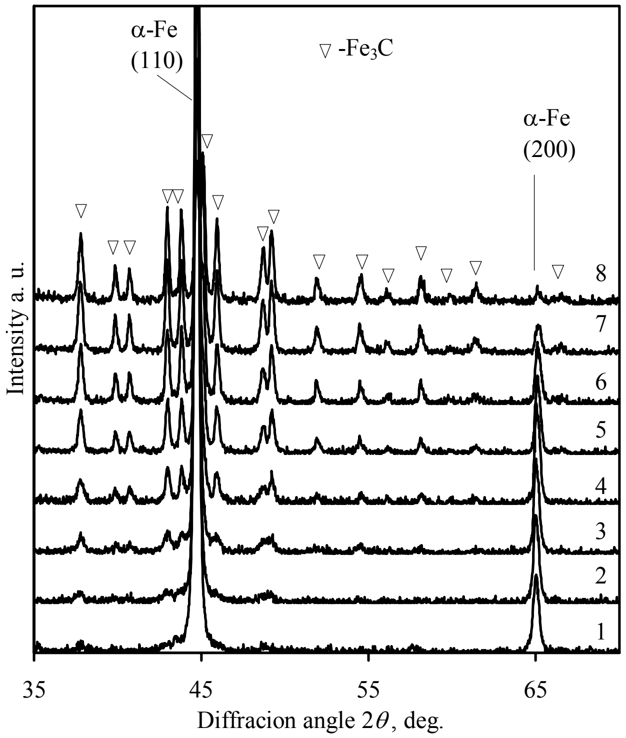

Fig. 4. X-ray diffraction patterns of the steel У8. Surface mechanically polished – 1; electrochemically etched in 5 % HCl solution: 15 s – 2; 45 s – 3; 90 s – 4; 3 min. – 5; 5 min. – 6; 15 min. – 7; 1 h – 8

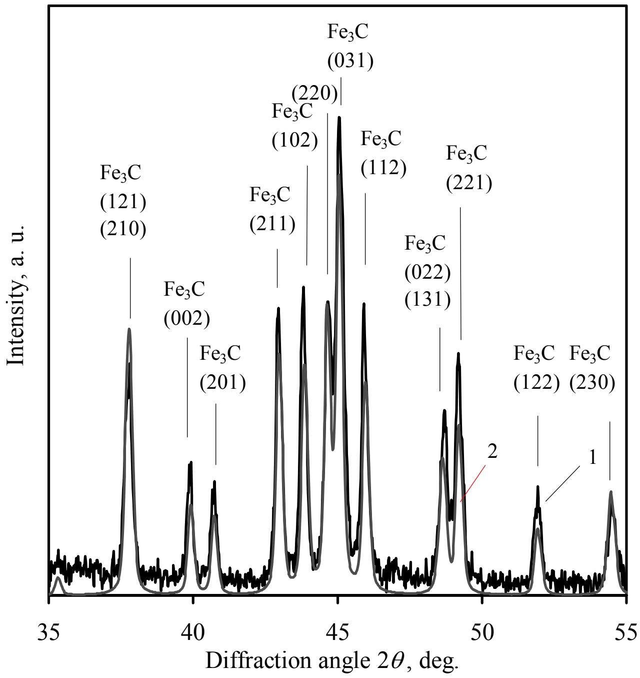

Fig. 5. X-ray diffraction pattern of the steel У8 etched electrochemically for 1 hours in 5 % HCl solution – 1 and Fe$_3$C standard diffraction curve taken from PDF-2 data base – 2

Differently from optical microanalysis, here we can clearly identify carbide phases and distribution of alloying elements in various carbide compounds depending on different exploitation and thermal regimes.

We can see that steel 12X1MΦ exploited for $1.42 \cdot 10^5$ hours (Fig. 6, curve 1) preserved the initial steel structure best of all, i.e. here appeared relatively intensive diffraction peaks of cementite (Fe_3C), diffraction peaks of vanadium carbide (VC, V_8C_7) and molybdenum carbide (Mo_2C) are not sharp, and diffraction peaks' intensity of the carbide phase ($M_{23}C_6$) formed from other alloying elements is even lesser, where M stands for a mixture of iron and substitutional solute atoms (Cr, V, Mo).

If compared to above-mentioned steel, in steel 12X1MΦ, that was exploited for $1.51 \cdot 10^5$ h (Fig. 6, curve 2), the amount of cementite decreased more than twice (identified by peaks' intensity), diffraction peaks of VC, V_8C_7 and Mo_2C increased, also chromium carbide (Cr_7C_3) and special chromium carbide (Cr, Fe)$_7C_3$, that has got iron, diffraction peaks appeared.

In diffraction pattern of steel 15X1M1Φ, exploited for $1.59 \cdot 10^5$ hours (Fig. 6, curve 4), differently from above-mentioned steels, intensive molybdenum carbide (M_6MoC_2) diffraction maximums can be seen. Here M – alloying element. PDF-2 data base gives compound Co_6MoC_2 (cubic lattice, space group Fd3m No. 227), which interplanar spacings d_{hkl} best suit our recorded pattern, only slightly biased. It means that the found compound's lattice parameters have only a slight difference, and instead of Co here is another element, and there is no Co in this steel. Another compound's – Fe_3Mo_3C – lattice parameters are also very close to our investigated steel's carbide phase, but here the difference is bigger than with Co_6MoC_2. This means that no data in PDF-2 data base were found about the compound that formed in our case.

The most intensive peaks of carbide phase can be seen in seam steel diffraction pattern (Fig. 6, curve 5). Here VC, V_8C_7 diffraction peaks intensities are approximately equal to those of steel 12X1MΦ ($1.51 \cdot 10^5$ h) (Fig. 6, curve 2), but very intensive peaks of M_6MoC_2 appeared and some sharp diffraction peaks remained unidentified. They could be attributed to boron carbides BC_4, $B_{13}Cr$ (Mo, M) C, but additional elemental examination of composition would be needed for more precise analysis.

By the way, seam steel's phase composition and the amount of carbides (Fig. 6, curve 5) looks very similar to steel 15X1M1Φ, that was exploited for $1.59 \cdot 10^5$ h, kept for 5 days at 675 °C temperature and slowly cooled (Fig. 6, curve 6). In XRD patterns of them intensity of diffraction peaks of the carbides M_6MoC_2 and (Cr, Fe) $_7C_3$ are almost the same, and VC, V_8C_7 – bigger in the steel that was affected by high temperature. Furthermore, in the last-mentioned steel's diffraction pattern the very intensive peaks of carbide $M_{23}C$ are visible. So, by affecting steel in this way, the process of carbide forming and their coagulation is stimulated.

In XRD pattern of the steel 15X1M1Φ, which was exploited for $158 \cdot 10^5$ h, kept for 0.5 h at 1000 °C temperature and slowly cooled (Fig. 6, curve 3) very intensive VC, V_8C_7 peaks are seen, the peaks of other carbides are hardly seen or totally vanished. It means that even in such temperature carbides had to melt, but slow cooling enabled forming of thermodynamically most stable carbides.

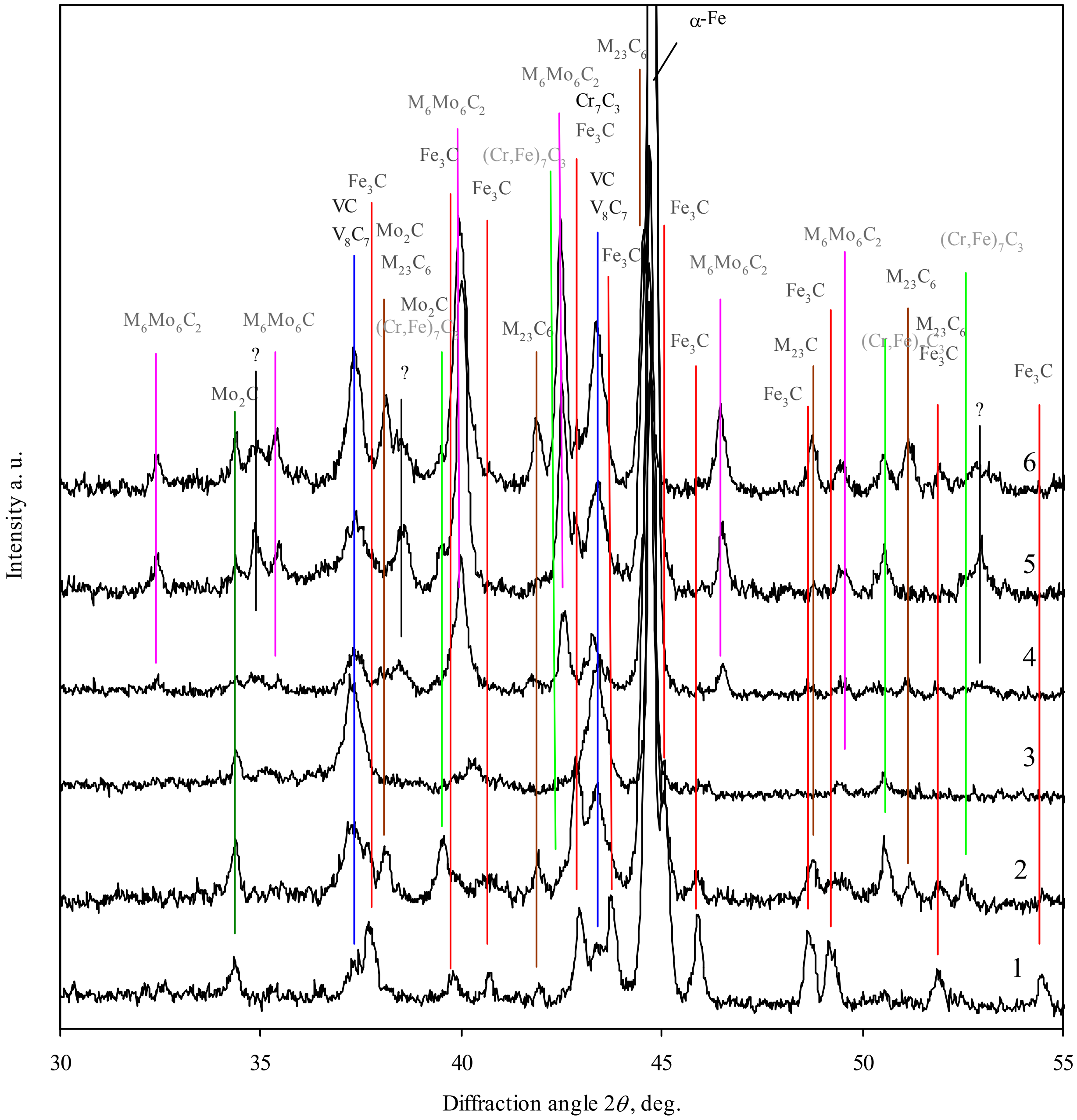

Fig. 6. X-ray diffraction patterns of heat resistant steel etched electrochemically for 105 min. in 5 % HCl solution: 1 – steam pipeline steel 12X1MΦ exploited for $1.42 \cdot 10^5$ h, 2 – steam pipeline steel 12X1MΦ exploited for $1.51 \cdot 10^5$ h, 3 – main steam collector's steel 15X1M1Φ exploited for $1.59 \cdot 10^5$ h, kept for 30 min. at 1000 °C temperature and slowly cooled, 4 – 15X1M1Φ steel exploited for $1.59 \cdot 10^5$ h, 5 – welding seam steel, 6 – steel 15X1M1Φ exploited for $1.59 \cdot 10^5$ h, kept for 5 days at 675 °C temperature and slowly cooled

During the service exposure mostly aged main steam collector's steel 15X1M1Φ exploited for $1.51 \cdot 10^5$ h was chosen to show the possibility to do the reconstruction of steel crystalline structure for the power plant components' life extension. For this reason steel was heat treated for 0.5 h at 1000 °C and quickly cooled in water, after that it was heat treated for 5 h at 400, 500, 600 and 700 °C and quickly cooled in air to room temperature. Their diffraction patterns after electrochemical etching in 5 % HCl are shown in Fig. 7.

An as-quenched structure of 15X1M1Φ steel consists of martensite and austenite (Fig. 7, curve 1), and carbide phase is dissolved (no diffraction peaks).

After tempering in above-mentioned four stages the martensite loses tetragonality, austenite decomposes and carbide phase forms in steel crystalline structure (Fig. 7, curves 2 – 5).

During the heat treatment of steel at 400 °C, the cementite diffraction peaks appear in diffraction pattern (Fig. 7 curve 2).

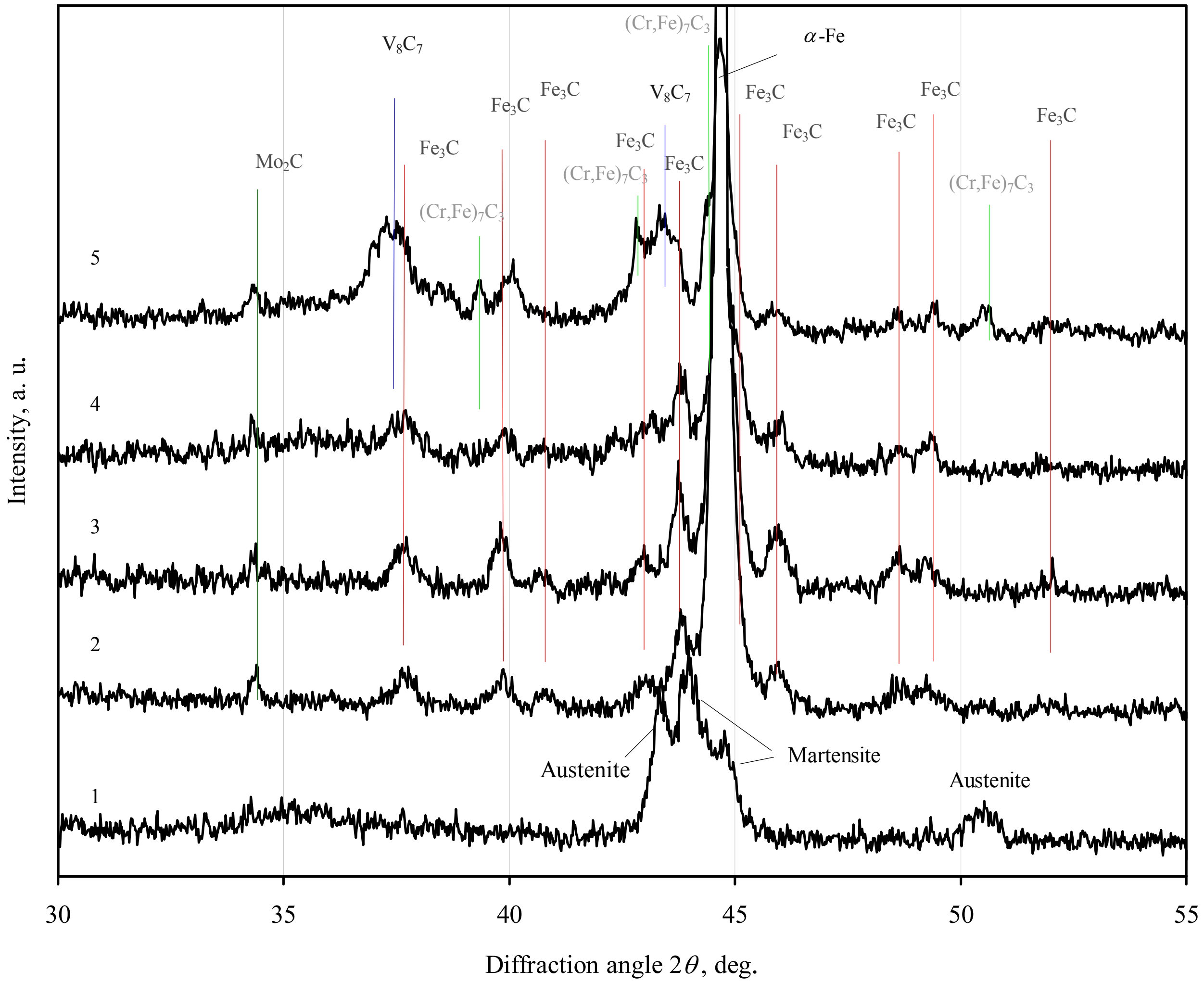

Fig. 7. X-ray diffraction patterns of steel 15X1M1Φ exploited for $1.59 \cdot 10^5$ kept for 0.5 h at 1000 °C, water – cooled to the room temperature – 1 and regenerative heat treated for 5 h at: 400 °C – 2, 500 °C – 3, 600 °C – 4 and 700 °C – 5

After tempering of steel at 700 °C, the thermodynamically more stable than cementite carbides (V_8C_7, (Cr, Fe)$_7$C3) already formed (Fig. 7, curve 5).

Diffraction pattern 3 in Fig. 7 shows the most perfect crystalline structure of cementite that allows to determine the most suitable temperature – 500 °C for regenerative heat treatment of aged 15X1M1Φ steel. However, in order to produce a stable microstructure of steel the higher regenerative heat treatment temperature is required, because tempering at a higher temperature than service temperature gives better long-term creep properties [2]. In this case for 15X1M1Φ steel, tempering at a temperature of approximately 600 °C would be preferable.

CONCLUSIONS

Electrochemical etching in 5 % HCl solution of low alloy power plant steels is an effective procedure to selectively extract carbide phase precipitates from steel matrix for XRD analysis.

Optical microscopy does not enable clearly distinguish the state of carbide phase precipitation.

Differently from the optical microscopy, the XRD technique enables to study properly the matrix and the various types of carbide precipitates formed at different heat treatment temperatures in both bulk specimens and selectively extracted samples. The important factor of steel's obsolescence and features change is alloying elements' diffusion from ferrite into the boundaries of matrix grains and the precipitating of the carbide phase. This factor can be measured by identifying carbide phases from X-ray diffraction patterns and the intensity of those peaks defines the amount of the carbide phases.

The accomplished XRD analysis of the carbide phase in heat resistant steels allows to state that only steel 12X1MΦ exploited for $1.42 \cdot 10^5$ h (Fig. 6, curve 1) and partly steel 12X1MΦ exploited for $1.51 \cdot 10^5$ h (Fig. 6, curve 2) meet the requirements of exploitation structure. Talking about the rest steels, the regenerative heat treatment should be projected following the recommendations given in literature [16, 17].

For life extension of power plant components made from steel 15X1M1Φ the most suitable restoration procedure of crystalline structure and properties of steel is heat treatment at 1000 °C, quenching and tempering at 600 °C.

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