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## MICROSTRUCTURAL ASPECT OF LONG TERM SERVICE OF THE AUSTENITIC TP347HFG STEEL

### WPLYW DŁUGOTRWALEJ EKSPLOATACJI NA MIKROSTRUKTURĘ STALI TP347HFG

The paper presents the results of research on the microstructure of austenitic creep-resisting TP347HFG steel after long-term service. The tests of the microstructure were performed using SEM and TEM. The identification of precipitates was carried out using selective electron diffraction. It has been shown that long-term service mostly contributes to the processes of precipitation of secondary NbC carbides inside grains and  $M_{23}C_6$  carbides on grain boundaries. In the microstructure of the examined steel, also the processes of polygonization and recrystallization of the matrix were revealed.

*Keywords:* TP347HFG steel, microstructure, TEM

W pracy przedstawiono wyniki badań mikrostruktury austenitycznej, żarowytrzymałej stali TP347HFG po długotrwałej eksploatacji. Badania mikrostruktury przeprowadzono przy użyciu techniki SEM i TEM. Identyfikację wydzielań wykonano wykorzystując selektywną dyfrakcję elektronów. Wykazano, że długotrwała eksploatacja badanej stali przyczyniła się głównie do procesów wydzielania: węglików wtórnych NbC wewnątrz ziaren oraz węglików  $M_{23}C_6$  po granicach ziaren. Ujawniono również w mikrostrukturze badanej stali procesy poligonizacji i rekrytalizacji osnowy.

### 1. Introduction

A niobium - containing, creep resistant austenitic TP347HFG steel with applied fine - grained microstructure has been widely used in the power generating industry and in the chemistry industry. It is used as piping and super-heater tubes in steam power plants and boiling water reactors, because of its good mechanical properties and corrosion resistance [1, 2]. Weldability of steels [3, 4], including austenitic creep-resisting steels, is an important criterion of their application in power engineering, in the construction of new power units and repair of the serviced ones [2, 5, 6].

The basic requirement set for steels in power industry is high stability of microstructure allowing long faultless operation in creep conditions. Nevertheless, the necessity to perform tests is indispensable for the assessment of the degree of microstructure degradation running during the service. It requires conducting complete research in each case, including microstructural tests to determine the usability of a given element for further safe operation. The assessment of results obtained in the research requires the knowledge of changes in the microstructure, including the knowledge of the courses of precipitation processes. Therefore, it is indispensable to build characteristics and data bases of materials by studying these materials after various periods of service [6, 7]. The

paper presents the results of research on the microstructure of austenitic TP347HFG steel after long-term service.

### 2. Material for research

The tested material was samples taken from a section of a coil of the boiler superheater serviced for about 105 000 hours at the steam pressure of 12.5 MPa and temperature of around 540°C. Chemical composition of the examined steel is presented in Table 1.

TABLE 1  
 Chemical composition of TP347HFG steel, mass%

C	Mn	Si	P	S	Cr	Ni	Nb	N
0.09	1.56	0.47	0.023	0.003	18.38	11.99	0.61	0.04

#### 2.1. Methodology of research

Observations and record of images of the microstructure were performed with the use of scanning electron microscope Joel JSM 6610LV (SEM) on metallographic specimens etched

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with the  $M_{19}Fe$  coefficient, and by means of transmission electron microscope TITAN 80-300 (TEM) using thin foils. Analysis of precipitation processes was carried out using selective diffraction of electrons.

## 2.2. Results of research and discussion

An example of the microstructure of TP347HFG steel after service observed by means of SEM is illustrated in Fig. 1. The TP347HFG steel after service was characterized by the microstructure consisting of austenite grains with visible annealing twins and numerous precipitates of diverse morphology. Some of these precipitates were arranged in strips in the matrix. The presence of twins in the microstructure of austenitic steels is a characteristic of metals with A1 lattice type. On the boundaries of grains, numerous fine precipitates were observed, however, no precipitates on the twin boundaries were seen.

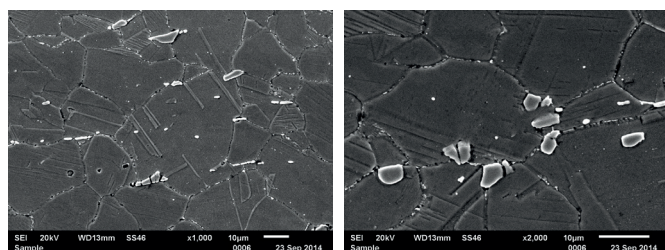


Fig. 1. Microstructure of TP347HFG steel after long-term service

The size of austenite grain in the examined steel, determined with the use of drawing standards according to ASTM scale, amounted to 8/7. The designed fine-grained microstructure of TP347HFG steel (grain size  $\geq 8$ ) ensures high resistance to oxidation and higher plasticity (expressed as the elongation growth in creep tests) with the creep resistance similar to that of coarse-grained steels [2]. Fine-grained microstructure of TP347HFG steel also has an influence on a slower decrease in impact strength during its long-term service [8, 9]. Performed identifications of precipitates revealed two types of carbides in the microstructure: NbC carbides and  $M_{23}C_6$  carbides (Fig. 2, 3). The NbC precipitates occurred in the examined steel as the following two types:

- as large primary carbides of the mean diameter amounting to 1.85  $\mu\text{m}$ . The primary NbC carbides are precipitated in the last phase of coagulation from the liquid [10];
- fine-dispersive secondary precipitates of the mean diameter of around 45 nm.

The precipitations of primary NbC carbides were revealed not only inside but also on the boundaries of grains. These precipitates, because of their size, do not have an influence on the growth of precipitation strengthening of the investigated steel. Their main role is binding carbon atoms, which results in the limiting of the  $M_{23}C_6$  carbides precipitation on the boundaries of grains and inhibiting of the grain growth. Primary NbC carbides are characterized by high stability – their growth was not observed even after 70 000 hours of ageing at the temperature of 700°C [10]. Primary NbC carbides should be treated as unfavorable precipitates, since the nucleation and

development of creep cracking can occur on their interphase boundary - carbide/matrix [2, 11].

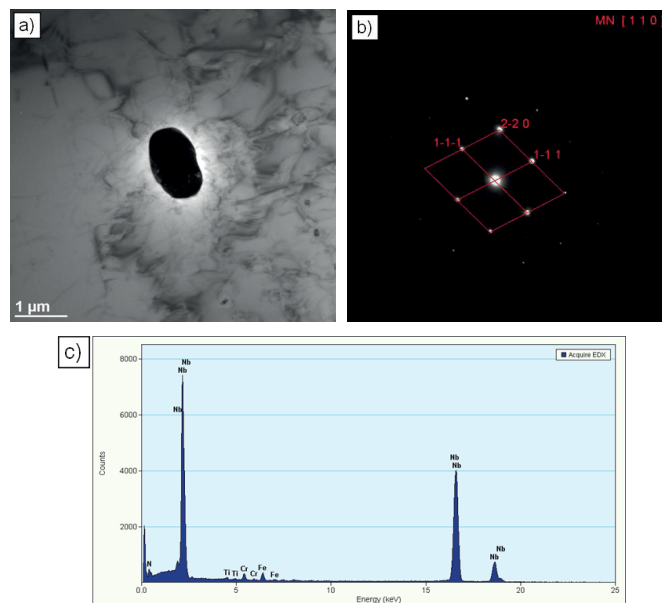


Fig. 2. Primary NbC precipitates: a) morphology of carbide, b) solved diffraction; c) characteristic X-ray spectrum

Fine-dispersive secondary NbC carbides precipitated inside the grains contribute to the increase in creep resistance, as well as in strength properties [11], by inhibiting the movement of dislocations (Fig. 4).

On the boundaries of grains in the examined steel, apart from the primary NbC carbides, also the  $M_{23}C_6$  carbides were observed (Fig. 5). The mean diameter of these precipitates after service amounted to around 197 nm. The precipitation of  $M_{23}C_6$  carbides on grain boundaries in austenitic steels depends on the character of the boundary. Privileged grain boundaries are those characterized by high degree of coincidence  $\Sigma$ , or those of high misorientation angle  $\Theta$  [12, 13]. The precipitation of  $M_{23}C_6$  carbides on grain boundaries in austenitic steels is connected with limited solubility of carbon in the matrix with temperature.

At the temperature of 1100°C, in steel 316, the solubility of carbon amounts to around 0.16 wt.%, whereas at the temperature of 600°C, it amounts to 4 ppm [14]. The precipitation of  $M_{23}C_6$  carbides in non-stabilized austenitic steels is a privileged precipitation process [11]. Whereas in the steels stabilized with titanium or niobium, the precipitation of  $M_{23}C_6$  carbides is limited [15] or practically eliminated [16]. The precipitation of  $M_{23}C_6$  carbides on grain boundaries can contribute to the growth of creep strength of the examined steel as a result of the limitation in mobility of grain boundaries (pinning effect). The precipitation of  $M_{23}C_6$  carbides on grain boundaries in the investigated steel at the temperature of service can also result in the formation of near-boundary areas depleted of chromium. The effect of this process can be an increase in the liability of the examined steel to intercrystalline corrosion. Similar effect of the near-boundary areas depletion of chromium in steels stabilized with titanium or niobium was observed in the work [17].

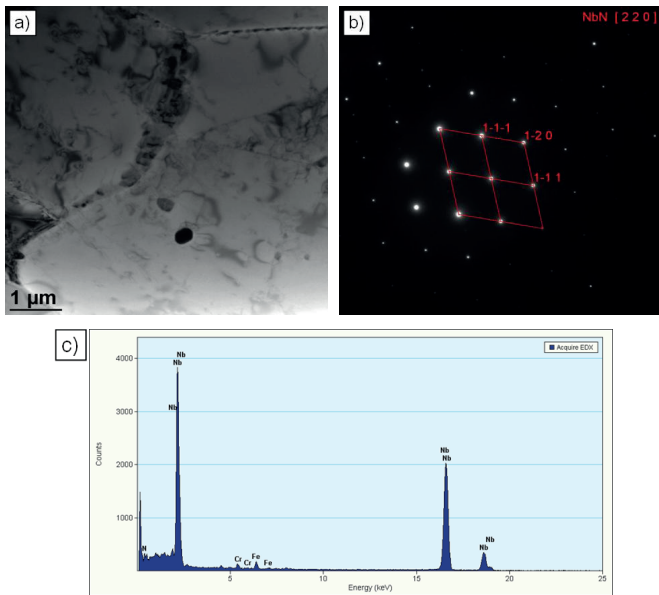


Fig. 3. Secondary NbC precipitates: a) morphology of carbide, b) solved diffraction; c) characteristic X-ray spectrum

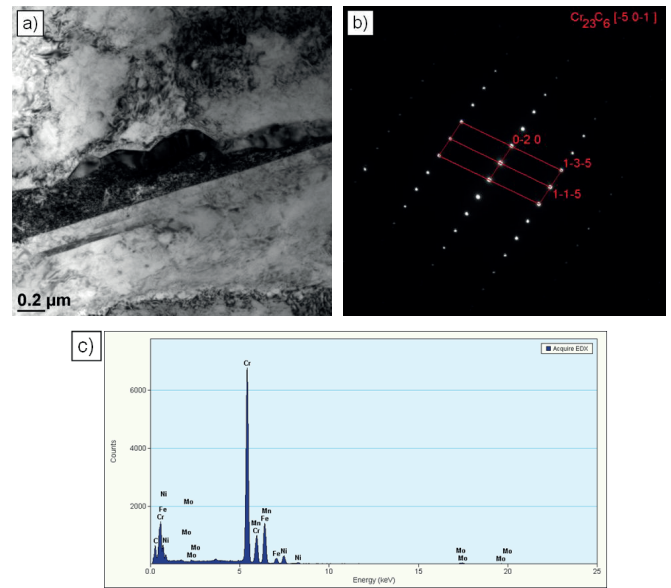


Fig. 5. The precipitation of  $M_{23}C_6$  carbide: a) morphology of carbide, b) solved diffraction; c) characteristic X-ray spectrum

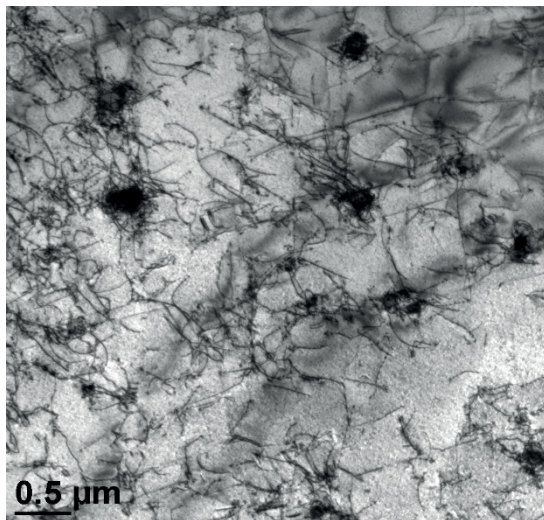


Fig. 4. Interaction of dislocations with secondary NbC precipitates

In the examined steel, however, no precipitation processes on the twin boundaries were observed. It can result from their lower energy compared to the wide-angle boundaries, or from high degree of their coherence with the matrix. Moreover, there were no precipitations of intermetallic phases observed in the examined steel, which mostly results from the low temperature of their service.

In addition, the observations have shown the occurrence of the process of polygonization (Fig. 6) and recrystallization of the matrix (Fig. 7) in the examined steel. The recrystallization front was inhibited by the carbides precipitated on the grain boundaries (Fig. 7). The temperature of recrystallization of the 347 type of steel lies within the range of  $232 \div 746^\circ\text{C}$ , at the temperature of melting between  $1400 \div 1425^\circ\text{C}$  [18]. Matrix recovery, as a result of annihilation of the dislocations and their entanglement in the boundaries, contributes to the softening process. The recrystallization process influencing the growth of grain size also results in the matrix softening and can lead to a decrease in corrosion resistance of the examined steel.

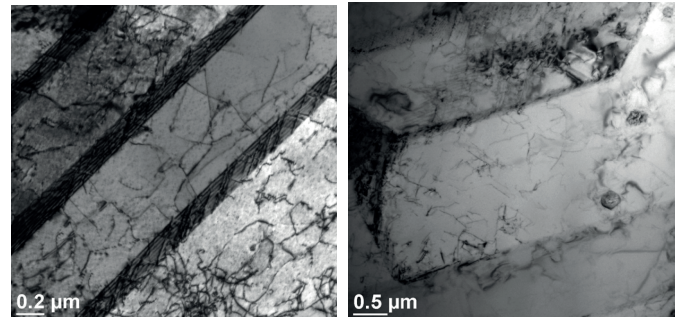


Fig. 6. Recovery process of austenitic matrix in the examined steel

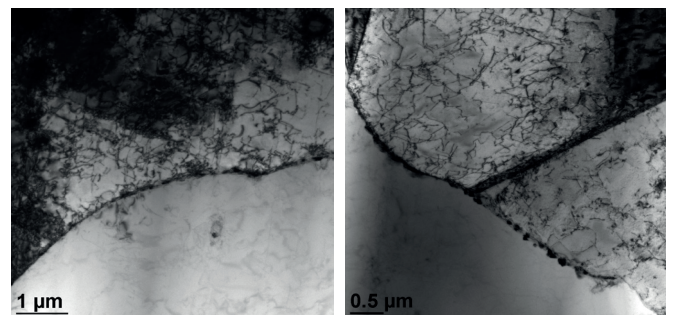


Fig. 7. Delay of matrix recrystallization processes by precipitates on grain boundary

The lack of access to data concerning the microstructure in the as-received condition limits the possibility of analyzing the changes that run in the examined steel during the service. However, considering the applied heat treatment for this grade of steel – hyperquenching in water from the temperature of  $1180 \div 1250^\circ\text{C}$  [19], and literature data [1, 2, 8, 10, 18], it can be assumed that in the as-received condition, the investigated steel was characterized by the microstructure consisting of austenitic matrix and primary NbC precipitations. It shows that the degradation of the microstructure of the examined steels during the service mostly occurred through the precipitation of secondary carbides, i.e. NbC and  $M_{23}C_6$  and the processes of recovery and recrystallization of the matrix.

### 3. Summary

The steel subject to research was austenitic creep-resisting TP347HFG steel after long-term service at the temperature of around 540°C. Performed research has shown an insignificant degree of degradation of the microstructure of the analyzed material. It has been proved that long-term service contributed mostly to the precipitation of secondary NbC carbides inside the grains and  $M_{23}C_6$  carbides on the grain boundaries. The precipitation of  $M_{23}C_6$  carbides on the grain boundaries can contribute to the occurrence of the so-called intercrystalline corrosion. In the examined steel, no precipitations of intermetallic phases or precipitation processes on twin boundaries were observed. The revealed processes of polygonization and recrystallization of the matrix lead to its softening, which can cause not only a decrease in the strength properties, but also a fall of the corrosion resistance.

### REFERENCES

- [1] K. Yoshikawa, H. Teranishi, K. Tokimasa, H. Fujikawa, M. Miura, K. Kubota, *J. Mater. Eng.* **10**, 69 (1988).
- [2] C. Chengyu, Y. Hongyao, X. Xishan, Advanced austenitic heat-resistant steels for ultra-super-critical (USC) fossil power plants, in: E. V. Morales, (Ed.) Alloy steel – properties and use, (2011).
- [3] T. Węgrzyn, J. Piwnik, B. Łazarz, D. Hadryś, *Arch. Metall. Mater.* **58**, 555 (2013).
- [4] T. Węgrzyn, J. Piwnik, D. Hadryś, *Arch. Metall. Mater.* **58**, 1067 (2013).
- [5] J. Dobrzański, J. Pasternak, A. Zieliński, in J. Lecomte – Becker (Eds.) 9th Liege Conference on Materials for Advanced Power Engineering, 412-423 (2010).
- [6] J. Dobrzański, H. Paczkowska, B. Kowalski, J. Wodzyński, *Prace IMŻ* **1**, 33 (2010) (in Polish).
- [7] A. Zieliński, J. Dobrzański, *Prace IMŻ* **3**, 42 (2013) (in Polish).
- [8] J. Jianmin, M. Montgomery, O.H. Larsen, S. A. Jensen, *Mater. Corros.* **56**, 459 (2005).
- [9] V.B. Trindade, P. Krupp, B.Z. Hanjari, S. Yang, H. – J. Christ, *Mater. Resear.* **8**, 371 (2005).
- [10] J. Erneman, M. Schwind, H.-O. Andrén, J.-O. Nilsson, A. Wilson, J. Ågren, *Acta Mater.* **54**, 67 (2006).
- [11] K.H. Lo, C. H. Shek, J.K.L. Lai, *Mater. Sc. Eng. R* **65**, 39 (2009).
- [12] Y. Zhou, K.T. Aust, U. Erb, G. Palumbo, *Sc. Mater.* **45**, 49 (2001).
- [13] R. Jones, V. Randle, G. Owen, *Mater. Sci. Eng. A* **496**, 256 (2008).
- [14] A.F. Padilha, P.R. Rios, *ISIJ Inter.* **42**, 325 (2002).
- [15] J.M. Letnaker, J. Bentley, *Metall. Trans.* **8A**, 1605 (1977).
- [16] A.S. Grot, J. E. Spruiell, *Metall. Trans.* **6A**, 2023 (1975).
- [17] T. Thorvaldsson, G.L. Dunlop, *J. Mater. Sci.* **18**, 793 (1983).
- [18] D. Lopez-Lopez, A. Wong-Moreno, R. Perez-Campos, A. Jardon-Benitez, *Corrosion 2002*, Paper No. 02383 (2012).
- [19] Datasheet, DMV 347 HFG. Stainless Tubes, 2008.