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# Computer-assisted analysis of changes in the microstructure of P92 steel after exposure at elevated temperature

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#### **ABSTRACT**

**Purpose:** The purpose of the work was to analyse the components of X10CrWMoVNb9-2 (P92) steel in initial state and after long-term annealing for up to 100,000 h at 600 and 650°C.

**Design/methodology/approach:** The material for investigation was X10CrWMoVNb9-2 (P92) steel in the form of a Ø160x40mm tube. The images of microstructure were recorded using the scanning electron microscopy. The quantitative analysis of precipitates was carried out using the image analysis system NIKON EPIPHOT200 & LUCIA G v.5.03.

**Findings:** The investigations of microstructure and the quantitative analysis of precipitates in the tested steel in initial state and after long-term annealing allowed the impact of temperature and time on stability of the microstructure of the tested 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 the structure and in the precipitation processes are applied to evaluation of the condition of the elements in further industrial service.

**Keywords:** Microstructure; Long-term annealing; Hardness; X10CrWMoVNb9-2 (P92) steel

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METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

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#### 1. Introduction

The construction and operation of boilers with supercritical parameters has resulted in commencement of extensive research on steels for critical components of boilers, carried out jointly by boiler manufacturers and research bodies. In particular, it concerns critical components working under supercritical temperature and stress conditions. The effect of this research is numerous studies concerning functional properties of new-generation steels used under creep conditions [1-12]. The designers expect reliable steel products with guaranteed functional properties that would allow using them for boiler components to be operated under creep conditions for min. 200 thousand hours. In turn, the diagnostics and periodical inspections of these materials during the operation and tools made of them require knowledge of changes in functional properties with time of their service [13-16]. Therefore, conducting a set of investigations to allow building steel characteristics based on investigations of mechanical properties and microstructure is so important [17-28].

The X10CrWMoVNb9-2 (P92) steel was developed by modification of chemical composition of steels containing approx. 9%Cr, which have been used in the power industry so far. This steel is characterised by high mechanical properties; among other things, its creep strength is higher by approx. 20-25% than that of the steels used so far.

Long-term impact of elevated temperature during the operation of pressure equipment and systems brings about changes in the microstructure of the material resulting in reduction of mechanical properties. These changes determine the life time of components, which are now designed for 200,000 hours of operation. However, it does not mean that failure-free operation is ensured for such a long time. Therefore, the diagnostic works and inspections, and possible repairs are carried out during the boiler's lifetime to ensure safe and failure-free operation of power units.

In cooperation with other research centres (Silesian University of Technology, Czestochowa University of Technology, AGH University of Science and Technology in Krakow) as well as the boiler factories Rafako S.A. and

SEFKAO S.A. – the leading manufacturers of boilers, the Institute for Ferrous Metallurgy performs the works on the advance and verification testing of new-generation steels for the power industry [2,9,10,17,24,31,32].

This study presents the results of investigations on the components of P92 steel microstructure that affect its stability. These investigations included the analysis of the microstructure and quantitative analysis of precipitates. Steel in initial state and after long-term exposure at elevated temperature was subject to investigation.

The developed P92 steel characteristics with regard to the above-mentioned investigations are used in the assessment of changes in functional properties of the material of elements working under creep conditions.

### 2. Research methodology, material for investigation

The material for investigation was X10CrWMoVNb9-2 (P92) steel in as-received (initial) state and after long-term exposure at elevated temperature. Long-term term annealing was carried out at 600 and 650°C for up to 100,000 hours.

The microstructure investigations were carried out with Inspect F scanning electron microscope (SEM) on conventionally prepared metallographic microsections etched with ferric chloride.

The quantitative analysis of precipitates was carried out using the image analysis system NIKON EPIPHOT200 & LUCIA G v.5.03. The scale marker as shown in the photos was used for calibration of the image analysis system. Calibration coefficient: 1 pixel= 0.040  $\mu$ m. A measuring frame of 1020 x 940 pixels was applied on every analysed photo.

Material condition was evaluated based on the diagram of changes in microstructure after long-term service at elevated temperature [30].

The chemical composition of the tested steel is summarised in Table 1.

Table 1.

Material for investigation – chemical composition of P92 steel with reference to the requirements [29]

									1				
Steel	Element contents, wt%												
grade	-	С	Mn	Si	P	S	Cr	Mo	V	W	Nb	В	N
P92	Tested material	0.10	0.45	0.17	0.01	0.01	9.26	0.47	0.20	1.95	0.059	0.009	0.04
	according	0.07-	0.30-	max	max	max	8.5-	0.30-	0.15-	1.5-	0.04-	max	0.03-0.07
	to [29]	0.13	0.60	0.50	0.02	0.01	9.5	0.60	0.25	2.0	0.09	0.006	

#### 3. Research results and discussion

#### 3.1. Microstructure of P92 steel

In its initial state, the P92 steel is characterised by the microstructure with predominating tempered lath martensite and numerous precipitates.

In the microstructure of the tested steel, there was lath martensite substructure with elongated sub-grains and high density of dislocation as well as polygonised substructure of ferrite grains.

The tempered martensite microstructure with numerous precipitates is typical of this group of steels [9]. The examples of microstructures of the tested steel in initial state are shown in Figures 1 and 2.



Fig. 1. Microstructure of P92 steel in initial state observed using TEM

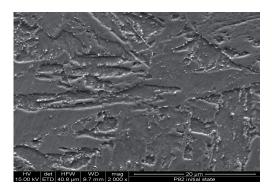


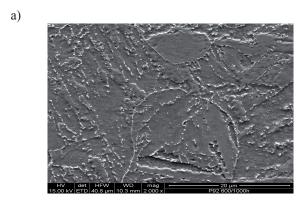
Fig. 2. Microstructure of P92 steel in initial state observed using SEM

The identification of precipitates revealed the existence of the following types of precipitates in the tested steel in initial state: MX and  $M_{23}C_6$ . The MX-VN and NbX precipitates

were observed mainly inside grains – at the dislocations and subgrain boundaries, while  $M_{23}C_6$  carbides were revealed at the former austenite grain boundaries and at the martensite lath boundaries [5].

The literature data [3, 5] also indicate that there may be complex precipitates in the tested steel, referred to as the "V-wings". These precipitates consist of a spherical NbX precipitate and VX lamellar precipitates that nucleate on them. However, precipitates of this type were not observed in the tested steel.

The microscopic observations revealed no essential changes in the microstructure of the tested steel after annealing at 600 and 650°C for up to 1000 h (Figs. 3a,b).



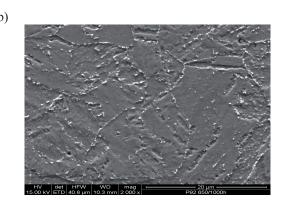


Fig. 3. Microstructure of P92 steel after annealing for 1,000 h at: a) 600°C; b) 650°C, SEM

The visible changes in the microstructure, as compared to the initial state, can be observed on samples annealed for 10,000 hours. The characteristic microstructure images after annealing at 600 and 650°C for 10,000, 30,000, 70,000 and 100,000 h are shown in Figures 4-7.

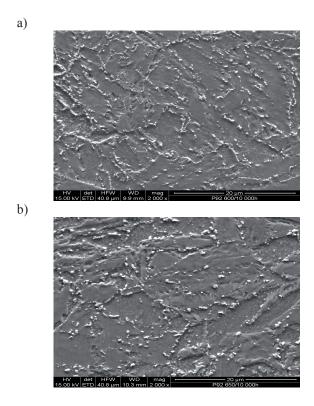


Fig. 4. Microstructure of P92 steel after annealing for 10,000 h at: a) 600°C; b) 650°C, SEM

The increase in the size of precipitates was found after this time of annealing. As it should have been expected, their size was bigger after long-term annealing at 650°C. In the microstructure of P92 steel after 10,000 h annealing, the effects of further progressing processes related to martensite tempering — softening of matrix can be observed. The result of these processes is bigger and more densely arranged  $M_{23}C_6$  carbide precipitates at the former austenite grain boundaries and on martensite laths. In addition to  $M_{23}C_6$  carbides at the grain boundaries, the Laves phase precipitates is observed too [5,9]. The annealing at 650°C contributes to local decay of the lath structure of tempered martensite.

The annealing for up to 30,000 h resulted in local decay of the lath structure of tempered martensite areas at test temperature of 600°C (Fig. 5a). The annealing at 650°C resulted in further progressing decay of the lath structure of tempered martensite microstructure (Fig. 5b). This microstructure was characterised by still preserved martensitic microstructure with numerous different size precipitates at the former austenite grain and martensite lath boundaries.

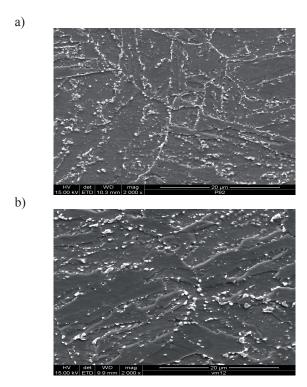


Fig. 5. Microstructure of P92 steel after annealing for 30,000 h at: a) 600°C; b) 650°C, SEM

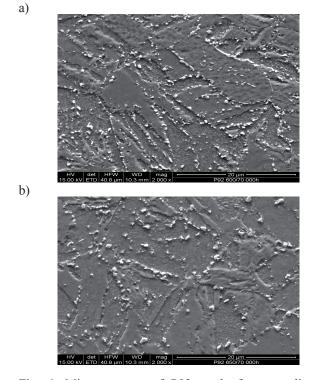


Fig. 6. Microstructure of P92 steel after annealing for 70,000 h at: a) 600°C; b) 650°C, SEM

As compared to the microstructure observed for the same duration of annealing and temperature of 600°C (Fig. 5a), the difference in the size of existing precipitates is noticeable (Fig. 5b). In both the analysed microstructures, areas were observed where the amount of precipitates at the grain boundaries was so high that they formed the so-called continuous network of precipitates.

The extension of the duration of annealing at 600°C up to 70,000 h revealed the increase in the size of precipitates (Fig. 6a), while the image of the microstructure of the tested steel annealed at test temperature of 650°C for 70,000 h shows, in addition to the coagulation of precipitates, further decay of the martensite lath structure (Fig. 6b). This microstructure is characterised by partially degraded areas of martensite with precipitations of large carbides at the former austenite grain boundaries, martensite laths and inside grains.

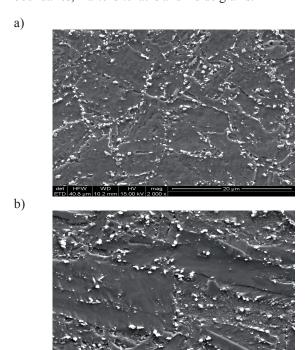


Fig. 7. Microstructure of P92 steel after annealing for 100,000 h at: a) 600°C; b) 650°C, SEM

The microstructure observed after annealing at 600°C for 100,000 h reveals a partial decay of the lath structure of tempered martensite areas and advanced precipitation process at the former austenite grain boundaries and martensite laths, which manifests itself in the increase (Fig. 7a) and coagulation of precipitates that form chains at the former austenite grain boundaries. The highest degradation level due to long-term exposure at elevated temperature was observed for steel annealed at 650°C – Fig. 7b. This micro-

structure is characterised by precipitates with the highest diameter and highest decay of the lath structure of martensite.

Table 2. Classification of changes in the structure of P92 steel after long-term annealing [30]

long-term annealing	g [30]					
Material state	Classification of structural changes					
	in accordance with Fig. 12					
Initial state	Martensite plates: class 0;					
	precipitates: class o;					
	destruction processes: class 0					
	material state: CLASS 0;					
	exhaustion degree: 0;					
Annealing	Martensite plates: class 0/I;					
1000h/600°C	precipitates: class o/a;					
Annealing	destruction processes: class 0					
1000h/650°C	material state: CLASS 1;					
	exhaustion degree: approx. 0.2					
Annealing	Martensite plates: class 0/I;					
10,000h/600°C	precipitates: class o/a;					
,,	destruction processes: class 0					
	material state: CLASS 1;					
	exhaustion degree: approx. 0.2					
Annealing	Martensite plates: class 0/I;					
10,000h/650°C	precipitates: class a;					
10,00011/020 €	destruction processes: class 0					
	material state: CLASS 1/2;					
	exhaustion degree: approx. 0.2-0.3					
Annealing	Martensite plates: class 0/I;					
30,000h/600°C	precipitates: class o/a;					
30,000m/000 C	destruction processes: class 0					
	material state: CLASS 1;					
	exhaustion degree: approx. 0.2					
Annealing	Martensite plates: class 0/I;					
30,000h/650°C	precipitates: class a;					
Annealing	destruction processes: class 0					
70,000h/600°C	material state: CLASS 1/2;					
70,00011/000 C	exhaustion degree: approx. 0.2-0.3					
Annealing	Martensite plates: class I;					
70,000h/650°C	precipitates: class a;					
70,00011/030	destruction processes: class 0					
	material state: CLASS 2;					
	exhaustion degree: approx. 0.3					
Annealing	Martensite plates: class I;					
100,000h/600°C						
100,00011/000 C	precipitates: class a;					
	destruction processes: class 0 material state: CLASS 2;					
A nnoolin ~	exhaustion degree: approx. 0.3					
Annealing	Martensite plates: class I;					
100,000h/650°C	precipitates: class a/b;					
	destruction processes: class 0					
	material state: CLASS 2/3;					
	exhaustion degree: approx. 0.3-0.4					

For recorded structure images of P92 steel after long-term annealing the exhaustion degree was assigned (Table 2) based on own classification of the Institute for Ferrous Metallurgy [30].

## 3.2. Analysis of the increase in the size of particles of P92 steel after exposure at elevated temperature

For the recorded model images of the microstructure of P92 steel, both in initial state and after long-term annealing, the distribution of diameters of the occurred precipitates was determined. The results in the form of distributions of equivalent diameters of precipitates occurring in the analysed states of the tested P92 steel are shown as histograms in Figs. 8-16. These histograms take into consideration the empirical distributions of relative and cumulative frequency.

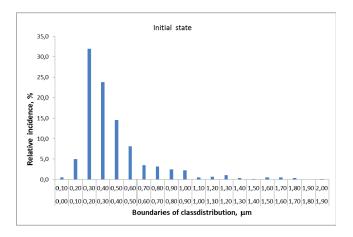


Fig. 8. Chart of empirical distribution of diameters of equivalent particle of the steel in the initial state P92

Changes in the size of particles in P92 steel in initial state and after annealing at 600 and 650°C unanimously indicate that their equivalent diameters increase as the temperature level and time of annealing grow. It was observed that the rate of particle growth at the annealing temperature of 650°C was significantly higher than that at 600°C. It was also observed that at 600°C, which is similar to the expected working temperature, there was a significant increase in mean diameter of precipitates within the time of up to 1,000 h, and the increase was very slow, whereas in case of annealing at 650°C the continuous increase in mean diameter of precipitates was observed depending on the time of annealing (Fig. 16).

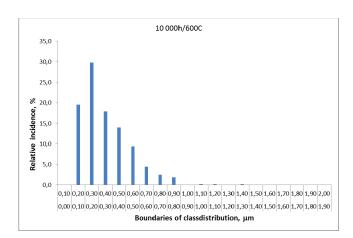


Fig. 9. Chart of empirical distribution of diameters of equivalent particle of P92 steel after annealing 10,000 h/600°C

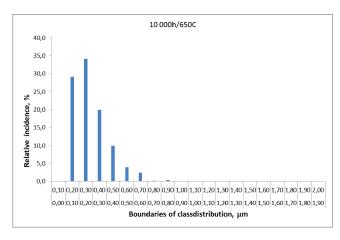


Fig. 10. Chart of empirical distribution of diameters of equivalent particle of P92 steel after annealing 10,000 h/650°C

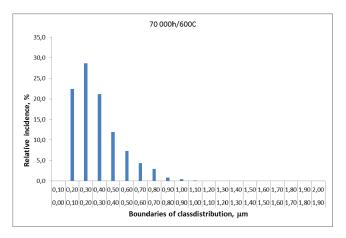


Fig. 11. Chart of empirical distribution of diameters of equivalent particle of P92 steel after annealing 70,000 h/600°C

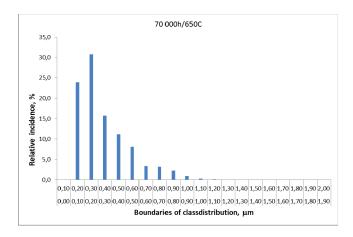


Fig. 12. Chart of empirical distribution of diameters of equivalent particle of P92 steel after annealing 70,000 h/650°C

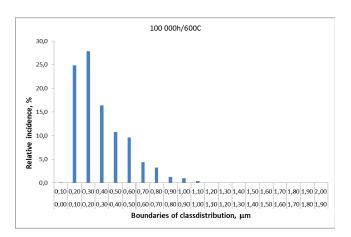


Fig. 13. Chart of empirical distribution of diameters of equivalent particle of P92 steel after annealing 100,000 h/600°C

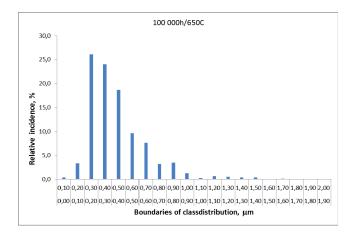


Fig. 14. Chart of empirical distribution of diameters of equivalent particle of P92 steel after annealing 100,000 h/650°C

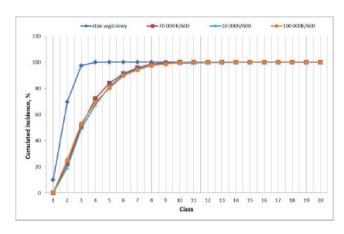


Fig. 15. Specification of cumulative frequency distributions for the precipitates in the steel P92 after long-term annealing at 600°C

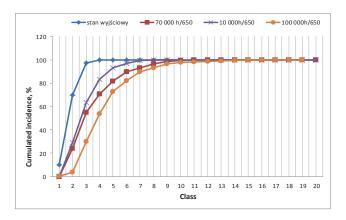


Fig. 16. Specification of cumulative frequency distributions for the precipitates in the steel P92 after long-term annealing at 650°C

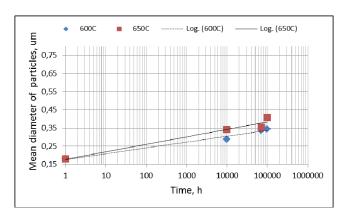


Fig. 17. Diagram of influence of annealing temperature and time on the mean particle diameter of P92 steel

By comparing the obtained test results and the analysis of increase in mean diameter of precipitates, it can be noticed that changes in the size of precipitates after 10,000 h/650°C annealing are similar to the results obtained after 100,000 h annealing at 600°C (Fig. 17).

Table 3 summarises the results of measurements of mean precipitate diameter with reference to time and temperature of annealing. The obtained results were referred to the exhaustion extent of P92 steel in accordance

with own classification of the Institute for Ferrous Metallurgy. Thus, the computer-assisted analysis of the image of changes in the size of precipitates is one of the components used in the evaluation of residual life of the tested steel.

The comprehensive evaluation of the exhaustion extent of P92 steel after exposure at elevated temperature should also include the disintegration of martensite structure.

Table 3.

Classification of changes in the structure of P92 steel after long-term annealing with reference to mean diameter of the occurring precipitates

Material state	Mean diameter,	Standard	Min. diameter,	Max. diameter,	Precipitates *	Martensite	Exhaust
	μm	deviation	μm	μm	<u>.</u>	plates *	degree*
Initial state	0.178	0.061	0.063	0.388	O	0	0
10,000 h/600°C	0.289	0.124	0.119	0.878	o/a	0/I	up to 0.2
10,000 h/650°C	0.342	0.180	0.110	1.382	a	0/I	approx. 0.2-0.3
70,000 h/600°C	0.337	0.165	0.110	1.020	a	0/I	approx. 0.2-0.3
70,000 h/650°C	0.354	0.189	0.110	1.133	a	I	approx. 0.3
100,000 h/600°C	0.345	0.186	0.045	1.089	a	0/I	approx. 0.2-0.3
100,000 h/650°C	0.407	0.218	0.090	1.658	a/b	I	approx. 0.3-0.4

<sup>\*</sup> Classification of the Institute for Ferrous Metallurgy [30]

#### 4. Conclusions

The analysis of the image of structure of P92 steel after annealing at 600 and 650°C for up to 1000 hours revealed no essential changes in the microstructure. Further extensions of the time of exposure at elevated temperature results in noticeable changes in the microstructure. The microstructure of P92 steel observed after 100,000 h reveals a partial decay of the lath structure of tempered martensite areas and advanced precipitation processes, especially at the former austenite grain boundaries, resulting in formation of the so-called continuous network of precipitates. The increase in the size of precipitates was revealed, which unambiguously indicated the occurring precipitation coagulation process, with higher dynamics of changes at 650°C.

The confirmation of changes in the structure is the quantitative analysis of the increase in occurring precipitates, which revealed high stability of changes in the

structure image at 600°C. It is particularly noticeable in Fig. 15, which presents the specifications of frequency distributions of cumulated precipitates.

The dynamics of changes in the structure due to the increase in temperature is presented in Fig. 17, where sizes of precipitates after 10,000 h/650°C annealing are similar to the results obtained after 100,000 h annealing at 600°C.

The quantitative and qualitative analysis of temperature impact on degradation of mechanical properties and structure of P92 steel is used in the evaluation of changes in functional properties of the material of components working under creep conditions.

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