Archives of Materials Science and Engineering

Volume 60
Issue 2
April 2013
Pages 72-81

International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Characteristics of changes in properties and structure of X10CrMoVNb9-1 steel due to long-term impact of temperature and stress

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Received 16.02.2013; published in revised form 01.04.2013

ABSTRACT

Purpose: this article presents the results of mechanical and structure testing of X10CrMoVNb9-1 (P91) steel in initial state and after annealing, creep tests and long-term service under creep conditions

Design/methodology/approach: The material for investigations was X10CrMoVNb9-1 (P91) steel in the form of two test pieces of tubes with dimensions of Ø355.6x50 mm and Ø325x38 mm in initial state and after 90,000 h service under creep conditions, respectively. The mechanical and structure testing was carried out on material in initial state and after long-term service under creep conditions. The microstructure was observed using scanning electron microscope.

Findings: The mechanical, creep and structure testing of steel in initial state has confirmed that the tested steel meets the requirements of PN-EN 10216-2. Long-term annealing as well as hardness and impact strength testing have allowed the influence of long-term impact of temperature and time on properties and structure of X10CrMoVNb9-1 (P91) steel to be evaluated.

Practical implications: The presented method can be used for evaluation and qualification of structural changes in power station boiler components operating under creep conditions.

Originality/value: The presented results of changes in mechanical properties, structure and precipitation processes are applied to evaluation of the condition of elements for further industrial service.

Keywords: Mechanical properties; Creep test; Structure; Degradation after annealing; Hardness; Impact strength; X10CrMoVNb9-1 (P91) steel

Reference to this paper should be given in the following way:

A. Zieliński, J. Dobrzański, Characteristics of changes in properties and structure of X10CrMoVNb9-1 steel due to long-term impact of temperature and stress, Archives of Materials Science and Engineering 60/2 (2013) 72-81.

PROPERTIES

1. Introduction

The basic requirement for all creep-resisting steels is that they should maintain, for a long time, the assumed mechanical properties, high-temperature creep resistance and heat resistance as the elements of safe (reliable) operation within the design working parameters of boiler [1-8]. For safety assessment of pressure equipment, in which new creep-resisting steels were used, it is essential that a number of tests should be carried out to allow the evaluation of their suitability for further service beyond the design work time under specified conditions [7-16].

This evaluation is enabled by materials characteristics, built for steels in initial state as well as after different times of exposure under conditions of long-term impact of temperature and stress [13,17-21].

In cooperation with other research centres in Poland and abroad (COST 522, 536) and Fabryka Kotłów Rafako S.A. - the leading manufacturer of boilers, the Institute for Ferrous Metallurgy performs the works on the advance and verification tests of new-generation steels for the power industry [2,7,8,15,17,19].

This paper presents the results of testing mechanical properties in initial state and after long-term impact of elevated temperature and stress on change in properties of X10CrMoVNb9-1 (P91) steel.

2. Material for investigations

The material for investigations was two test pieces of heavy wall tubes made from X10CrMoVNb9-1 steel in initial state, dimensions ϕ355.6x50 mm, and after 90,000 h service at a temperature of 540°C and pressure of 18 MPa, dimensions ϕ325x38 mm.

Chemical composition of test pieces with reference to the requirements EN 10216-2:2002 is summarised in Table 1.

3. Range of investigations

For material sampled from test piece of tube in initial state with diameter of ϕ355.6 mm and wall thickness of 50 mm the mechanical testing in initial state and after long-term annealing for up to 5.000 h was carried out. Analogously, the structure testing was carried out and life time was determined in abridged creep tests.

Similarly to material in initial state, the mechanical and structure testing was carried out and life time was determined in abridged creep tests for material of tube after 90,000 h service too.

4. Test results

4.1. Mechanical properties of X10CrMoVNb9-1 steel in initial state and after long-term annealing

The obtained test results of tensile strength TS, yield point YP, elongation and reduction of area in static tensile test at room temperature and at temperature levels of 200, 300, 400, 450, 500, 550 and 600°C for the tested steel in initial state are presented in Fig. 1, while the change in these properties after annealing at 650 and 650°C for 1000, 3000 and 5000 h is presented in Figs. 2 and 3, respectively. The results of impact energy KV at a temperature between -60 and +20°C for material in initial state are presented in Fig. 4, while the values of impact energy after annealing at 650 and 650°C for 1000, 3000 and 5000 h are presented in Fig. 5.

The results obtained in static tensile test at room and elevated temperature for steel in initial state comply with the requirements of PN-EN 10216-2 [3]. Long-term annealing for up to 5000 h, both at 600°C and 650°C, did not result in significant changes in mechanical properties. No significant changes in plastic properties were observed either. The reduction in impact energy after annealing for 5000 h runs at the level of approx. 10%. The impact strength within the temperature range between -60 and +20°C has revealed that the nil ductility transition temperature of the tested steel, according to the criterion of 27 J, is much below -60°C at which the impact strength is at the level of 70 J.

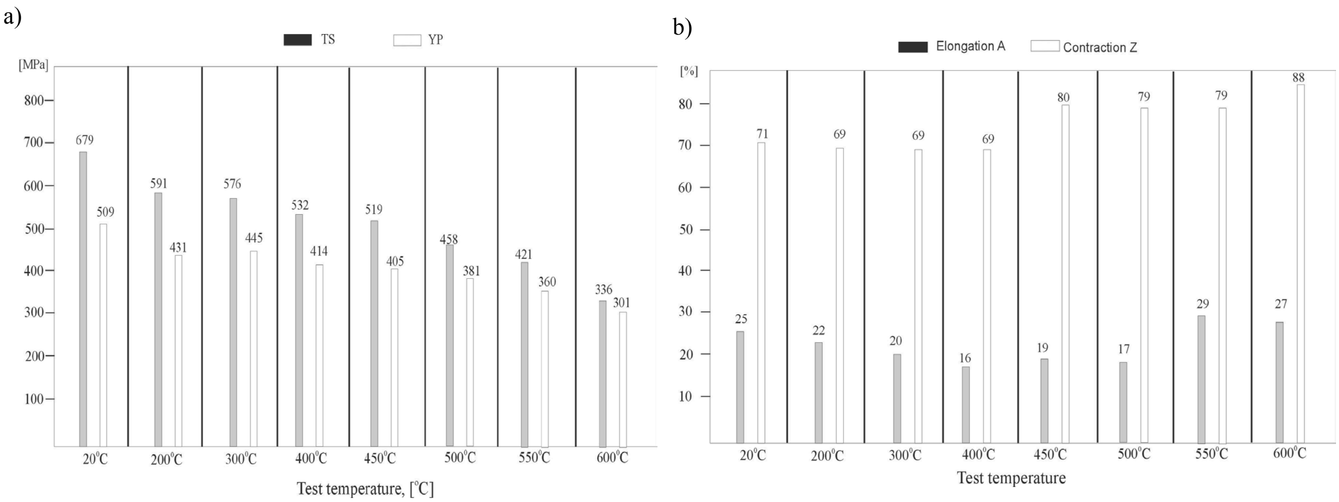

Fig. 1. a) Values of tensile strength and yield point of tested steel in initial state; b) Values of elongation and reduction of area of tested steel in initial state

Table 1.
Material for investigation - chemical composition of tested X10CrMoVNb9-1 (P91) steel with reference to the requirements EN 10216-2

No	Chemical composition [%]												
	C	Mn	Si	P	S	Cr	Ni	Mo	V	Cu	Nb	Al	N
tube ϕ355.6x50 m	0.12	0.38	0.26	0.013	0.004	8.74	0.17	0.96	0.23	0.11	0.06	0.02	0.04
tube ϕ325x38 mm	0.11	0.41	0.28	0.016	0.004	8.80	0.26	0.91	0.22	0.12	0.07	0.01	0.03
ASME Case2179	0.08-0.12	0.30-0.60	0.20 0.50	max 0.02	max 0.010	8.0-9.5	max 0.40	0.85 1.05	0.18-0.25	max 0.30	0.06-0.10	max 0.040	0.03-0.07

a) b)

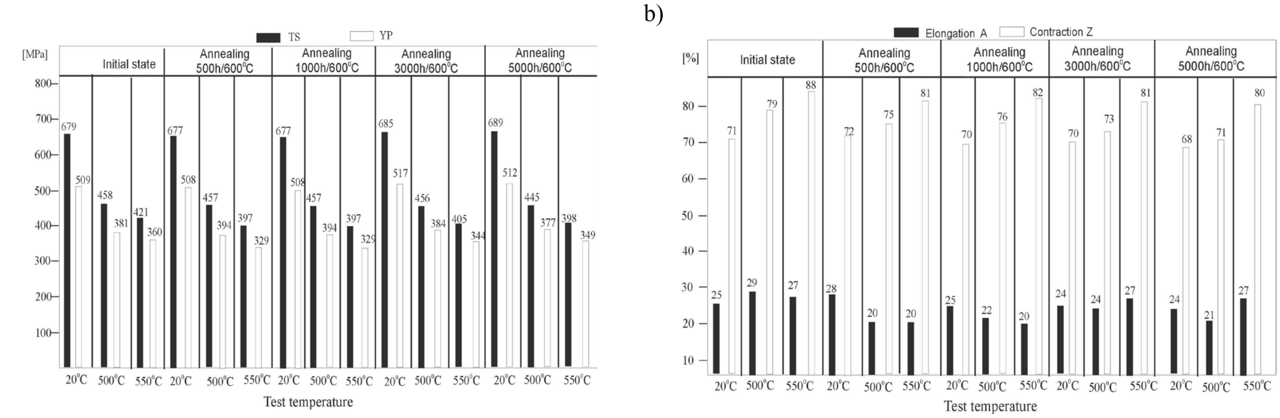

Fig. 2 a) Values of tensile strength and yield point after annealing at 600°C for 1000, 3000 and 5000 h; b) Values of elongation and reduction of area after annealing at 600°C for 1000, 3000 and 5000 h

a) b)

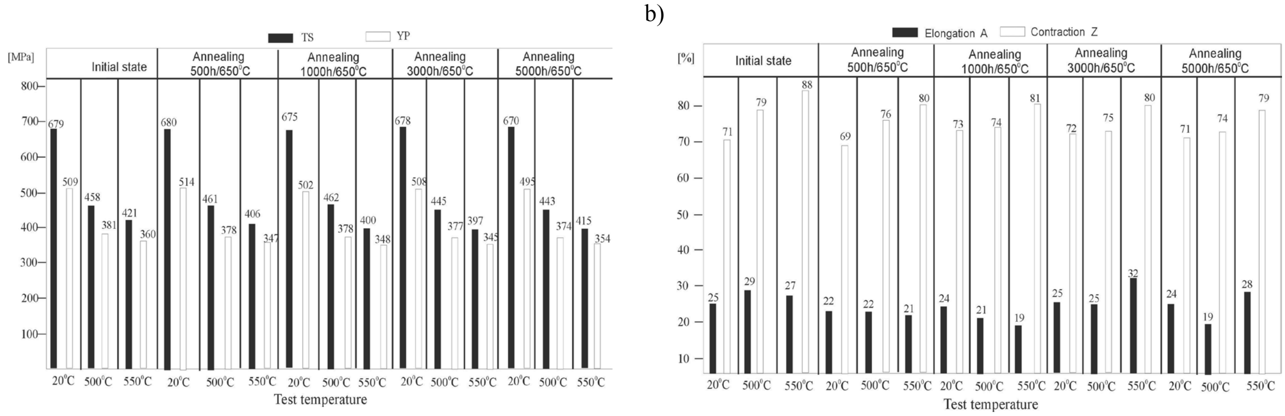

Fig. 3 a) Values of tensile strength and yield point after annealing at 650°C for 1000, 3000 and 5000h, b) Values of elongation and reduction of area after annealing at 650°C for 1000, 3000 and 5000h

4.2. Mechanical properties of X10CrMoVNb9-1 steel after 90.000 h service under creep conditions

The test results of tensile strength TS, yield point YP, elongation and reduction of area in static tensile test at room temperature are presented in Fig. 6, while the values of yield point within the test temperature range of 200-600°C are presented in Fig. 7.

The obtained test results for material after long-term service for 90,000h at working parameters of 540°C/18MPa are at the level of values for material in initial state (Fig. 1). However, significant decrease in impact energy as compared to the initial state of tested steel was observed. The brittle fracture appearance transition temperature determined based on the criterion of 27 J runs at -15°C (Fig. 8).

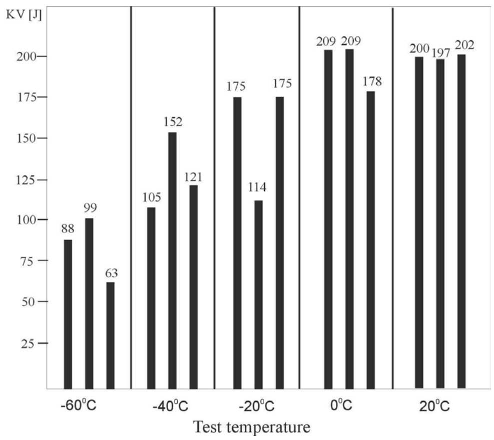

Fig. 4. Impact energy of tested steel in initial state

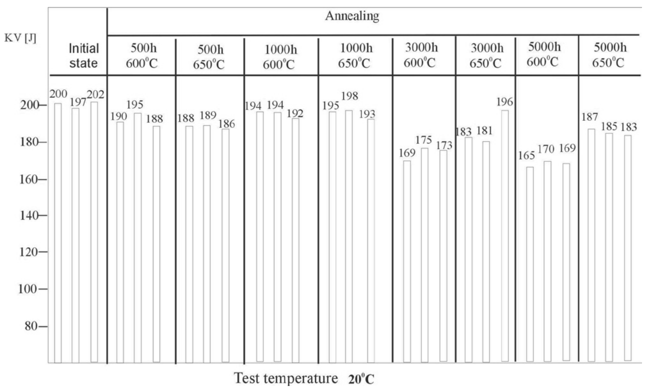

Fig. 5. Impact energy of tested steel after annealing at 600 and 650°C for 1000, 3000 and 5000 h.

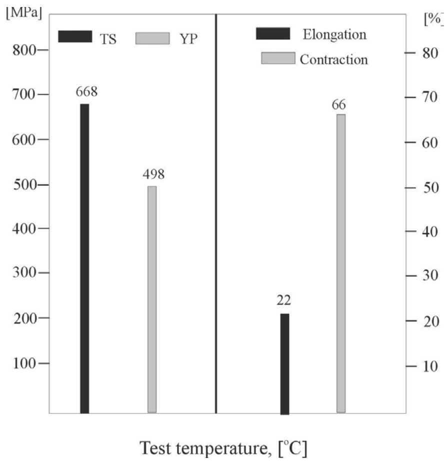

Fig. 6. Values of tensile strength and yield point, elongation and reduction of area of X10CrMoVNb9-1 steel after 90,000 h service.

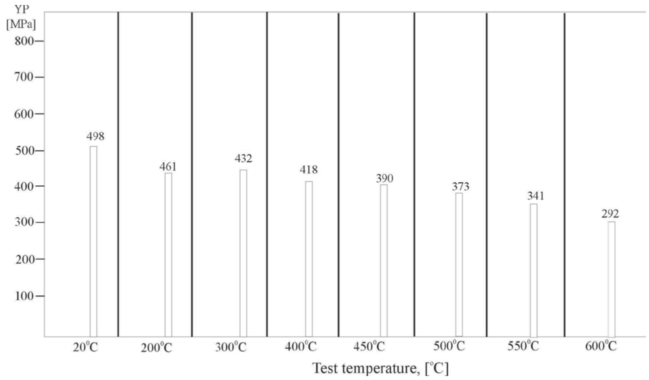

Fig. 7. Values of yield point at elevated temperature of X10CrMoVNb9-1 steel after 90,000 h service

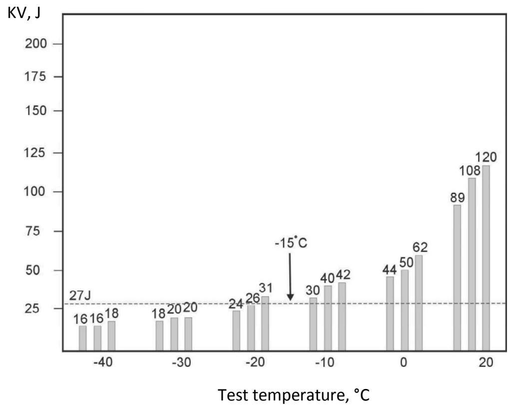

Fig. 8. Brittle fracture appearance transition temperature of X10CrMoVNb9-1 steel after 90,000 h service

4.3. Abridged creep tests

The abridged creep tests without elongation measurement of during the test were carried out using single-sample machines for creep tests manufactured by the Institute for Ferrous Metallurgy. These machines are equipped with load applying systems located in the heating chambers with constant temperature to provide stable test temperature level over the gauge length of a test piece and during the test, with accuracy of 1 degree at test temperature of up to 800°C.

The tests were performed on standard test pieces with ratio l_0/d_0 = 5, gauge length l_0 = 25 mm and gauge diameter of test piece d_0 = 5 mm, sampled along the pipe axis.

The abridged creep tests were carried out for X10CrMoVNb9-1 steel in initial state and after 90,000 h service.

The creep test parameters with test results are summarised in Table 2 and presented graphically in Figs. 6, 7 and 8 as the relationship $\log t_r = f(T_b)$ at test stress σ_b = 100 and 120 MPa for steel in initial state and σ_b = 100 MPa for tested steel after service, respectively. Table 3 summarises the forecast life time of tested steel for the expected operating temperature depending on the assumed operating stress σ_b.

Table 2.
Results of abridged creep tests of X10CrMoVNb9-1 steel

Material condition	Test stress σ_b [MPa]	Test temperature Tb [°C]^0				
		620	640	660	680	700
		Time to rupture t_z [h]				
initial state	100	(9553)	1285	270	29	7
	120	1613	430	51	11	4
after 90,000 h service	100	6993	1110	177	22	5

Note: Bracketed Figure refers to test in progress

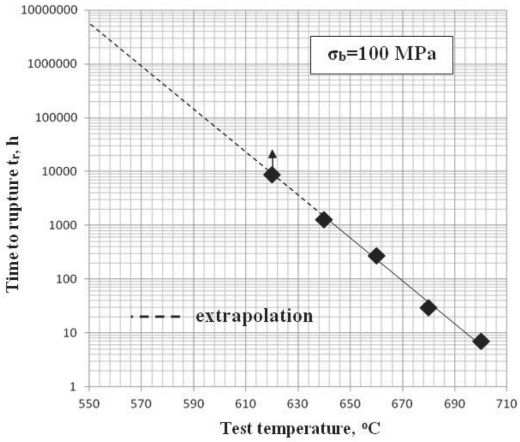

Fig. 6. Results of abridged creep tests presented as the relationship logtz = f(T_b) for X10CrMoVNb9-1 steel in initial state

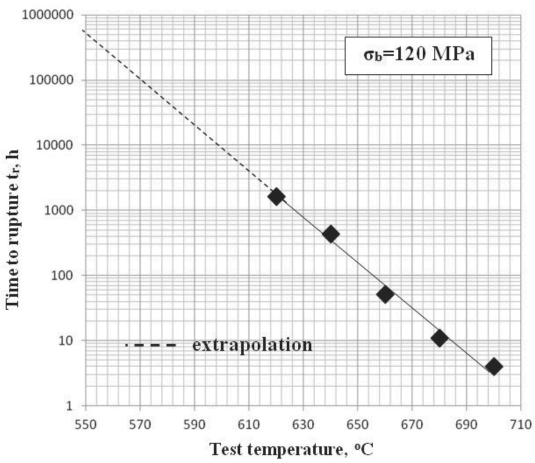

Fig. 7. Results of abridged creep tests presented as the relationship logtz = f(T_b) for X10CrMoVNb9-1 steel in initial state

The obtained results of abridged creep tests allowed the life time of tested X10CrMoVNb9-1 steel in initial state and after 90,000 h service under creep conditions to be estimated. The creep tests allow finding out that the tested material in the form of a test piece of X10CrMoVNb9-1 steel tube with diameter of ϕ355.6 mm and wall thickness of 50 mm meets the requirements of creep strength with reference to data provided in PN-EN 10216-2, while the life time of the material of test piece of tube after 90,000h service at 540°C and steam pressure of 18 MPa is by approx. 40% lower as compared to tested steel in initial state.

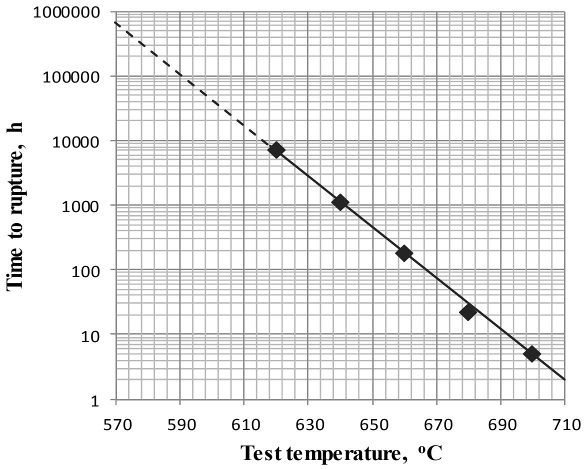

Fig. 8. Results of abridged creep tests presented as the relationship logtz = f(Tb) for X10CrMoVNb9-1 steel after 90,000 h service.

Table 3.
Forecast life time for tested X10CrMoVNb9-1 steel determined in abridged creep tests

Material condition	Assumed operating stress σ_r [MPa]	Assumed temperature of further operation T_r [ºC]	Estimated life time [h]
initial state	100	580	~350 000
	120		~45 000
after 90,000 h service	100		~200 000

4.4. Influence of long-term annealing on changes in structure

The structural investigations of X10CrMoVNb9-1 steel in initial state and after long-term annealing were carried out with scanning and transmission electron microscope.

The long-term impact of high temperature on the material structure is controlled by thermally activated processes. As the temperature rises, these processes are accelerated, at the same time reducing the set of functional properties of the material and component made of it. The microstructure of steel is subject to changes consisting in decay and/or growth of initial structural components and precipitation processes the course of which is the competing dissolution and secondary precipitation processes. The evaluation of changes in structure with regard to the level of functional properties is one of the elements of materials characteristics of steels designed for service under creep conditions.

The structure investigations were carried out for tested steel in initial state, after annealing at 600 and 650°C for 1000, 3000, 5000 and 10000 hours and after 90,000 h service.

The X10CrMoVNb9-1 steel in initial state is characterised by tempered martensite structure with fine-dispersion MX precipitations, mainly of vanadium, and $M_{23}C_6$ carbides, rich in vanadium and niobium. The precipitations occur both at the grain boundaries and inside ferrite grains. The precipitation hardening is the result of existence of numerous fine carbonitride precipitations, which have also significant impact on improvement in creep strength. The $M_{23}C_6$ carbides existing in steel stabilise the lath martensite structure.

Characteristic images of the structure of tested steel in initial state are shown in Fig. 9.

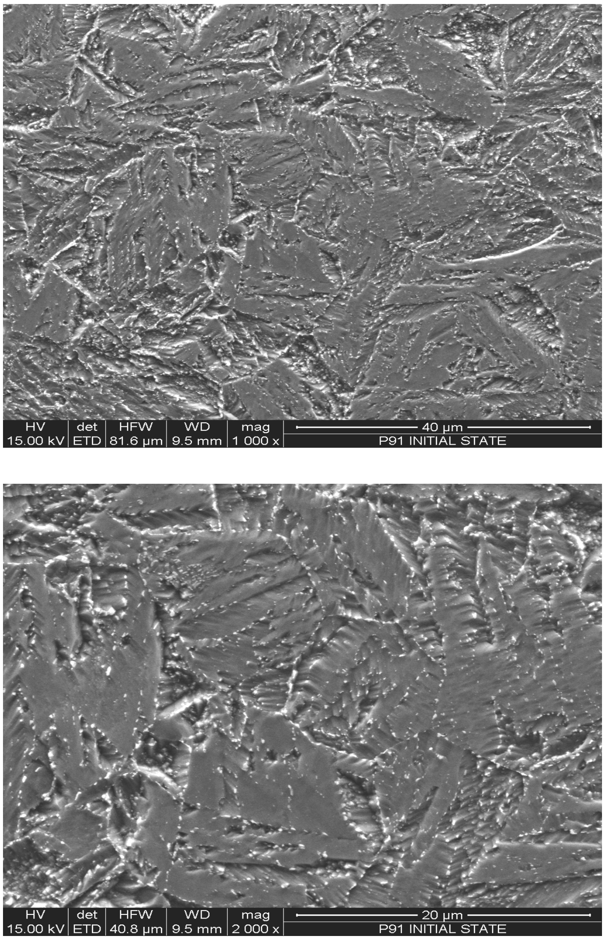

Fig. 9. Structure of X10CrMoVNb9-1 steel in initial state, observed with scanning electron microscope (hardness 232 HV10)

The annealing of X10CrMoVNb9-1 steel for 1000 h did not result in visible changes in structure image of tested steel observed with scanning electron microscope at both 600°C and 650°C (Figs. 10, 11). However, the first subtle changes illustrating the structural changes were observed after annealing at 650°C for 3000 h. The increase in amount of carbide precipitations at former austenite grain boundaries, at martensite lath boundaries and inside grains was visible (Fig. 12). However, no degradation processes in the form of decay of martensite lath structure were observed. The visible changes in structure image, as compared to the initial state, caused by simultaneous temperature and time impact could be observed on samples annealed for 5000 and 10000 hours (Figs. 13-16). Further increase in size of precipitations was found after this time of annealing. As it should have been expected, their size was bigger after long-term annealing at 650ºC (Figs. 14, 16).

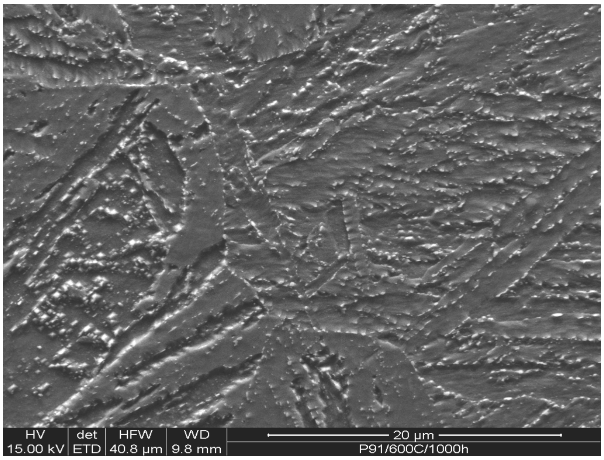

Fig. 10. Structure of P91 steel after annealing at 600°C for 1000 h, observed with scanning electron microscope (hardness 218 HV10)

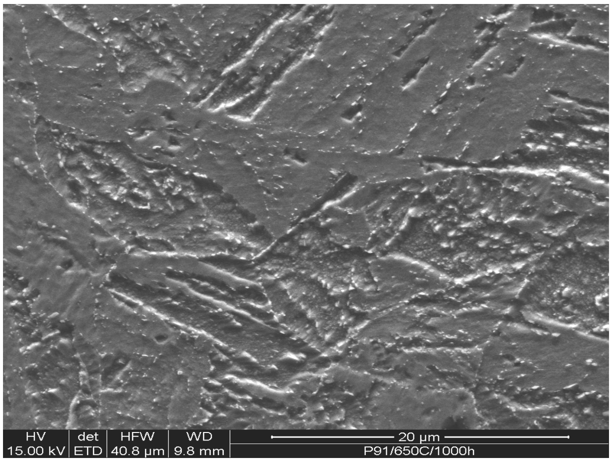

Fig. 11. Structure of X10CrMoVNb9-1 steel after annealing at 650°C for 1000 h, observed with scanning electron microscope (hardness 224 HV10)

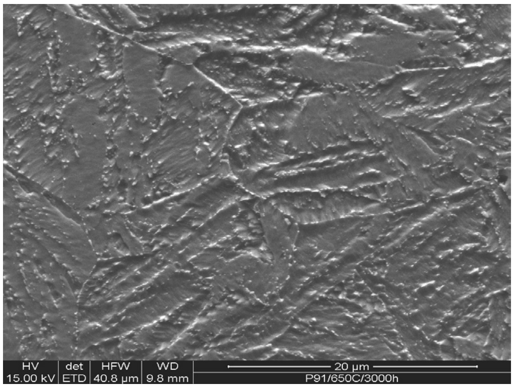

Fig. 12. Structure of X10CrMoVNb9-1 steel after annealing at 650°C for 3000 h, observed with scanning electron microscope (hardness 229 HV10)

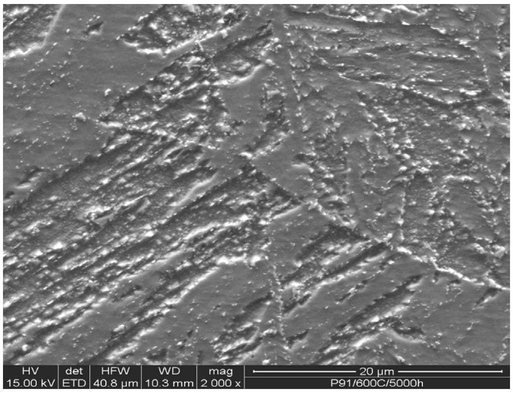

Fig. 13. Structure of X10CrMoVNb9-1 steel after annealing at 600°C for 5000 h, observed with scanning electron microscope (hardness 219 HV10)

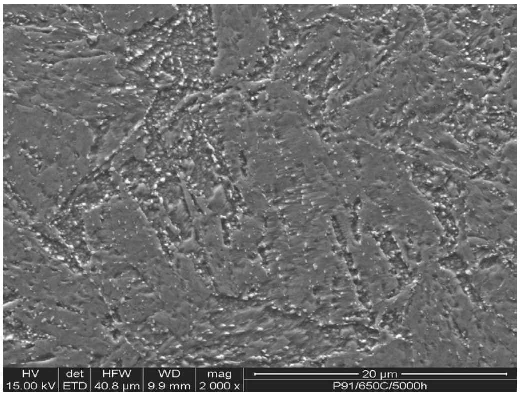

Fig. 14. Structure of X10CrMoVNb9-1 steel after annealing at 650°C for 5000 h, observed with scanning electron microscope (hardness 215 HV10)

In the structure of X10CrMoVNb9-1 steel after 10,000 h annealing (Figs. 15, 16), the effects of progressing martensite tempering processes can be observed. They result in bigger and more densely arranged $M_{23}C_6$ carbide precipitations at former austenite grain boundaries and inside ferrite grains.

In addition, the identification of precipitation phase was carried out with transmission electron microscope for material after 3000 h annealing at 650°C. The example of microstructure image with identification of $M_{23}C_6$ carbide and Laves phase is presented in Figs. 17, 18.

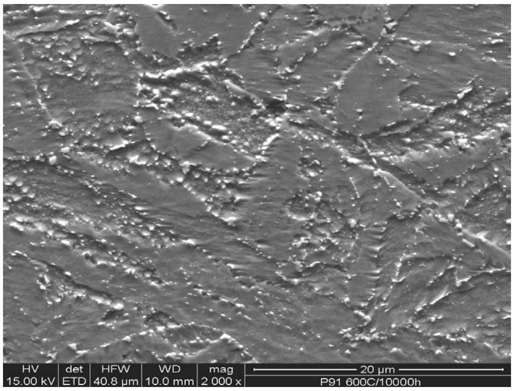

Fig. 15. Structure of X10CrMoVNb9-1 steel after annealing at 600°C for 10000 h, observed with scanning electron microscope (hardness 218 HV10)

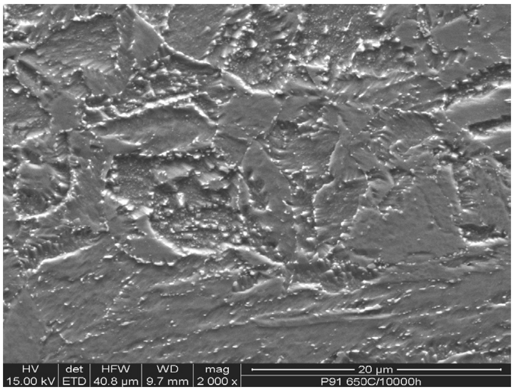

Fig. 16. Structure of X10CrMoVNb9-1 steel after annealing at 650°C for 10000 h, observed with scanning electron microscope (hardness 216 HV10)

5. Influence of long-term temperature and stress impact on changes in structure

The observation of structure of X10CrMoVNb9-1 steel exposed to simultaneous impact of temperature and stress was made for the test piece of tube after 90,000 h service at a temperature of 540°C and steam pressure of 18 MPa (Fig. 19) and after creep tests at 550°C/9852h/220MPa (Fig. 20), 600°C/7431 h/100 MPa (Fig. 21) and 650°C/9500 h/80 MPa (Fig. 22).

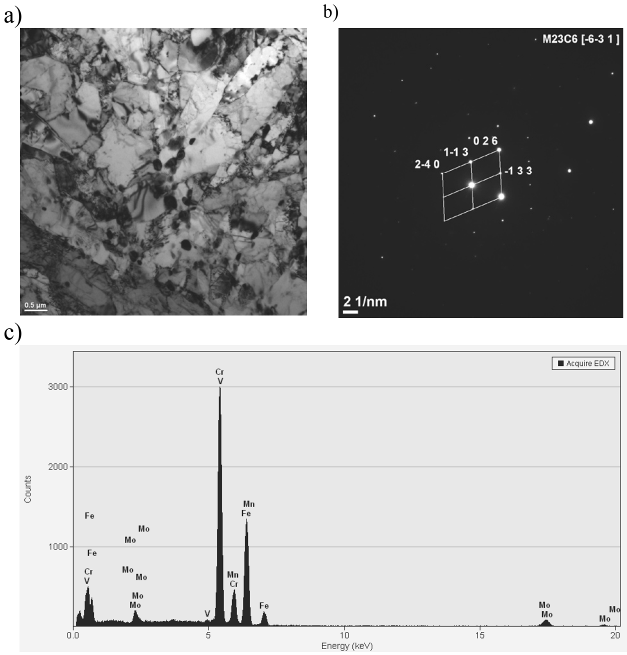

Fig. 17. Microstructure of X10CrMoVNb9-1 steel after annealing at 650°C for 3000 h observed with TEM a) bright field, b) diffractogram, c) EDS spectrum for $M_{23}C_6$ phase

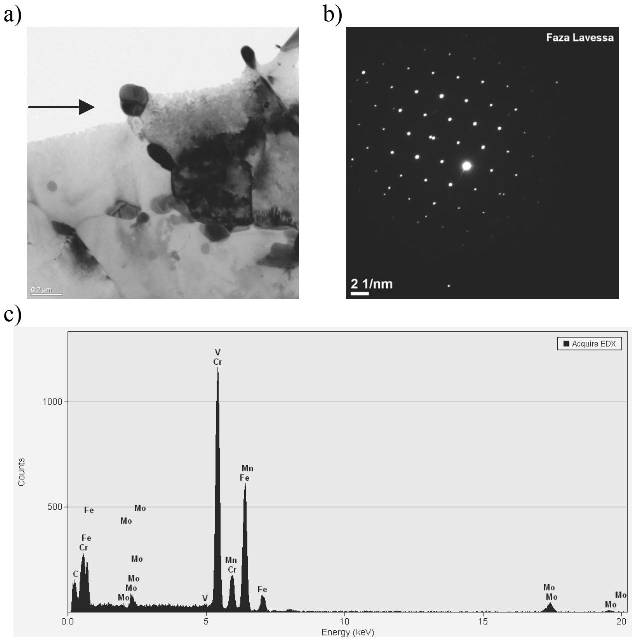

Fig. 18. Microstructure of X10CrMoVNb9-1 steel after annealing at 650°C for 3000 h observed with TEM a) bright field, b) diffractogram, c) EDS spectrum for Laves phase

As compared to the structure of material in initial state, partial decay of lath structure of tempered martensite areas and slight increase in size of precipitations at former austenite grain boundaries and inside grains was revealed for material after 90,000 h service. The X-ray phase analysis of existing precipitations showed that the main phase in tested steel after service consisted of $Cr_{23}C_6$ Isowit and large amounts of Fe_2Mo.

Changes in the image of structure of X10CrMoVNb9-1 steel subject to simultaneous impact of temperature and stress are caused by decay of martensite lath structure and increase in size of precipitations, both inside grains and at grain boundaries.

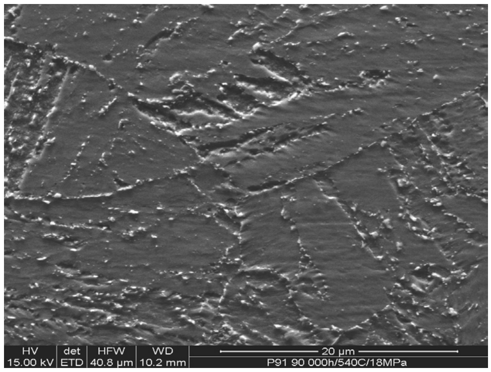

Fig. 19. Structure of X10CrMoVNb9-1 steel after 90,000 h service under creep conditions (hardness 212 HV10)

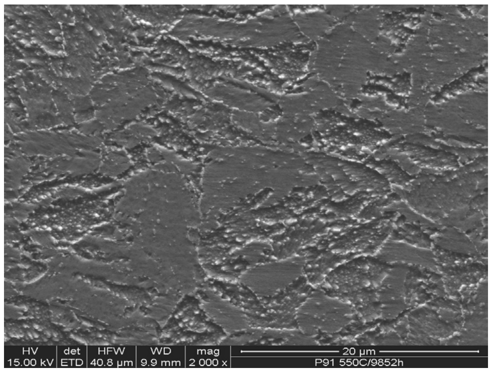

Fig. 20. Structure of X10CrMoVNb9-1 steel after creeping at 550°C/9852 h/220 MPa (hardness 211 HV10)

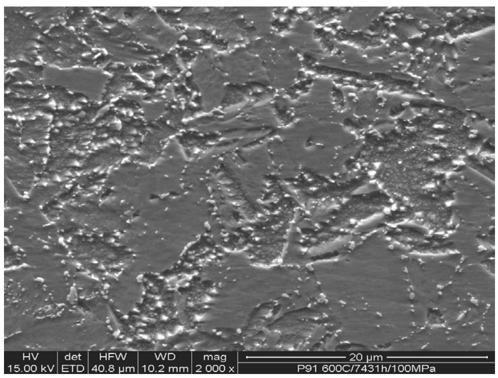

Fig. 21. Structure of X10CrMoVNb9-1 steel after creeping at 600°C/7431 h/100 MPa (hardness 209 HV10)

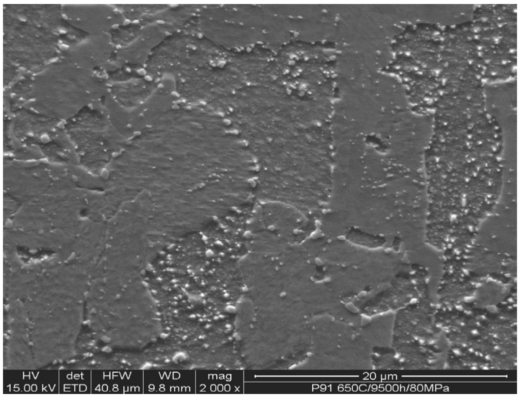

Fig. 22. Structure of X10CrMoVNb9-1 steel after creeping at 650°C/9500h/80MPa (hardness 188 HV10)

These changes are clearly visible for tested steel after creeping at 600°C/7431 h/100 MPa where decay of tempered martensite lath structure and increase in size of precipitations, mainly at grain boundaries, is observed, while for the material of tested steel after creeping at 650°C/9500 h/80 MPa the observed structure is characterised by decay of martensite lath structure with numerous considerable precipitations inside grains and at grain boundaries.

6. Conclusions

The tests carried out as a part of this paper allowed the mechanical properties of X10CrWMoVNb9-2 steel in initial state to be evaluated and compared to the results obtained after long-term annealing at 600 and 650ºC.

1. The determined values of tensile strength R_m, yield point $R_{0.2}$, elongation A_5 and reduction of area Z at room and elevated temperature meet the requirements for this steel.
2. Based on the completed impact strength tests it can be found that simultaneous impact of temperature and time result in slight decrease in impact strength, measured by impact energy, from approx. 200 J for initial state to approx. 180 J for tested steel after 5000 h annealing. Long-term service at 540°C for 90,000 hours resulted in shifting the brittle fracture appearance transition temperature to -15°C.
3. No significant differences in the structure image of X10CrMoVNb9-1 steel after long-term annealing for up to 10000 h were observed. The comparison of observed structure occurred as a result of simultaneous impact of temperature and time of long-term annealing to the structure of tested steel after creep tests, i.e. with additional impact of load, revealed that the structure degradation degree after annealing was slight, whereas partial decay of lath structure of tempered martensite areas with visible considerable carbide precipitations at former austenite grain boundaries and inside ferrite grains occurred as early as after approx. 7000 h creeping at a temperature of 600°C and stress of 100 MPa. Significant structural changes manifesting themselves in decay of martensite lath structure and increase in amount and size of precipitations, both at grain boundaries and inside grains, were observed after creeping at a temperature of 650°C, stress of 80 MPa and time of 9500 h. In addition, a significant difference manifesting itself in higher structure degradation in the image of structure after annealing at 650°C was found.
4. The tests have confirmed the literature data [20] according to which the X10CrMoVNb9-1 steel under simultaneous impact of temperature and time is characterised by the expected stable structure. However, it manifests itself in slight decrease in impact strength and structural changes the nature of which does not arouse reservations with regard to expectations for these steels.

This paper presents only some of the results of completed tests. The phase composition analyses of precipitations, microstructural observations with TEM, qualitative and quantitative analysis of type and composition of precipitations in X10CrMoVNb9-1 steel, both after long-term annealing and after long-term creep tests, are also being conducted.

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