Influence of thermal effect on the structure and properties of die steel 70Kh3G2FTR(m)

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Conventional heat-resistant die ferrite-pearlite steels with carbide and mixed (carbide-intermetallic) hardening are softened very intensively after heating above 350-400 °C, what leads to rapid tool failure. Heat-resistant austenite steels and alloys also does not solve the problem of operating resistance of die tools, due to their tendency to cracking, poor machinability by cutting, high cost and scarcity of alloying elements included in their composition.
Investigations of structural state, thermophysical properties and physical and technical characteristics of the developed die steel 70Kh3G2FTR(m) were carried out in this work under thermal exposure. Considerations about the structural stability of the alloy are confirmed by the data of X-ray diffraction analysis within the temperature range from −187 °C to 300 °C, as well as of transmission electron microscopy (TEM) at all stages of heat treatment and thermal exposure. It is shown that the developed steel 70Kh3G2FTR(m) after the proposed heat treatment procedure is characterized by thermal and structural stability within the studied temperature range up to 450 °C, due to influence of a microalloying complex in the form of dispersed carbide inclusions registered by TEM. The dispersion hardening mechanism in the developed alloy prevents motion of dislocations during heating, distorts and seals the atomic crystal structure, slowing down diffusion processes and migration transmissions of grain boundaries.
Key words: sparingly alloyed steels, carbide hardening, heat treatment, microalloying, transmission microscopy, diffraction analysis, thermophysical properties.
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Introduction

Increase of workability of heavy-loaded metallurgical tools is an actual production and scientific-research problem. Resistance of die tools, which depends on material and heat treatment procedure, is the main parameter determining die tool quality. Use of new grades of alloy steels, as well as development and improvement of their thermal hardening procedures, provide a complex approach to solving the problem of improvement of properties and workability of tools, which are manufactured from these steels [1].

It is known that pressure casting (PC) is one of the most efficient production methods for manufacture of complicated thin-wall parts from aluminium alloys. Based on the requirements of international standards, the components of pressure casting dies in PC machines should keep 25,000-30,000 pressing operations without deterioration of mechanical properties and operating resistance [2-4].

Influence of operating conditions on workability of pressure casting dies in PC machines is considered in the work [5]; it is noted there, that cyclic temperature-force and physical-chemical interaction between material of forming elements and liquid melt occurs during operation. Simultaneous stopping of melt feed at the finishing moment of filling causes a powerful hydraulic shock, what provides forming of complicated stress state in pressure casting dies (in combination with thermal effect from pouring metal – up to 1100 °C for Fe-base alloys). After definite number of pressing operations (thermocycles) in the most loaded die surface layer, origination and further propagation of thermal fatigue cracks occurs; it leads to destruction, especially in the points of stress concentration.

Metal scientists have the opinion, that stability in cyclic loading and thermal effect conditions is the main parameter determining workability of metallurgical equipment, i.e. dies and punches of PC machines. Such stability depends directly on material chemical composition and method of thermal hardening.

Accumulated experience of industrial operation of PC machines revealed the main requirements for the steels used in the components of pressure casting dies: quenching ability 56-58 HRC for surface strength within the range 800-900 MPa, with corresponding hardenability not lower than 52 HRC at the depth 80-100 mm; wear resistance in the conditions of static and cyclic loading; manufacturing ability during thermal hardening, with saving constant sizes during operation [6].

At present time, pressure casting die parts, dies, punches, stamping dies are manufactured both as-forged and as-cast, using mainly medium-carbon tool steels (such as 4KhMFS, 5KhNM, 5KhGM). However, these materials are characterized by insufficient operating resistance, when they are used

for manufacture of the components of pressure casting dies in PC machines; the first cracks and erosion network appear starting from the temperature 300-350 °C in the conditions of small thermal loading cycles and force effect. Taking into account the above-described circumstances, development of the rational technology for thermal hardening of sparingly alloyed die steels, which allows to provide the required mechanical and operating properties of metallurgical tools under cyclic thermal effect, seems to be rather actual metallurgical task [7].

The aim of this research concludes in determination of the influence of structure and phase composition for the developed die steel 70Kh3G2FTR(m) on providing of thermal stability within the temperature range from –187 °C to 300 °C.

Materials and methods of the research

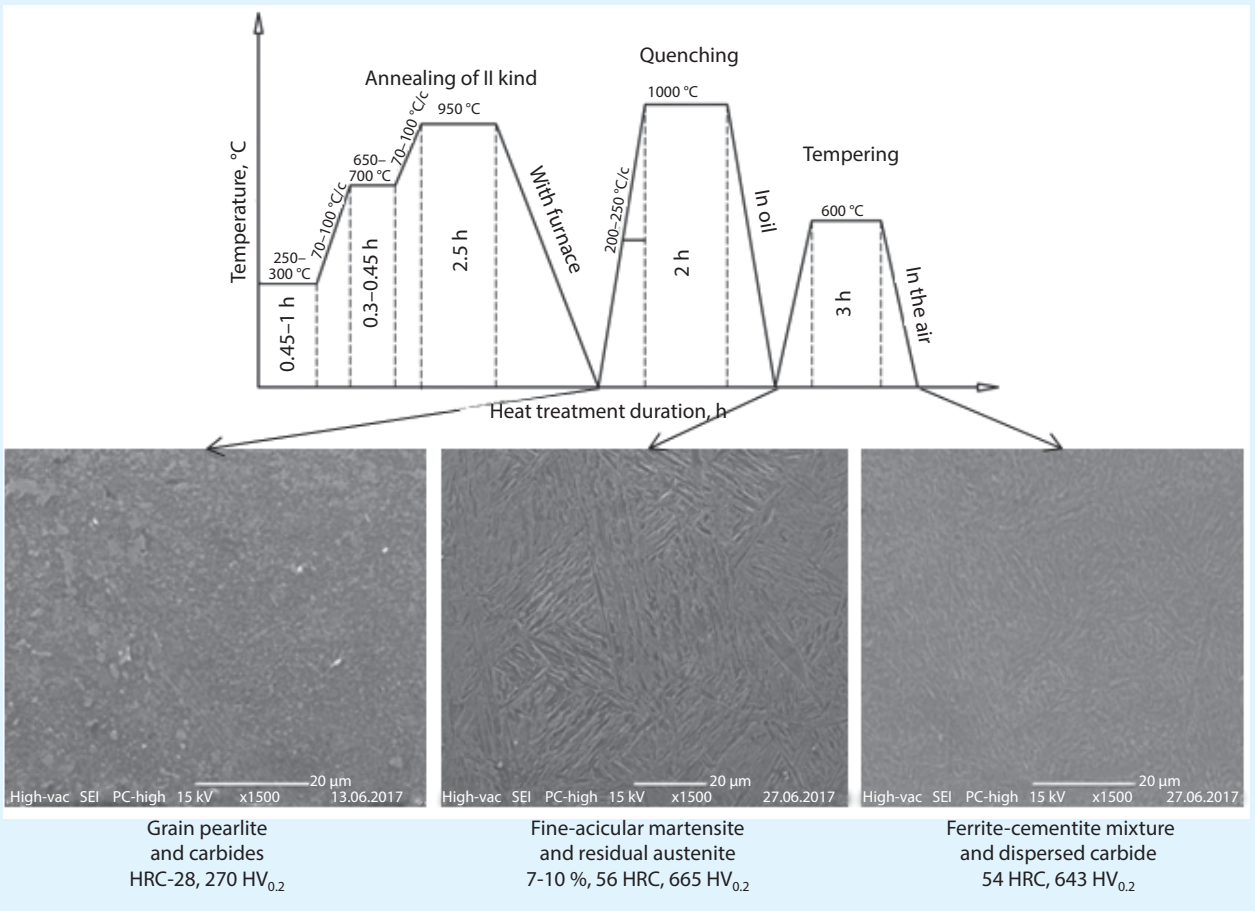
The previously developed and patented tool steel 70Kh3G2FTR(m), which was recommended for manufacture of dies for hot deformation, were used as basic material for conducting of this research [8]. The chemical composition of the pilot melt in this research differs from the patented one by presence of the modifying elements (selenium, zirconium and tantalum) in its composition; these modifiers were introduced in the known steel for the first time, in order to provide refinement of primary cast structure. The experimental modified steel was marked as 70Kh3G2FTR(m) (**Table 1**).

Determination of temperature conductivity of the samples was carried out via the method of laser flash in correspondence with ASTM E1461-07 and using the NETZSCH LFA447 NanoFlash unit. The experiments were conducted with the samples having diameter 12.7 mm and thickness 0.5-1.0 mm, which were obtained via cold pressing; the measurements were carried out within the temperature range from 25 °C to 300 °C [9].

X-ray structural examination of the samples of the steel 70Kh3G2FTR(m) were conducted at the room temperature via diffractometer Rigaku Mini Flex 600, using CoKa-radiation and linear semiconducting detector D/tex Ultra

Table 1. **Chemical composition of the modified steel 70Kh3G2FTR(m), % (mass.)**

C	Si	Mn	S	P	Cr	V	Ti	Ni	Cu	Al	Co	Se	B	Zr	Ta
0.72	0.67	2.46	0.019	0.01	3.31	0.4	0.16	0.24	0.1	0.002	0.019	0.12	0.0021	0.0011	0.018



Grain pearlite and carbides HRC-28, 270 $HV_{0.2}$

Fine-acicular martensite and residual austenite 7-10 %, 56 HRC, 665 $HV_{0.2}$

Ferrite-cementite mixture and dispersed carbide 54 HRC, 643 $HV_{0.2}$

Fig. 1. Technological chart and 70Kh3G2FTR(m) steel controlled structures, ×1000

(Rigaku, Japan). X-ray structural examination within the temperature range from –187 °C to 300 °C were carried out via multifunctional diffractometer Rigaku Ultima IV, using CoKa–radiation, graphite monochromator on diffracted beam of scintillated detector and low/medium-temperature auxiliary device LMT (Rigaku, Japan). Analysis of the spectra was conducted using the software product PDXL2 (Rigaku, Japan) and the database of powder diffraction patterns PDF-2 (ICDD); phase analysis was implemented via the Ritweld method, lattice period was determined via interpolation method using rectifying Taylor-Sinclair-Nelson-Riley function. Accuracy of determination of the crystal lattice parameters made ±0,0005 Å, the error of determination of the phase composition was ±5 %.

To conduct the researches, a transmission electron microscope JEM-1400 with accelerated voltage 120 kV (JEOL, Japan) was used. Scanning electron microscopy was carried out using the microscope TescanVega 3SB with energy-dispersion auxiliary device X-Act Oxford Instrument for micro-X-ray spectral analysis with accelerated voltage 20 kV. Sample preparation was conducted via the method of electrolytic polishing in the unit Struers Tenupol as well as via the method of ion pickling in the unit PIPS Gatan.

Obtained results

Based on the previously conducted researches [10], technological parameters of thermal hardening were substantiated. These parameters provide the required complex of mechanical properties of the steel 70Kh3G2FTR(m): hardness 50-55 HRC, fatigue strength not lower than 1480 MPa, impact toughness within the range 49-59 J/cm² [11].

The obtained results allowed to recommend the following heat treatment procedure for a punch of pressure casting machine for the steel 70Kh3G2FTR(m): annealing of II kind at 950 °C, holding during 2-3 hours (combining heating and cooling with a furnace); quenching in oil at 1000 °C with consequent high-temperature tempering at 560-600 °C, holding during 3 hours and cooling in the air (**Fig. 1**) [12]. It was established that this heat treatment procedure allows to form the required structure and finished parameters in the developed microalloyed steel, according to the technical and designing documentation for a pressure casting die of pressure casting machine for aluminium alloys.

Investigations via transmission electron microscopy (TEM) were conducted at different stages of heat treatment. It was established after annealing of II kind and cooling with a furnace, that the structure is presented by dispersed lamellar ferrite-cementite distance appr. 100-150 nm (**Fig. 2*a***).

In addition to lamellar pearlite, spherical inclusions with diameter about 100-200 nm were revealed in the structure. All inclusions, which were analyzed via electron diffraction patterns, were interpreted based on suggestion of carbide phase Cr_7C_3 (dimensional group Pnma); corresponding light field diffraction images are presented on the **Fig. 3**.

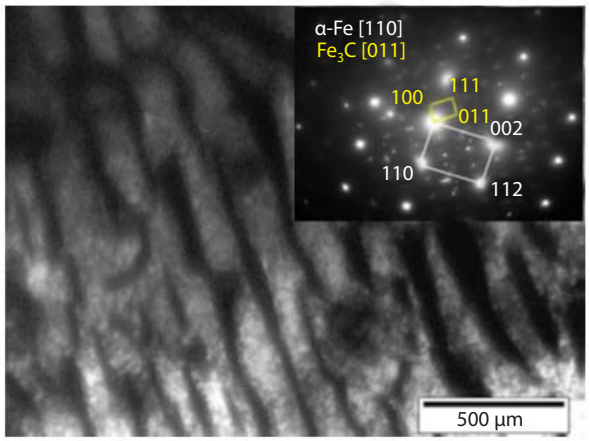


Fig. 2. Light field diffraction image of the pearlite structure, which is observed in the steel 70Kh3G2FTR(m) via the TEM method after annealing of II kind (corresponding electron diffraction pattern is presented at the insert image)

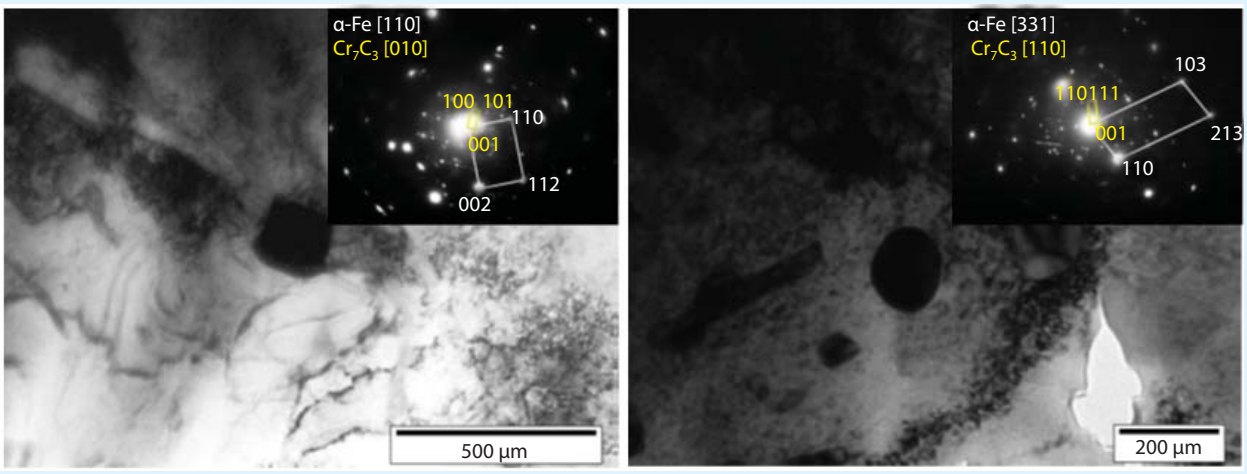
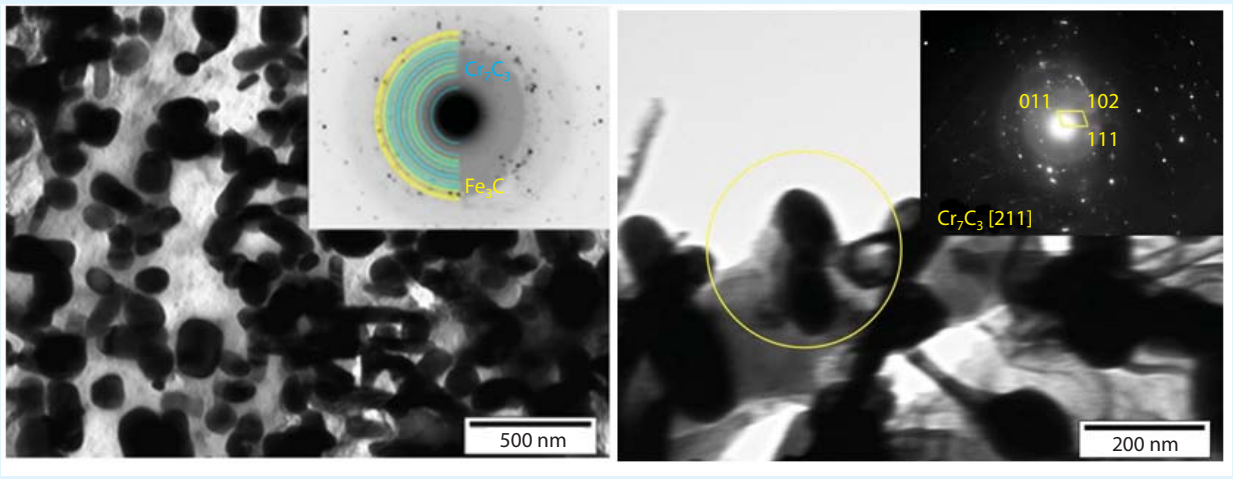
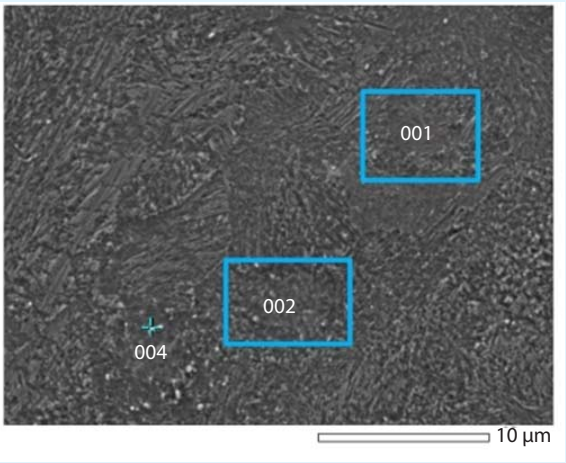


Fig. 3. Light field diffraction images of carbides particles, which were revealed in the steel 70Kh3G2FTR(m) via the TEM method after annealing of II kind and cooling with a furnace (corresponding electron diffraction patterns are presented at the insert images)



a *b*

Fig. 4. Light field diffraction image of the steel 70Kh3G2FTR(m) microstructure after quenching from 1000 °C and tempering at 600 °C via the TEM method (corresponding electron diffraction pattern is presented at the insert images)



%(mass.)

№ of spectrum	C	Si	Mn	V	Cr	Ti	Fe
001	5,02	0,22	0,57	1,67	1,05	0,36	91,11
002	6,41	0,18	1,05	1,25	1,12	0,95	89.04
004	14,04	2,36	3,69	0,27	4,84	1,87	72,93

Fig. 5. The structure and results of micro-X-ray spectral analysis of the developed steel 70Kh3G2FTR(m) after heat treatment (content in %, mass.)

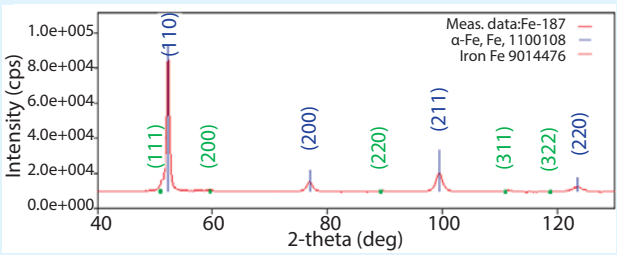


Fig. 6. Combined electron diffraction pattern of the steel 70Kh3G2FTR(m) after heat treatment, based on the results of phase analysis within the temperature range from −187 °C to 300°C, in CuK_α radiation

However, the lattice parameters in different electron diffraction patterns vary by the value exceeding the measurement error, what can testify on variation of carbides composition.

Consequent quenching and high-temperature tempering at the temperature 600 °C led to forming of the grain pearlite structure (**Fig. 4*a***). The electron diffraction pattern, which was obtained from all area that is seen on this figure, was characterized by reflections located on interplane distances and corresponding to the Cr_7C_3 phase (in addition to reflections corresponding to cementite). Presence of this phase was confirmed by obtaining of a point electron diffraction pattern from the particle which is marked on the Fig. 4*b*.

Conduction of additional investigations of carbide phases via the method of micro-X-ray spectral analysis displayed that more complicated carbide compounds, containing high-melting-point elements (titanium and vanadium), in addition to Cr_7C_3-type carbides, are presented in structure of the developed steel 70Kh3G2FTR(m) (**Fig. 5**).

Testing on determination of structural thermal stability and transition temperatures after complete cycle of heat treatment (annealing at 950 °C; quenching in oil from 1000 °C; tempering at 600 °C) was carried out using temperature phase analysis. The electron diffraction pattern, which was obtained in the temperature range from −187 °C to 300 °C, is shown on the **Fig. 6**.

Based on the data of electron diffraction patterns, it was revealed that no phase transformations occur in this temperature range, only variation of lattice distance takes place (**Table 2**).

The obtained data testify on high phase stability of the developed steel 70Kh3G2FTR(m) the temperature range from −187 °C to 300°C, what allows to predict high operating parameters of this steel [13].

The conducted analysis of temperature conductivity (**Fig. 7**) displays that the data on two samples No. 3 and No. 4 coincide practically completely in initial state without heat treatment. At the same time, the developed steel in "raw" state is characterized by higher temperature con-

ductivity in the range 6.0-6.5 mm²/s, while the data on two samples No. 1 and No. 2 after heat treatment are different and correspond to the values in the range 4.0-4.5 mm²/s.

As soon as electronic conductivity is the main mechanism of temperature conductivity, oscillations of atoms and dispersion of electrons intensify with testing temperature rise. In its turn, it causes expected reducing of temperature conductivity, which is especially visible in the initial state, when temperature conductivity decreases intensively with testing temperature rise. The samples, which were tested after heat treatment, are more stable during heating up to 300 ºC: temperature rise leads to slow decrease of temperature conductivity. It can be seen that the samples after heat treatment are characterized by principally lower temperature conductivity in comparison with the initial state (cast ferrite-carbide structure with presence of carbide network and heterogeneous distribution of carbide inclusions), what is explained by heat treatment effect. This heat treatment provides structure ordering, complication of atomic-crystalline structure and increase number of defects as well as distortion of crystalline structure, what in its turn hampers motion of electrons and supports their dissemination [14]. Carbide inclusions, which were identified by TEM, play this role in the examined alloy.

Based on the analysis of temperature conductivity, thermal capacity (determined by differential scanning calorimetry – DSC method) and analysis of samples density, thermal conductivity of the examined samples was calculated via the equation (1) (**Table 3**).

$$\lambda(t)=a(t)\cdot d_k\cdot C_p(t) \quad (1)$$

where $\lambda(t)$ – thermal conductivity at the preset temperature, Wt/m·K

$a(t)$ - value of temperature conductivity for the temperature t, mm²/s

d_k - material density, g/cm³

$C_p(t)$ – specific thermal capacity, J/(g·K)

According to the results of calculations, presented in the Table 3, it can be seen that thermal conductivity of the alloy in the initial state is higher than that in the alloy after heat treatment. However, the alloy after heat treatment is more stable during temperature rise, its thermal capacity varies within the narrow range of values in comparison with the initial state without heat treatment.

The graphs of thermal capacity variation are presented on the **Fig. 8**. It can be seen that the steel 70Kh3G2FTR(m) before heat treatment saves the stable values of thermal capacity up to the temperatures 350-400 ºC (Fig. 8*a*), while after hardening heat treatment we can see variations to the side of higher temperature range of stable thermal capacity values. Stability of the values is observed up to the temperatures 650-750 ºC (Fig. 8*b*), what provides possibility of stable

Table 2. **Temperature relationship of lattice distance in α-phase (Im3m) for the steel 70Kh3G2FTR(m) after heat treatment**

Temperature, ºC	Lattice period in α-phase (Im3m), nm
300	0.28848±0.00008
200	0.28815±0.00008
100	0.28784±0.00008
25	0.28765±0.00008
-187	0.28721±0.00008

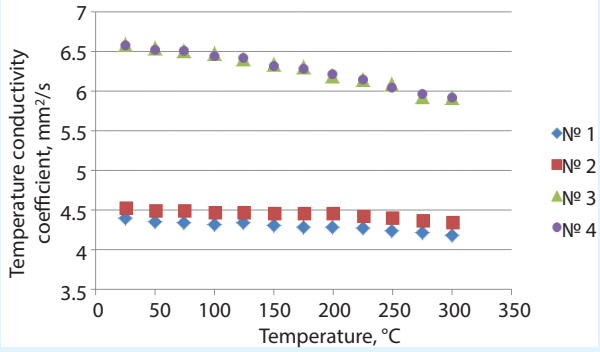


Fig. 7. Relationship between the temperature conductivity coefficient and testing temperature for the steel 70Kh3G2FTR(m) (the samples No. 1 and No. 2 – afterheat treatment; the samples No. 3 and No. 4 – without heat treatment, in initial state)

Table 3. **Thermal-physical properties of the examined samples**

T, ºC	25	50	75	100	125	150	175	200	225	250	275	300
Initial sample (density 7.62 g/cm³)	Temperature conductivity, mm²/s											
	6.59	6.54	6.50	6.47	6.40	6.34	6.29	6.19	6.13	6.08	5.95	5.92
	Thermal capacity, J/(g·K)											
	-	0.50	0.51	0.52	0.52	0.52	0.52	0.51	0.51	0.50	0.50	0.49
	Thermal conductivity, Wt·m⁻¹·K⁻¹											
	-	24.92	25.26	25.64	25.36	25.12	24.92	24.06	23.82	23.16	22.67	22.1
Sample after heat treatment (density 7,73 g/cm³)	Temperature conductivity, mm²/s											
	4.39	4.36	4.35	4.33	4.33	4.31	4.28	4.28	4.26	4.24	4.22	4.18
	Thermal capacity, J/(g·K)											
	-	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.49	0.49
	Thermal conductivity, Wt·m⁻¹·K⁻¹											
	-	16.85	16.81	16.73	16.73	16.66	16.54	16.54	16.46	16.39	15.98	15.8

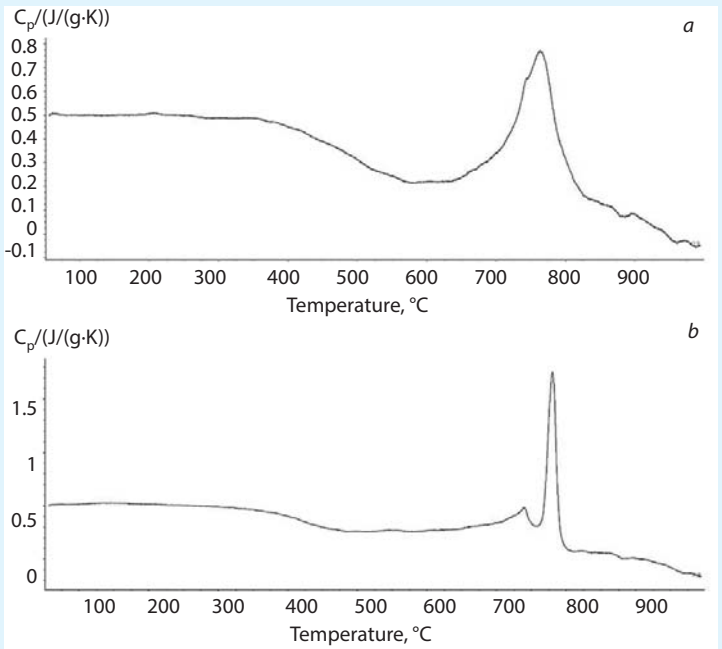


Fig. 8. The results of analysis of thermal capacity of the steel 70Kh3G2FTR(m): in the initial state (*a*), after heat treatment (*b*)

operation with this steel after the complex of measures aimed on thermal hardening without forming of hot cracks in the above-mentioned temperature range.

The graphic relationships, which are presented on the Fig. 8, contain definite endothermic effect within the temperature range 750-780 ºC. Endothermic effects are most probably connected with Fe polymorphic transformations (BCCL to FCCL). Value of this effect is connected supposedly with the volumes of phase transformations, because there is more ferrite in the developed steel (in the initial state, without heat treatment) than in the sample after thermal hardening; it is confirmed by the TEM data.

The values of thermal-physical parameters obtained in this research show good correlation with the previously obtained values of mechanical and technological properties in the above-noted temperature range for the steel 70Kh3G2FTR(m) [15].

Conclusion

It was established that hardening heat treatment of the developed steel 70Kh3G2FTR(m) treatment (annealing at 950 °C; quenching in oil from 980-1000 °C; tempering at 600 °C) not only provides hardening effect owing to extraction of dispersed carbides with complicated composition, but also additionally stabilize the values of thermal-physical parameters within the temperature range from –187 °C to 350 °C.

Assessment of thermal structural stability displayed that such thermal-physical parameters of the steel 70Kh3G2FTR(m) as thermal capacity and temperature conductivity are characterized by increased stability up to 300-400 ºC after heat treatment, while thermal capacity after hardening heat treatment varies seriously to the higher temperature range of stability and holds stability up to 750 ºC. Stability of thermal-physical parameters is explained by presence of dispersed carbide phases of Cr_7C_3 type and more complicated carbide inclusions, containing high-melting titanium and vanadium, in the structure of sparingly alloyed steel.

The regularities of variation of the phase composition within the temperature range of thermal stability assessment are established. It was revealed that the phase composition, which is presented as fine-lamellar ferrite-cementite mix with carbide hardening, does not vary with temperature rise up to 300 ºC, what correlates with the previously obtained data of dilatometric analysis. Assessment of the values of temperature conductivity, thermal capacity and heat conductivity allows to predict trouble-free operation of the die steel 70Kh3G2FTR(m) as a material for the components of pressure casting dies of pressure casting machines for aluminium alloys.

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