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MATERIAL

# Microstructural evolution of aged heat-resistant cast steel following strain controlled fatigue



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#### ABSTRACT

The paper presents the results of research on the microstructure of high-chromium martensitic GX12CrMoVNbN9-1 (GP91) cast steel after the isothermal ageing process and fatigue process. The fatigue process was performed at room temperature and elevated temperature (600 °C), with the value of total strain amplitude  $\varepsilon_{ac}$  amounting to 0.25% and 0.60%. Microstructural tests of GP91 cast steel were carried out by means of high-resolution transmission electron microscope. Quantitative study performed by means of TEM included the characteristics of changes in the dislocation substructure and morphology of M<sub>23</sub>C<sub>6</sub> carbides. Performed research has shown that the microstructure of the examined cast steel after ageing is characterized by partly remaining lath microstructure with numerous precipitations of the MX and M<sub>23</sub>C<sub>6</sub> type, as well as the Laves phase. It has been shown that the fatigue test at room temperature contributes to the process of dislocation strengthening of the examined cast steel. The increase of fatigue test temperature influences the degree of increase in the matrix softening. The degree of softening of the cast steel microstructure at elevated temperature depends also on the value of strain amplitude  $\varepsilon_{ac}$ . The softening process of the examined cast steel was connected with the decrease of dislocation density and increase of subgrains.

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#### 1. Introduction

An increase in the parameters of operation of power units has contributed to the introduction of new grades of materials into the power industry, such as high-chromium cast steels of 9–12% Cr type, designed for cylinders of steam turbines. One of the representative of this new group of cast steels is GX12CrMoVNbN9-1 (GP91) cast steel created on the basis of chemical composition of P91 steel. The GP91 cast steel is characterized by high mechanical properties, higher than the properties of low alloy CrMo or CrMoV cast steels used so far, which allows using these casts in power units designed for work at the so-called supercritical parameters [1,2].

Cylinders of steam turbines of large power, next to impellers, belong to the most loaded elements of turbines. Their service in power units has a cyclic character, and the typical working cycle consists of start-up, set operation, and down-time. At the first and third stage of the cycle, there is a large changeability of thermal

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loads, as well as mechanical loads, often exceeding the value of yield strength. On many occasions, the cyclic changeable effect of temperature and stress, after a certain number of cycles, can contribute to the occurrence of deformations and cracks of fatigue character. According to the data included in the work [3], low cycle fatigue, or the thermal-mechanical fatigue, can constitute up to around 65% of all damages/failures of steel castings.

The service of materials at elevated temperature causes a number of phenomena and changes in the microstructure, which results in a gradual decrease in mechanical properties of the material, including the capability of shifting cyclic changeable loads. The range of changes in mechanical properties occurring as a result of degradation of the microstructure, caused by the effect of temperature and time, and in the creeping conditions also by the effect of stress, can be easily predicted, whilst their quantitative change cannot. Therefore, it is necessary to perform the research on the course of these changes, for example through the process of isothermal annealing at the temperature close to the temperature of the expected service [4–6]. The aim of the performed research was to determine the stability of the microstructure of GP91 cast steel after long-term ageing, subject to the fatigue test at room temperature and at the temperature of 600 °C.

#### 2. Material and methodology of research

The investigated material was high-chromium martensitic GX12CrMoVNb9-1 (GP91) cast steel of the following chemical composition (mass%): 0.12C, 0.47Mn, 0.31Si, 0.014P, 0.004S, 8.22Cr, 0.90Mo, 0.12V, 0.07Nb, and 0.04N. The examined samples were after 8000 h of ageing at the temperature of 600 °C, as well as after ageing and fatigue test at room temperature and at 600 °C, with the value of total strain amplitude  $\varepsilon_{ac}$  amounting to 0.25% and 0.60%.

The influence of long-term ageing on mechanical properties of GP91 cast steel is presented in the works [5,7], whilst the influence of ageing and fatigue test on fatigue properties – in the works [8,9]. Microstructural tests of GP91 cast steel were carried out by means of high-resolution transmission electron microscope, JOEL JSM 3010. Particles of secondary phases were extracted in carbon replicas. Moreover, the identification of particles was carried out with the use of thin foils. They were analyzed using selected area electron diffraction (SAED). Thin foil for transmission microscopy was prepared by window thinning method using 10%perchlore acid in methanol as electrolyte. Extraction replicas were prepared from well polished and carbon coated specimens using Vilella's reagent. The fatigue test samples were sectioned parallel to the stress-loading direction. In addition to the microstructural tests, the tests using quantitative analysis of image were carried out to determine: the mean diameter of subgrains, the density of dislocations, and the mean diameter of M<sub>23</sub>C<sub>6</sub> carbides. The dislocation density inside the subgrains was determined using the images of microstructure obtained in the light visual field in the dynamic conditions of double-beam diffraction [7]. The size of the subgrains was determined on the basis of determining the equivalent circle diameter (ECD) of subgrain.

#### 3. Research results and discussion

#### 3.1. Microstructure of GP91 cast steel after ageing

The microstructure of GP91 cast steel after ageing was characterized by the maintained lath microstructure of tempered martensite, whose shape was inherited from lath martensite with diverse size of subgrains and numerous precipitates. Moreover, in the microstructure of the cast steel after ageing, also the microareas of polygonized ferrite were observed (Fig. 1).

In the microstructure of the aged cast steel, similarly as in the as-received condition, the presence of  $M_{23}C_6$  carbides/carbonitrides of the MX type (NbC, VX) was revealed. The  $M_{23}C_6$  carbides were precipitated on the boundaries of prior austenite grains, on the boundaries of laths, and along lath boundaries. The precipitates located along the lath boundaries imposed the pinning effect as shown in Fig. 1a,b. Whereas the MX precipitates were observed inside the laths (distributed uniformly within the lath boundaries). The amount of  $M_{23}C_6$  carbides precipitated on the boundaries of grains was so large that they formed the so-called continuous grid of precipitates in some areas (Fig. 1b). The relative increase of the number of  $M_{23}C_6$  carbides precipitated on the boundaries was accompanied by the process of their coagulation (Table 1).

The increase in the mean diameter of  $M_{23}C_6$  carbides (Table 1), with the assumption of their constant volume fraction, according to Ostwald's rule, contributes to the reduction of the number of these precipitates and leads to the growth of spacing of the particles. The result is that the role of these carbides as an inhibiting force for the migration of dislocation boundaries is limited. The lack of  $M_{23}C_6$  carbides precipitated on the boundaries of martensite laths results in a growth of creep rate and advanced process of recovery and polygonization of the matrix [10].

The microstructure of GP91 cast steel in the as-received state (after heat treatment), in spite of many hours of holding at the temperature of tempering/stress relief annealing, was characterized by non-equilibrium microstructure of high dislocation density [5]. The tendency towards decreasing the internal energy of the dislocation microstructure has contributed, as a result of ageing, to a decrease in the density of free dislocations and growth of the subgrain size, through the processes of recovery of the matrix (Table 2). In steels/cast steels of the 9–12% Cr type, the mechanism of hardening with subgrains is the dominant mechanism of strengthening [11] and constitutes the main obstacle in the movement of dislocations. The subgrain boundary migration is



Fig. 1. Microstructure of GP91 cast steel after ageing at the temperature of 600 °C.

#### Table 1

Mean diameter of  $M_{23}C_6$  carbides in the as-received condition, after ageing, and after ageing and fatigue test.

Parameters temperature, C/strain, % n		Minimum diameter, nm	Maximum diameter, nm	Mean diameter, nm	Standard deviation	
As-received condition		357	30.13	381.64	127.27	58.14
Ageing 600 °C/8000 h		308	30.79	484.9	161.12	56.16
Room	0.25	426	43.74	366.27	160.06	57.48
	0.60	395	34.27	464.46	159.60	78.74
600	0.25	502	33.04	375.44	154.13	66.80
	0.60	404	46.20	430.40	162.44	52.17

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