The influence of elevated temperatures on microstructure of cast irons for automotive engine thermo-mechanical loaded parts

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Abstract: The influence of elevated temperatures (550 °C and 850 °C) on microstructure and resistance to oxidation of gray iron quality EN-GJL-250 and ductile irons qualities: EN-GJS-400-18, EN-GJS-500-7, EN-GJS-SiMo4-1 and EN-GJS-AXNiCr20-2 were analyzed in this paper. The highest resistance to oxidation and microstructure changes at 550 °C and 850 °C shows austenitic ductile iron quality EN-GJS-AXNiCr20-2, followed by ferritic ductile iron quality EN-GJS-SiMo4-1. During oxidation of those cast irons at elevated temperatures two oxide layers over the surface of samples were occurred. Outward layer is easy detachable and consists of iron oxide. Inward layer is tightly connected with metal matrix and prevents progression of oxidation to the samples inside. This oxide layer consists of Fe, SiO₄ (favalite) or complex oxides (Fe, Cr, Ni, Si, and O). Due to favorable high-temperatures properties, those materials are suitable for construction of thermo-mechanical loaded parts of automotive engine, such as turbocharger housings and exhaust manifolds. Ferritic ductile iron quality EN-GJS-400-18, pearlitic ductile iron quality EN-GJS-500-7 and pearlitic gray iron quality EN-GJL-250 did not show an adequate resistance to oxidation and microstructure changes at 550 °C and 850 °C.

Key words: cast irons, elevated temperatures, microstructure

INTRODUCTION

Automotive industry absorbs about 1/3 of total produced castings in the industrial developed countries. Increased use of cars is not only facing with the energy problems, but also with ecological problems, which include global warming, air pollution, acid rains, ozone destruction, greenhouse effect and waste disposal. Cars manufacturers must increase efficiency of the engines, i.e. reduce the emission of harmful gases and fuel consumption and also increase engine performance and comfort. In order to achieve these demands, new systems of fuel injection and treatment of exhaust gases have been developed, turbochargers are used more, as well as the modern concepts of cars production (downsizing, light weight constructions etc.)^[1, 2].

Higher temperatures of combustion and higher pressures in the cylinders result in higher thermal and mechanical loads of individual components of automotive engine (engine block and cylinder head, turbocharger housing, exhaust manifold etc.). Therefore, modern concepts of automotive engines production require the application of high quality materials, i.e. materials that have adequate high-temperatures properties

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Cast irons with graphite, i.e. gray cast irons which apply for automotive engine components production which are exposed to elevated temperatures (engine blocks, cylinder heads, turbocharger housing, exhaust manifolds etc.) must have adequate resistance to oxidation and growth, resistance to thermal fatigue and sudden temperature changes and adequate mechanical properties^[3–9]. Therefore, engine blocks are made more and more of vermicular cast iron instead of gray cast iron, while turbocharger housings and exhaust manifolds are made of ferritic ductile irons (alloyed with 4,0-6,0 % Si) or austenitic ductile irons (alloyed with 18,0-36,0 % Ni)^[4, 10–13]. For the same structure of metal matrix, ductile iron has better properties at elevated temperatures than gray iron.

The influence of elevated temperatures on microstructure and resistance to oxidation of pearlitic gray iron and ferritic, pearlitic and austenitic ductile irons was analyzed RESULTS AND DISCUSSION in this paper.

MATERIALS AND METHODS

EN-GJL-250, pearlitic ductile iron quality EN-GJS-500-7, ferritic ductile iron quality EN-GJS-400-18, ferritic ductile iron alloyed with Si and Mo quality EN-GJS-SiMo4-1 and austenitic ductile iron quality EN-GJS-AXNiCr20-2 were heated at 550 °C and 850 °C in the furnace for annealing without protective atmosphere. Test samples were held at the mentioned temperatures for 5 h, and then cooled in furnace until the room temperature. For each temperature of examination, a new series of samples were used. The samples were put into the furnace in porcelain cups, which were previously annealed to constant mass at 850 °C. Cups and samples were weighted before and after the heating.

Metallographic examinations were performed by light microscope with digital camera and image analysis system before and after the heating at 550 °C and 850 °C. Shape of graphite, structure of metal matrix and thickness of the oxide layers on the surface of the samples were determined by metallographic examinations. Oxide layers on the samples of ferritic ductile iron quality EN-GJS-SiMo4-1 and austenitic ductile iron quality EN-GJS-AXNiCr20-2 were analyzed by scanning electron microscope with energy dispersive spectrometer (SEM + EDS).

Chemical composition of test samples

Table 1 shows chemical composition of cast irons test samples. Chemical composition of gray iron is usual for quality The samples of pearlitic gray iron quality EN-GJL-250. Higher content of pearlite

Content of element, $w/\%$	Quality						
	EN-GJL-	EN-GJS-	EN-GJS-	EN-GJS-	EN-GJS-		
	250	500-7	400-18	SiMo4-1	AXNiCr20-2		
С	3,43	3,71	3,60	2,90	2,38		
Si	2,15	2,79	2,01	4,61	2,74		
Mn	0,61	0,16	0,17	0,41	0,56		
S	0,090	0,005	0,020	0,015	0,003		
Р	0,031	0,044	0,027	0,045	0,016		
Cu	0,12	0,57	0,03	0,02	0,12		
Sn	0,010	0,014	0,008	0,005	0,006		
Cr	0,07	0,06	0,07	0,09	2,08		
Ni	0,05	0,06	0,02	0,01	21,73		
Мо	0,006	0,009	0,003	1,27	0,019		
Mg	-	0,026	0,054	0,047	0,058		

Table 1. Chemical composition of test samples

in metal matrix was achieved by using a higher content of Mn and Cu.

High content of pearlite in metal matrix of pearlitic ductile iron quality EN-GJS-500-7 was achieved by using higher content of Cu (0,57 %). The content of the carbide-forming elements was low.

Chemical composition of ferritic ductile iron quality EN-GJS-400-18 is usual for this quality. The content of elements which promote the formation of pearlite and carbides was low.

From the chemical composition of ductile iron quality EN-GJS-SiMo4-1 it could be seen that the melt was alloyed with Si (4,61 %) and Mo (1,27 %). By high content of Si it has been tried to achieve full ferritic metal matrix in as-cast condition. Moreover, a high content of Si results in moving of eutectoid transformation temperature toward higher values. In this way phase transformation to austenite moves toward higher temperatures and avoids volume changes which accompany this phase transformation. Moreover, higher Si content improves the resistance to oxidation. Addition of Mo improves the tensile properties and resistance to creep at elevated temperatures. The Mn content was relatively high (0,41 %), which is harmful because Mn segregates during solidification at grain boundaries and promotes the creation of pearlite and carbides. Content of other carbide-forming elements and elements which promote formation of pearlite was low.

Ductile iron quality EN-GJS-AXNiCr20-2 was highly alloyed with Ni (21,73 %), which resulted in creation of austenitic metal matrix in as-cast condition. Addition of Cr improves resistance to oxidation and tensile strength at elevated temperatures.

Results of mass examination of the samples before and after the heating

Table 2 shows results of mass examination of the test samples before and after the heating at 550 $^{\circ}$ C for 5 h.

Quality	Mass of sample, <i>m</i> /g	Mass (sample + cup) before heating, $m_{\rm bh}/{\rm g}$	Mass (sample + cup) after heating, m_{ah}/g	$\Delta m/{ m g}$
EN-GJL-250	18,1254	28,5151	28,5285	0,0134
EN-GJS-500-7	7,4301	17,7772	17,7797	0,0025
EN-GJS-400-18	8,6252	18,8286	18,8309	0,0023
EN-GJS-SiMo4-1	15,3985	22,9902	22,9914	0,0012
EN-GJS-AXNiCr20-2	21,6868	31,0594	31,0594	0,0000

Table 2. Mass of samples before and after the heating at 550 °C for 5 h

Table 3. Mass of samples before and after the heating at 850 °C for 5 h

Quality	Mass of sample, <i>m</i> /g	Mass (sample + cup) before heating, $m_{\rm bh}/{\rm g}$	Mass (sample + cup) after heating, m_{ah}/g	$\Delta m/{ m g}$
EN-GJL-250	16,3504	26,5537	26,9066	0,3529
EN-GJS-500-7	8,1970	15,7898	15,9146	0,1248
EN-GJS-400-18	8,3403	18,0365	18,1406	0,1041
EN-GJS-SiMo4-1	15,5324	24,8985	24,9052	0,0067
EN-GJS-AXNiCr20-2	10,2913	20,6811	20,6830	0,0019

From Table 2 it could be seen that all the test samples, except austenitic ductile iron (EN-GJS-AXNiCr20-2), show increasing of mass after heating at 550 °C, which is an indicator of oxidation, i.e. formation of oxide layer. Gray iron shows the largest increase of mass. Ferritic ductile irons (EN-GJS-400-18, EN-GJS-SiMo4-1) have a smaller increase of mass than pearlitc ductile iron (EN-GJS-500-7).

Table 3 shows results of mass examination of the test samples before and after the heating at 850 $^{\circ}$ C for 5 h.

From Table 3 it could be seen that all the test samples show increasing of mass after the heating at 850 °C, which is an indicator of oxidation, i.e. formation of oxide layer. Gray iron shows highest increase of mass. Austenitic ductile iron shows lowest increase of mass. Ferritic ductile irons have a smaller increase of mass than pearlitc ductile iron.

Microstructure analysis of the samples before and after the heating

From Figure 1 it could be seen that oxidation occurred during the heating of samples of gray iron quality EN-GJL-250 at 550 °C and 850 °C for 5 h. Formed oxide layers do not have the protective effect because they were not compact and tightly connected with metal matrix, which allows progression of oxidation to the samples inside.

During the heating at 550 °C, the changes in the metal matrix did not occurred. The partial decomposition of pearlite and the accompanying growth occurred during the heating at 850 °C.

From Figure 2 it could be seen that oxidation occurred during the heating of samples of ductile iron quality EN-GJS-500-7 at 550 °C and 850 °C for 5 h. Oxide layer formed on the surface of the sample during the heating at 550 °C was relatively



Figure 1. Optical micrographs of the oxide layers on the surface of the samples of pearlitic gray iron quality EN-GJL-250: a) after the heating at 550 °C, b) after the heating at 850 °C

thin and tightly connected with metal matrix. Oxide layer formed on the surface of sample during the heating at 850 °C was significantly thicker than oxide layer formed during the heating at 550 °C and was not tightly connected with metal matrix, which allows progression of oxidation to the sample inside.

During the heating at 550 °C, because of partial decomposition of pearlite, increasing of ferrite content in metal matrix occurred in relation to the state before heating. Further increase of ferrite content in metal matrix occurred during the heating at 850 °C.



Figure 2. Optical micrographs of the oxide layers on the surface of the samples of pearlitic ductile iron quality EN-GJS-500-7: a) after the heating at 550 °C, b) after the heating at 850 °C



Figure 3. Optical micrographs of the oxide layers on the surface of the samples of ferritic ductile iron quality EN-GJS-400-18: a) after the heating at 550 °C, b) after the heating at 850 °C

From Figure 3 it could be seen that oxidation occurred during the heating of samples of ductile iron quality EN-GJS-400-18 at 550 °C and 850 °C for 5 h.

Figure 3 a shows that oxide layer formed on the surface of the sample during the heating at 550 °C was relatively thin and tightly connected with metal matrix. Oxide layer formed on the surface of the sample during the heating at 850 °C was significantly thicker than oxide layer formed during the heating at 550 °C and was not tightly connected with metal matrix, which allows progression of oxidation to the sample inside (Figure 3 b).

Because of partial decomposition of pearlite during the heating at 550 °C, increasing of ferrite content in metal matrix occurred in relation to the state before heating. Decomposition of pearlite occurred in a greater extent during the heating at 850 °C.

From Figure 4 it could be seen that oxida-

tion occurred during the heating of samples of ductile iron quality EN-GJS-SiMo4-1-at 550 °C and 850 °C for 5 h.

The average thickness of oxide layer formed during the heating at 550 °C was 4,33 μ m, i.e. 77,72 μ m during the heating at 850 °C. This means that the alloyed ferritic ductile iron quality EN-GJS-SiMo4-1 has a much better resistance to oxidation than gray iron and non-alloyed ferritic and pearlitic ductile iron. Oxide layers were tightly connected with metal matrix and prevent progression of oxidation to the sample inside.

From Figure 5 it could be seen the layered structure of oxide layer. Outward layer consisted of iron oxide (Figure 6). Inward layer, i.e. the layer alongside the base (nonoxidized) material consisted of iron silicate (fayalite) (Figure 7). This oxide layer prevents progression of oxidation to the sample inside. In this way resistance to hightemperature oxidation was improved.



Figure 4. Optical micrographs of the oxide layers on the surface of the samples of ferritic ductile iron quality EN-GJS-SiMo4-1: a) after the heating at 550 °C, b) after the heating at 850 °C

During the heating at 550 °C and 850 °C increasing of ferrite content in metal matrix occurred in relation to the state before heating. Decomposition of pearlite occurred in metal matrix before the heating.

a greater extent during the heating at 850 °C. Higher content of Mn (0,41 %) is a potential cause of the presence of pearlite in



Figure 5. SEM micrograph of oxide layer formed on the surface of the sample of ductile iron quality EN-GJS-SiMo4-1 during the heating at 850 °C for 5 h. Places analyzed by the use of EDS are marked in the picture.



Figure 6. EDS spectrum of outward oxide layer formed on the surface of the sample of alloyed ferritic ductile iron quality EN-GJS-SiMo4-1 during the heating at 850 °C for 5 h. Place where analysis was performed is marked in the figure 5



Figure 7. EDS spectrum of inward oxide layer formed on the surface of the sample of alloyed ferritic ductile iron quality EN-GJS-SiMo4-1 during the heating at 850 °C for 5 h. Place where analysis was performed is marked in the figure 5

Because of higher Mo content (1,27 %), in ferritic ductile iron quality EN-GJS-SiMo4-1 precipitation of primary carbides at grain boundaries and secondary carbides in ferrite occurred (Figure 8). At low magnification, carbides precipitated at grain boundaries are similar to pearlite. A clear difference can be seen at higher magnification (Figure 8). In both cases these are complex carbides, which are composed of iron, molybdenum, silicon and carbon (Figure 9).

During the heating at 550 °C and 850 °C, increasing of amount of fine secondary carbides in ferrite occurred. Higher increase in the amount of carbides was present after the heating at 850 °C.



Figure 8. Optical micrographs of microstructure of ductile iron quality EN-GJS-SiMo4-1 before the heating, etched. From the figure it could be seen primary carbides at grain boundaries and fine secondary carbides in ferrite



b)

Figure 9. EDS spectum of primary (a) and secondary (b) carbides in ferritic ductile iron quality EN-GJS-SiMo4-1

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The presence of complex primary and resistance to creep and thermal fatigue. secondary carbides in the microstructure GJS-SiMo4-1 results in increasing of ten-

of alloyed ferritic ductile iron quality EN- From Figure 10 a it could be seen that oxidation occurred in a very small extent sile strength at elevated temperatures and during the heating of samples of austenitic



Figure 10. Optical micrographs of the oxide layers formed on the surface of the samples of austenitic ductile iron quality EN-GJS-AXNiCr20-1: a) after the heating at 550 °C, b) after the heating at 850 °C



Figure 11. SEM micrograph of oxide layer formed on the surface of the sample of austenitic ductile iron quality EN-GJS-AXNiCr20-1 during the heating at 850 °C for 5 h. Places analyzed by the use of EDS are marked in the picture.

ductile iron quality EN-GJS-AXNiCr20-2 at 550 °C. This resulted in the formation almost negligible oxide layer on certain places. Oxide layer formed on the surface of the sample during the heating at 850 °C was tightly connected with metal matrix (Figure 10 b). The average thickness of oxide layer was 45,58 µm. Oxide layer formed during the heating austenitic ductile iron at 850 °C was thinner than oxide layers formed during the heating other

analyzed materials at 850 °C. This shows the superiority of austenitic ductile iron in comparison to other analyzed materials.

From Figure 11 it could be seen the layered structure of oxide layer formed during the heating at 850 °C.

Inward oxide layer, i.e. the layer alongside the base (non-oxidized) material was complex oxide which consisted of Fe, Cr,



Figure 12. EDS spectra of oxide layer formed on the surface of the sample of austenitic ductile iron quality EN-GJS-AXNiCr20-1 during the heating at 850 °C for 5 h: a) inward oxide layer, b) outward oxide layer. Places where analysis were performed are marked in the figure 11

Ni, Si and O (Figure 12 a). Outward layer consisted of Fe and O (Figure 12 b). Complex oxide layer alongside the base material prevents progression of oxidation to the sample inside. In this way resistance to high-temperature oxidation was improved. During the heating at 550 °C and 850°C, the changes in the metal matrix did not occurred. Because there are no phase transformations, it could be concluded that the volume changes and the accompanying growth will not occur during the application of austenitic ductile iron at 550 °C and



Spectrum 1 Spectrum 1 Spectrum 1 Spectrum 1 0 1 2 3 4 5 6 7 8 9 10 Full Scale 2168 cts Cursor: 0.000 b)

Figure 13. a) SEM micrograph of primary carbides in austenitic ductile iron quality EN-GJS-AXNiCr20-1, b) EDS spectum of primary carbides in austenitic ductile iron quality EN-GJS-AXNiCr20-1

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Figure 14. Thickness of oxide layers formed on the surface of the samples during the heating at 550 °C for 5 h: 1 - gray iron quality EN-GJL-250; 2 - ductile iron quality EN-GJS-500-7; 3 ductile iron quality EN-GJS-400-18; 4 - ductile iron quality EN-GJS-SiMo4-1; 5 - ductile iron quality EN-GJS-AXNiCr20-2

850 °C. This shows the superiority of austenitic ductile iron in comparison to other analyzed materials.

During the solidification of ductile iron quality EN-GJS-AXNiCr20-1 precipitation of primary carbides at grain boundaries occurred (Figure 13a). These are complex carbides, which are composed of iron, chromium, manganese and carbon (Figure 13b).

Figures 14 and 15 summarily show results of measurements of oxide layers thickness formed during the heating at 550 °C and 850 °C for 5 h.

From the Figures 14 and 15 it could be seen that austenitic ductile iron quality EN-GJS-AXNiCr20-2 is the most suitable for construction of castings which will be applied at elevated temperatures. Gray iron



Figure 15. Thickness of oxide layers formed on the surface of the samples during the heating at 850 °C for 5 h: 1 - gray iron quality EN-GJL-250; 2 - ductile iron quality EN-GJS-500-7; 3 ductile iron quality EN-GJS-400-18; 4 - ductile iron quality EN-GJS-SiMo4-1; 5 - ductile iron quality EN-GJS-AXNiCr20-2

quality EN-GJL-250 is not suitable material for castings which will be applied at elevated and high temperatures.

CONCLUSIONS

From the examination of microstructure properties and oxide layers and measurements of mass before and after the heating of gray and ductile irons samples at 550 °C and 850 °C for 5 h the following conclusions could be performed:

during the heating at 550 °C and 850 °C for 5 h in the samples of pearlitic gray iron quality EN-GJL-250, pearlitic ductile iron quality EN-GJS-500-7, ferritic ductile iron quality EN-GJS-400-18, ferritic ductile iron quality EN-GJS-SiMo4-1 and austenitic ductile iron quality EN-GJS-SiMo4-1 and austenitic ductile iron quality EN-GJS-AXNiCr20-2 oxidation, increasing of

mass and microstructure changes occur,

- the highest resistance to oxidation, i.e. smallest thickness of oxide layer and smallest increasing of mass during the heating at 550 °C and 850°C was determined at austenitic ductile iron quality EN-GJS-AXNiCr20-2, followed by ferritic ductile iron quality
 EN-GJS-SiMo4-1, thereafter ferritic ductile iron quality EN-GJS-400-18 and pearlitic ductile iron quality EN-GJS-500-7. Pearlitic gray iron quality EN-GJL-250 shows the worst resistance to oxidation,
- during oxidation at elevated temperatures two oxide layers over the surface of the samples of ductile iron qualities EN-GJS-SiMo4-1 and EN-GJS-AXNiCr20-2 occur. Outward layer is easy detachable and consists of iron oxide. Inward layer is tightly connected with metal matrix and prevents progression of oxidation to the samples inside. This oxide layer consists of Fe₂SiO₄ (fayalite) or complex oxides (Fe, Cr, Ni, Si, and O),
- for all analyzed materials, thickness of oxide layer formed during the heating at 850 °C for 5 h is greater than thickness of oxide layer formed during the heating at 550 °C for 5 h,
- if oxide layer is compact and tightly connected with metal matrix it has a protective effect and prevents progreassion of oxidation to the sample inside,
- during the heating at 550 °C and 850 °C for 5 h in the samples of pearlitic gray iron quality EN-GJL-250, pearlitic ductile iron quality EN-GJS-500-7,

ferritic ductile iron quality EN-GJS-400-18 and ferritic ductile iron quality EN-GJS-SiMo4-1 decomposition of pearlite occur and increasing of ferrite content in metal matrix. This phase transformation occur in a greater extent during the heating at 850 °C and results in growth of cast irons,

- ductile iron has higher resistance to elevated temperatures than gray iron due to discontinuous nature of graphite nodules. Continuous nature of graphite lamellas in gray iron allows quick progression of oxidation to the sample inside,
- to achieve a high resistance to oxida-• tion, formation of scale and growth during the application of cast irons at elevated temperatures it is necessary to perform appropriate alloying to obtain full ferritic (alloying with 4,0-6,0 % Si) or austenitic (alloying with min. 18,0 % Ni) metal matrix, i.e. avoid formation of pearlite in metal matrix and accompanying phase transformations. For the same graphite shape, austenitic metal matrix gives superior properties than ferritic metal matrix, i.e. allows the application of casting at higher temperatures,
 - regarding to severe requests toward automotive industry in regard of increasing of engine efficiency and accompanying increasing of exhaust temperatures, it is obviously that demand for ferritic ductile irons which are alloyed with Si and Mo (SiMo) and austenitic ductile iron which are highalloyed with Ni will increase. Because of suitable properties of these materials at elevated temperatures, applica-

tion of low-alloyed and high-alloyed ^[8] gray irons for production turbocharger housings and exhaust manifolds, i.e. automotive engine thermo-mechanical loaded parts will be reduced. ^[9]

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