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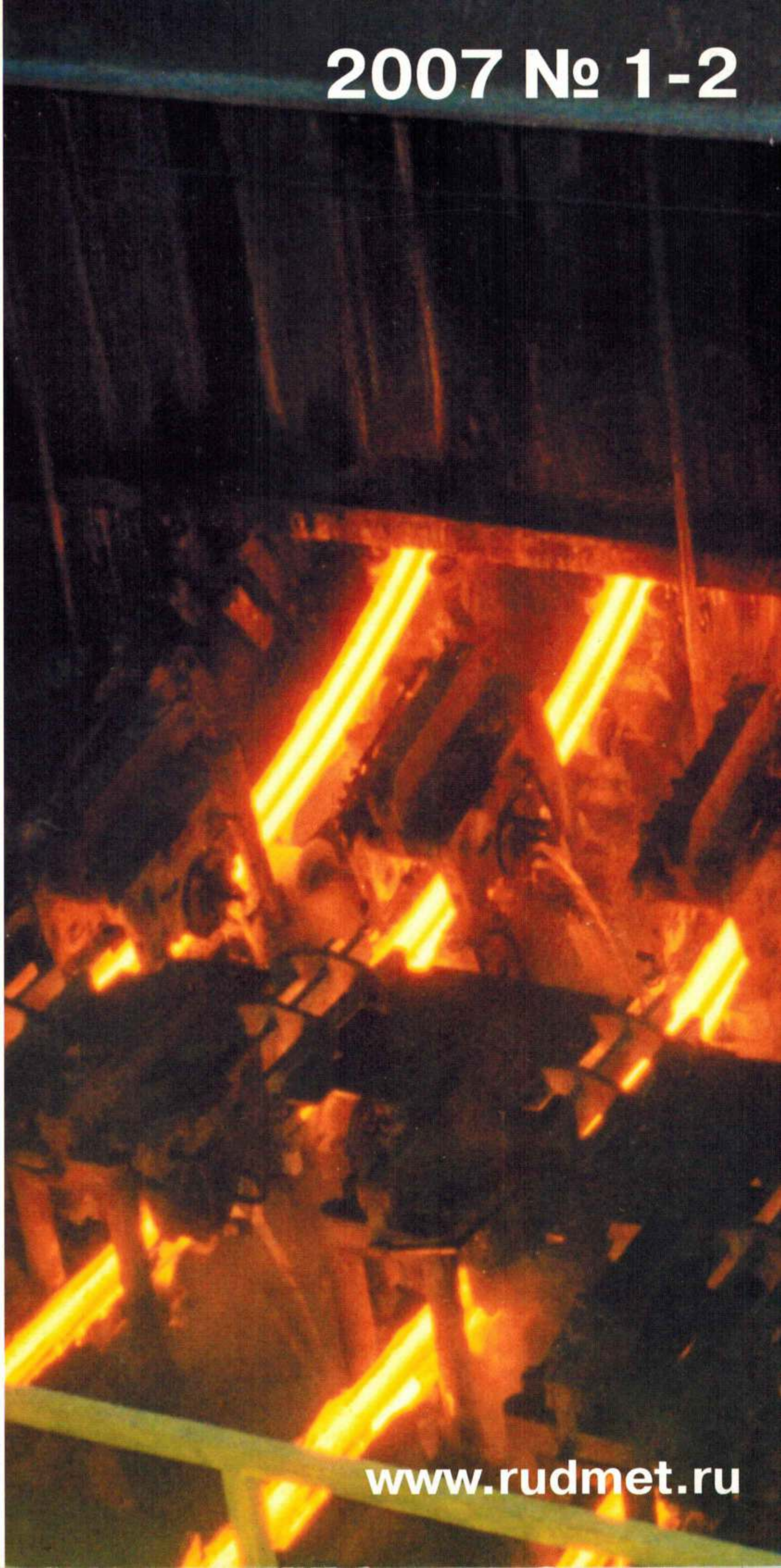

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Metallurgical expertise as the base for determination of nature of defects in metal products

The original technique of metallurgical expertise has been developed for searching the causes of defects in metal products. This technique includes panoramic metallographic investigations of metal defect areas using "Thixomet" image analyzer, micro X-ray analysis of nonmetallic inclusions revealed in defect area, thermodynamic simulation of phase formation processes occurring in liquid and solidifying steel as well as analysis of through technology of metal production. Such approach to investigation of defects allows to formulate reasoned opinions about their nature. The paper deals with some of defects of different steel grades; these defects have been originated due to nonmetallic inclusions in hot-rolled and cold-rolled sheet metal. Evolution of nonmetallic inclusions formed during steel deoxidation and ladle treatment as well as during consequent metallurgical operations has been shown. Technological recommendations for improvement of steel making practice for elimination of defects in metal products are proposed.

Introduction

The problem of rejection sorting occurring in finished products is always the subject of serious disagreements between steel makers and rollermen who try to find reasons of defects and parties in fault. Very often the fact of exist of nonmetallic inclusions near defect is itself considered as a base for this defect determination as a steelmaking one. However, sufficient arguments are required in every such case to justify this opinion, to understand the nature of these inclusions and to develop recommendations for defect elimination. The method of metallurgical expertise can solve not only a.m. problems of reasoned flaw separation between metallurgical stages, but also can be the base for development of technological recommendations for improvement of manufacturing technology [1, 2].

Materials and methods

The samples of finished metal products as well as samples taken at different stages of the operating production of low-carbon deep drawing steels, including IF-steels, hot-rolled sheet for high-strength shipbuilding steels as well as electric steels have been used as investigated materials.

The sections have been prepared at sample preparing equipment made by Buehler Ltd. Metallographic and micro X-ray investigations have been conducted on flat, longitudinal and transversal sections prepared from defective metal samples. In order to reveal microstructure of examined samples, etching in 3 % spirit HNO_3 solution has been undertaken.

Metallographic investigations have been conducted at Nikon Epiphot microscope equipped with Thixomet image analyzer. This analyzer allows to make panoramic images having size as large as required; they are prepared via the method of "matching in fly" of adjacent fields of view. While sample stage moves to the neighbour field of view, the former field of view is matched in "pixel-to-pixel" mode to a field grabbed previously. Thereby we can form the panoramic image of the structure with arbitrarily large square and at the same time with high resolution. Panoramic investigations allow to examine complete square of a macro-defect with resolution sufficient for analysis of steel microstructure, including identification of nonmetallic inclusions which decorate a defect.

Micro X-ray analysis of nonmetallic inclusions has been conducted using REM AVT-55 microscope (AKASHI) equipped by Link AN 10000/85S (GB) microprobe; CamScan MV2300D SEM microscope has been used for evaluation of content of light elements, if required.

Thermodynamic simulation of the processes of nonmetallic inclusions formation in liquid and solidifying steel has been carried out using original software [3]. The results of thermodynamic calculations are presented as isothermal sections of equilibrium diagrams for multi-component multiphase systems constituting surface of component solubility in metal (SCSM), and also the results themselves of simulation processes of nonmetallic inclusions formation in liquid and solidifying steel, undertaken within the framework of local equilibrium thermodynamics [3, 4, 10]. In the last case the results represented composition and mass of nonmetallic inclusions formed in liquid and solidifying steel.

Results and discussion

Previously we have carried out system-based investigations of the defects of slabs, hot-rolled and cold-rolled sheet, and the results of these investigations made the base for valid separation of defects between steelmaking and mill production stages [1–2]. These results have been used not only for development of corresponding classifiers and atlases of defects, but they were also the base for improvement of steelmaking technology as well as for development of automatic systems for on-line monitoring of slab quality.

The present investigation is concerned in details with only those defects, whose steelmaking nature of forming has been proved with arguments, while their forming is connected, as a rule, with nonmetallic inclusions originated in liquid and solidifying steel.

Low-carbon deep-drawing steels

Let us consider the most common defect of cold-rolled sheet known as "skin". The inclusions accompanying the

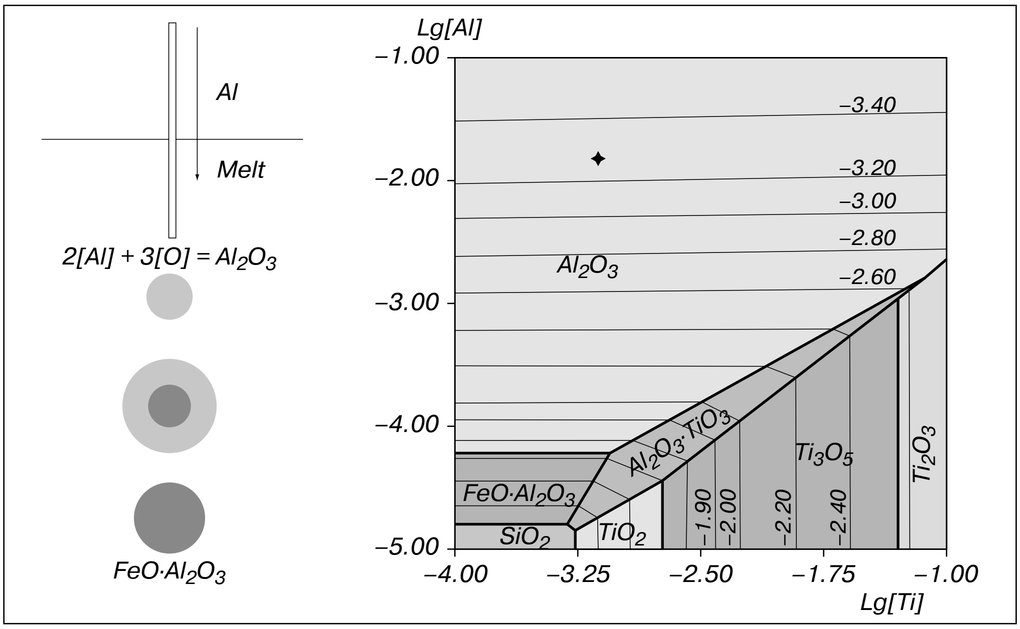

Fig. 1. Nonmetallic inclusions formation mechanism in IF-steel based on surface of component solubility in metal for Fe—0.01C—O—0.02Si—Ti—Al system at 1,600°C

"skin" defect can be separated on two kinds: small Al_2O_3—FeO oxides with size 3–10 microns (containing up to 3% of Fe) and more large oxides with size up to 25 microns (containing up to 20% of Fe). Both kinds of oxides revealed in the "skin" defect of cold-rolled sheet are products of deoxidation formed at steelmaking stage. It is confirmed by proximity of composition of these oxides and composition of nonmetallic inclusions found out in the samples taken consequently along whole technological route of steel production from ladle treatment and casting to hot and cold rolling of sheet.

The results of thermodynamic equilibrium calculations presented as the surface of component solubility in metal (SCSM) (Fig. 1) testify that figurative point of composition responding the real composition of deoxidated IF-steel is located in the area of Al_2O_3 existence, i. e. deoxidation products containing exclusively pure Al_2O_3 should form in liquid steel at aluminum concentration 0.01–0.04 %. However, in the conditions of real steelmaking it is impossible to introduce aluminum in non-deoxidated melt with its immediate and uniform distribution in total volume. As soon as aluminum is introduced in non-deoxidated melt in the form of pigs or wire, all possible combinations of aluminum and oxygen (i. e. those falling into the area of hercynite existence) appear in different local points of this melt (Fig. 1). Indeed, solid inclusions of FeO·Al_2O_3 spinel or hercynite are formed in the melt at some distance from aluminum solubility area, where small amount of aluminum and rather high over-oxidability of metal still remain. These inclusions forming in the conditions of local equilibrium in over-oxidated steel with aluminum "traces" as a result are getting into deoxidated metal where they become non-equilibrium. Then aluminum dissolved in deoxidated steel will reduce iron from hercynite, and this interaction will make easy high melt adhesion of mFeO·$n$$Al_2O_3$.

It is difficult to remove such inclusions from metal and, on the contrary, the inclusions formed in deoxidated metal and having alumina composition practically do not interact with the melt, are hardly wetted by metal and are easily removed during metal ladle treatment and casting. Due to low diffusion mobility of reagents, the speed of solid phase reactions inside inclusions is low, thereby degree of iron reduction from mFeO·$n$$Al_2O_3$ compound depends on size of initial hercynite inclusions for other equal conditions. So, the larger is size of initial hercynite inclusion, the smaller is speed of hercynite reduction to alumina and the most stable is it in the melt. Indeed, as it was mentioned above, small oxides contained about 3 % Fe, while iron content in large inclusions reached 20 %.

Thereby metal over-oxidability before aluminum insertion into the melt has been responsible for originating of mFeO·nAl_2O_3 inclusions with different compositions in investigated steels. In this connection, steel should be previously deoxidated (e. g. with silicon) before adding aluminum. It is necessary even for IF-steels having restricted silicon content because Si introduced for preliminary deoxidation has been used practically completely for elimination of metal over-oxidability.

Steel cleanness has been evaluated using Thixomet image analyzer in correspondence with ASTM E 1245. Content of nonmetallic inclusions in steel decreases substantially during ladle treatment and reaches 0.0006–0.0013 % (vol.) in the sample taken from the mold. Taking into account average steel content, we can calculate equilibrium (at 1,600 °C) content of oxygen dissolved in steel; it makes 2–6 ppm what correlates well with measurement data of steel oxidability obtained practically via EMF method (3–4 ppm). Using the methods of

Elements	Chemical composition in points (oxygen is balance), wt. %															
	57	58	59	60	61	62	63	64	65	66	67	71	72	73	74	75
Al	48	41	41	21	23	0.4	41	52	46	15	28	0.5	52	31	43	28
Fe	5	4	10	8	3	74	4	0.5	4	27	20	74	1.4	26	19	34
Ca	11	11	13	21	-	0.2	10	-	2.5	6.7	1.0	—	—	—	—	—

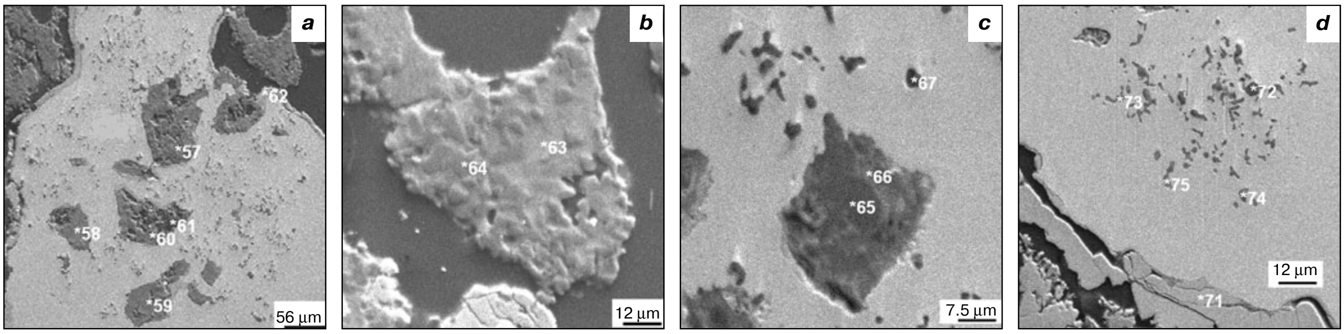

Fig. 2. Composition of nonmetallic inclusions in metallic beads

thermodynamic simulation of phase formation processes in liquid and solidifying melt [3], we can find volumetric content of secondary and ternary endogenous inclusions that will make 0.0006–0.0010 %; it also practically correlates with above mentioned experimental data obtained via the methods of quantitative metallography. We can make the conclusion about sufficiently complete removal of the main mass of primary nonmetallic inclusions using the existing technology of steel ladle treatment. It is confirmed also by composition of inclusions in metal samples at continuous casting, where only small inclusions with low content of iron oxides have been revealed.

In this investigation we did not reveal large primary inclusions with increased content of iron oxides in the samples before casting; however, they are presented in liquid metal because they have been found in pin point heterogeneity of a slab as well as in "skin" defect during consequent hot and cold rolling of continuously cast slab.

Additionally, large amounts of clusters of aluminum oxides with increased content of iron oxides have been found in metallic beads closed in rats on the surface of submerged nozzle of conticaster (Fig. 2). These rats consist of alumina inclusions assimilated by slag: up to 20 % of calcium oxides, up to 1 % of magnesium oxides and up to 27 % of iron oxides and the rest is aluminum oxides.

It should be mentioned that alumina inclusions absorbed by slag do not contain iron practically, while alumina-based inclusions in grasped metallic beads contain large amount of hercynite-based inclusions, in addition to isolated inclusions of practically pure alumina. It confirms once more a difficulty of removing hercynite-like inclusions from steel owing to their good wettability by steel. Metallic beads have been found enclosed in rather rough shell of iron oxides; it testifies about presence of secondary oxidation during casting.

Investigation of technological parameters of melts having "skin" defect on the surface of cold-rolled sheet has shown that these melts had amount of aluminum (introduced into liquid steel at different stages of steelmaking process) exceeded substantially the values noted in technological documentation. Possibility of "skin" defect formation increases with exceeding critical amount of aluminum introduced into steel. The more aluminum is introduced into liquid steel at all stages of steelmaking process, the more nonmetallic inclusions are revealed in the defect itself, and the more complicated is its morphology and appearance. Increased aluminum consumption at different stages of steelmaking process leads to increase of amount of deoxidation products, while these products provide clogging of steel pouring and submerged nozzles [5–7]. This fact is confirmed by increased values of aluminum oxidized during casting in the melts characterized by "skin" defect presence. In these melts concentration of aluminum been oxidized during casting reaches 0.051 %, while in the melts without "skin" this parameter does not exceed 0.005–0.010 %. To prevent increased aluminum oxidation during casting it is necessary to minimize its consumption and to improve the technology of protection of jet and metal mirror in steelmaking ladle and tundish as well as in mold.

High-strength shipbuilding steels

Below we shall show how improvement of deoxidation technology for sheet steels of shipbuilding purpose can provide high ductility of such steels in z-direction. We have examined steels processed by aluminum and silicocalcium according to different deoxidation schemes. Before this investigation, only partial cutting off furnace slag has been undertaken during steel tapping from the furnace, while aluminum has been added at once up to 3 kg/t, without preliminary deoxidation by ferrosilicon or ferromanganese. Final deoxidation by silicocalcium has been conducted by additives of this ferroalloy in barrels fixed on traveling bar. Such technology did not provide high metal quality, thereby up to 30 % of production volume has been rejected as defective via z-properties.

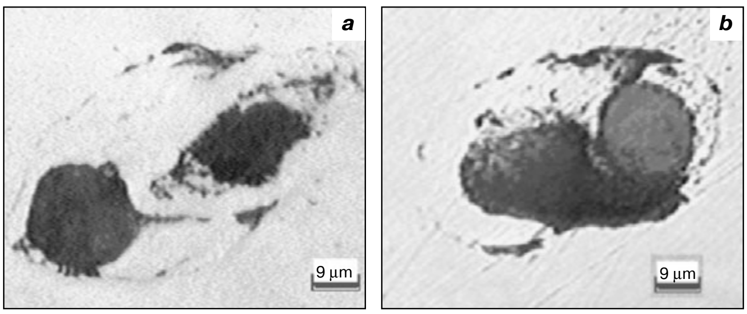

Fig. 3. The cavities around the nonmetallic inclusion after tensile test

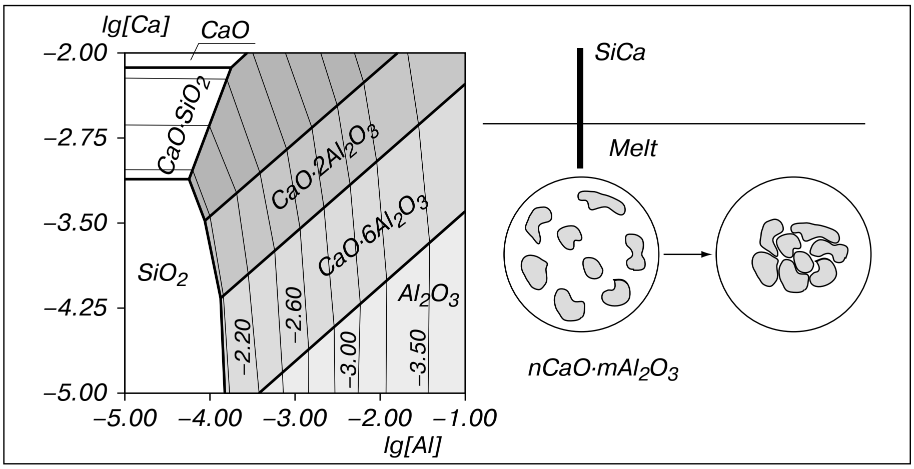

Fig. 4. Nonmetallic inclusions formation mechanism in high-strength shipbuilding steel based on surface of component solubility in metal for Fe—0.07C—O—0.25Si—Ca—Al system at 1,600 °C

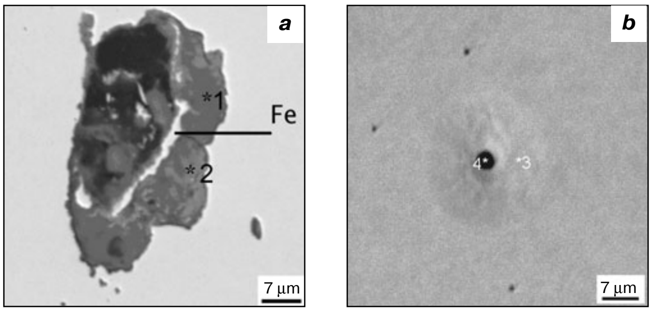

Fig. 5. Calcium hexa-aluminate (*a*) and manganese sulfide surrounded by sulfur segregation (*b*)

The results of investigations of steel samples molten via this technology have shown that clusters of large nonmetallic inclusions of different composition and morphology have been found out in the areas adjacent to destruction place. Size of these inclusions inside clusters reached 70 microns and volumetric content in destruction area — up to 1 %. These inclusions are distributed very irregularly as clusters, conglomerates and lines in the metallic matrix. Testing during deformation leads to forming of cavities (Fig. 3) that are source of micro-crack origination and cause of metal destruction. Micro X-ray analysis has shown that these inclusions consist of calcium aluminates of different composition, hercynite-based compounds, magnesial spinel and fayalite. The nature of inclusions forming of $FeO \cdot Al_2O_3$ system has been discussed above on the example of deep drawing steel. Let us consider consequently an origination of each of remaining kinds of inclusions.

Composition of calcium aluminate inclusions that are formed in this system can be determined according to the position of figurative point of steel composition on the corresponding SCSM (Fig. 4). It will be identified as calcium hexa- ($CaO \cdot 6Al_2O_3$) and bi- ($CaO \cdot 2Al_2O_3$) aluminates. These dark-grey crumbly round inclusions with size up to 70 microns have such features that every of them include internal metallic layers (Fig. 5, *a*).

The mechanism of such calcium-depleted and steel-impregnated porous deoxidation products forming consists in the following idea. Formation of calcium hexa- and bi- aluminates occurs in the interaction area between calcium and the melt. Clusters of inclusions coagulate and sinter in conglomerate; however, complete sintering is not reached due to high melting point of calcium-depleted aluminates, thereby steel melt remains between single particles. It is difficult to remove such inclusions from metal because they are kept in the melt owing to capillary forces. That is why it is required to exclude forming at least the most refractory calcium hexa-aluminates during deoxidation by silicocalcium. If calcium content is low, unfavorable inclusions of calcium hexa- and bi- aluminates are formed, and residual calcium concentration is insufficient for interaction with sulfur.

In inter-dendrite space of such metal we can observe wide areas of sulfur segregation with manganese sulfides in the center of such areas (Fig. 5, *b*).

As follows from simulation results for other equal conditions, the part of calcium hexa- and bi- aluminates decreases with elevation of calcium introduction in steel and the part of slag inclusions on the base of equimolar aluminates rises while residual calcium concentrations are combining sulfur in solid sulfides (Fig. 6). It is shown that steel deoxidation by 0.03 % Al is rather sufficient for active interaction between introducing calcium and sulfur. Consequent increase of concentration of introducing aluminum does not lead to efficiency rise of interaction between calcium and sulfur.

Presence of calcium-depleted aluminates, manganese sulfides as well as segregation areas around these inclusions in

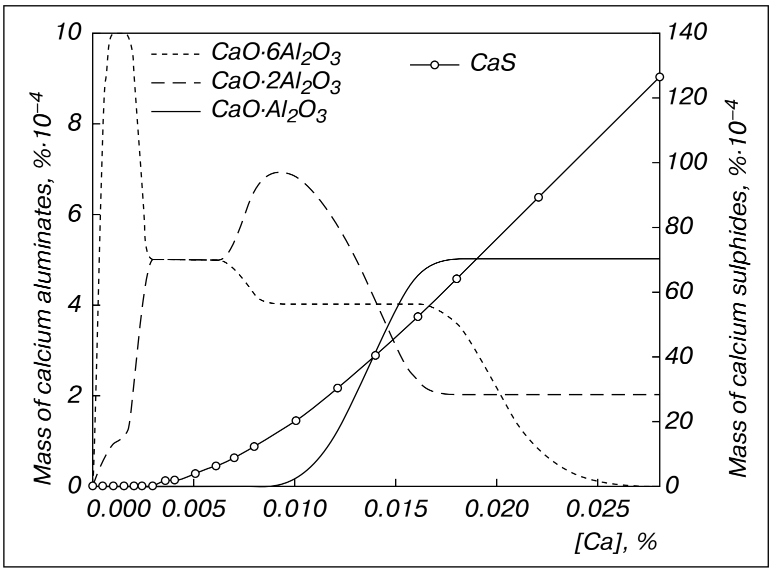

Fig. 6. Composition and mass of nonmetallic inclusions vs. calcium introduced in steel

steel testify that applying ladle treatment can not provide required calcium concentration in the solution; it leads to a forming of nonmetallic inclusions that are unfavorable in their morphology and it is difficult to remove them from steel [8–9].

Inclusions of $MgO \cdot Al_2O_3$ (magnesial spinel) have been revealed as large uniform clasters (Fig. 7, *a*) or small particles that are so-called bases or substrates for precipitation of calcium hexa- and bi-aluminates. Endogenous inclusions of magnesial spinel are formed (Fig. 7, *b*) due to alumothermal magnesium reduction from lining during steel tapping out of the furnace when steel jet contacts with aluminum located on the bottom of steel-pouring ladle or if aluminum is added into the melt just near the ladle lining [10].

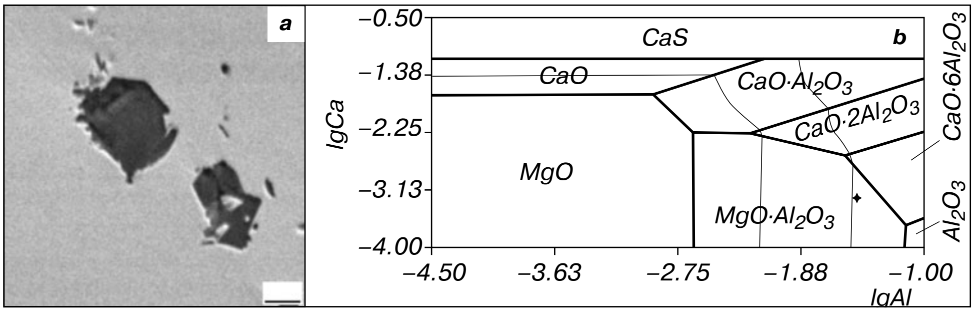

Fig. 7. Endogenous inclusions of magnesial spinel (*a*) and surface of component solubility in metal for Fe–0.07C–O–0.0005Mg–Ca–Al system at 1,550 °C (*b*)

Origination of magnesial spinel can have also exogenous nature, if the particles of magnesite refractories during steel tapping out of the furnace are broken away when contacting with liquid steel and are absorbed by the melt stream.

Revealed fayalite $FeO \cdot SiO_2$ inclusions have exogenous nature because they contain up to 50 % FeO, while steel deoxidation products with silicon contain not more than 10 % FeO, according to thermodynamic calculations.

The results of accomplished work for revealing the nature of inclusions became the base for improvement of steel ladle treatment technology; this improvement includes complete cutoff of furnace slag, preliminary steel deoxidation by ferrosilicon, lowering of aluminum consumption by 2 times and usage of tribe unit for aluminum and silicocalcium wire introduction. After such treatment, not more than 0.01% (vol.) of separate and uniformly distributed nonmetallic inclusions with size 3–30 microns have been revealed in the samples after their destruction; in this case cavities and cracks around inclusions were absent. The main mass of inclusions consists of equimolar calcium aluminates and calcium sulfides. Usage of the new technology allowed to decrease by 10 times rejects caused by properties in z direction.

Electric steels

Let us examine two kinds of defects mostly often met in these steels and called "through breakage".

Products of steel deoxidation and aluminum nitrides can be the cause of one kind of these defects forming. Clusters of nonmetallic inclusions such as $FeO \cdot Al_2O_3$, $3Al_2O_3 \cdot 2SiO_2$ and AlN have been revealed in the areas directly adjacent to the breakage place.

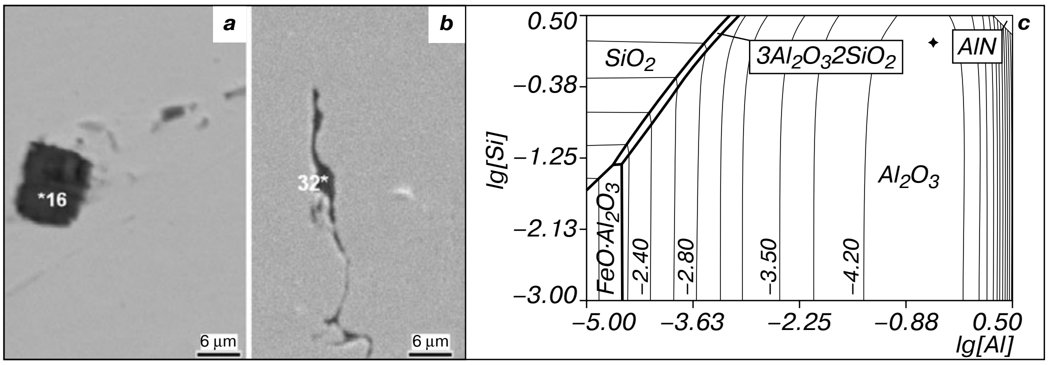

Fig. 8. Inclusions of AlN (*a*), $3Al_2O_3 \cdot 2SiO_2$ (*b*) in electric steel and surface of component solubility in metal for Fe–0.04C–0.25Mn–O–Si–Al system at 1,500 °C (*c*)

The nature of hercynite-based inclusions and the methods of prevention of such inclusions forming have already been examined before in this paper.

So we need to examine the nature and control methods for AlN and $3Al_2O_3 \cdot 2SiO_2$ inclusions (Fig. 8, *a, b*). The results of thermodynamic simulation testify that corresponding areas of these compounds exist on SCSM (Fig. 8, *c*).

If we take preset [N] content as much as 0.0057 % we shall find out that restriction of [Al] content less than 0.23 % can help to exclude forming AlN inclusions, while [Al] content more than 0.27 % will limit $3Al_2O_3 \cdot 2SiO_2$ forming. In other words, we need to have rather large amount of Al to minimize $3Al_2O_3 \cdot 2SiO_2$ forming, but not so large amount to provoke AlN forming in the end of solidification. At [N] > 0.0050 % it is impossible to find so-called "technological gates" of aluminum concentration that can exclude forming of both kinds of nonmetallic inclusions. Only at [N] = 0.0040 % such "technological gates" becomes rather wide ([Al] = 0.27–0.33 %) to be recommended for practical usage. The less nitrogen content in steel we shall have, the wider these "gates" will be meaning recommended aluminum content.

In order to exclude AlN forming, nitrogen can be removed out of solution using additives of more strong nitride-forming agent (titanium), but this recommendation is not valid for electric steels, because such modification will

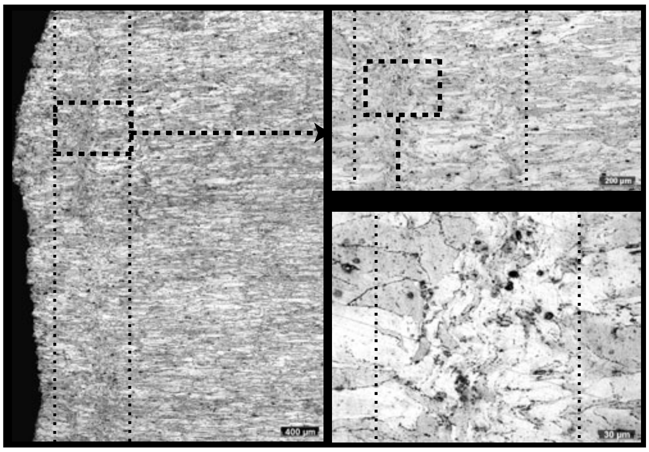

Fig. 9. Panoramic investigations of "through break" defect

lead to fine grain forming and steel magnetic properties losing.

Inclusions of aluminum nitrides (AlN) have been revealed in the melts with increased nitrogen content in finished steel and in the melts with increased aluminum content (when aluminum has been added during tapping and ladle treatment). It was established that high nitrogen content in steel is connected with increased nitrogen content in oxygen using for metal blowing in converter, and that size of inclusions becomes larger with decrease of slab solidification speed. This speed depends on steel temperature in the mold and casting speed.

Alongside with the above mentioned restrictions regarding "technological gates" of aluminum content, to prevent "through break" defect forming in the area of clusters of nonmetallic inclusions it is necessary to use purified oxygen for blowing in an oxygen converter, so that nitrogen content in finished metal should not exceed 0.0040%. Additionally it is necessary to exclude increased metal oxidability at converter tapping stage and to avoid chemical heating from the ladle treatment technology.

There is another kind of "through break" defect. Though nonmetallic inclusions being deoxidation products are also located in the neighbourhood of such defect, they were not the cause of break. Panoramic investigations of the flat thin section testified that deformation traces are completely absent in the sections directly adjacent to the place of metal break and along micro-cracks accompanying this break. We can observe here absolutely brittle destruction without any visible plastic deformation. On the contrary, the rest metal between breaks and cracks is characterized by large-size deformed grain (Fig. 9).

Enormous number of copper-based dispersed precipitations has been revealed along breaks and cracks. The same areas contain such strongly liquating elements as sulfur and phosphorus. Dispersed precipitations in the sheet defect areas testify that precipitation hardening of steel occurred in this case. It is accompanied with increase of yield strength and decrease of metal ductile parameters in local areas.

Increased concentration of copper, sulfur and phosphorus is a sequence of a string zonal segregation. The mechanism of such strings formation is the following. In the case of stopping of equipment operation, non-uniform slab cooling or its slow solidification, so-called "bridges" can be formed. These "bridges" block free access of liquid metal to underlying volumes of solidifying steel to compensate its shrinkage. Just before complete solidification of these underlying volumes, all impurities collected in the upper part of solidifying ingot due to zonal segregation are sucking into the narrow channels of interdendritic space. Thereby high concentration of phosphorus, sulfur and copper are localized in this interdendritic area, and their local concentration values exceed average concentration in steel by dozens of times.

The results of thermodynamic calculations for copper concentration values (0.07–3.5 %) determined in the strings of zonal segregation testify that temperature range of precipitation hardening makes 250...500 ^oC — 250...920 ^oC respectively. In this temperature range copper is extracted as dispersed particles, causing the effect of precipitation hardening.

Analysis of technological data of melting, ladle treatment and casting of the melts which samples have been affected with this defect has shown that casting of these melts is connected with stopping the equipment, non-uniform speed or low speed as well as with rather high metal overheating above liquidus temperature. All above mentioned factors strengthen the processes of isolation and segregation of impurities, and it leads to zonal chemical heterogeneity forming according to the mechanism of string forming described above.

To minimize or totally prevent appearance of "through break" defect in the areas of zonal segregation, it is necessary not to allow high values of metal overheating during casting, to provide uniform casting speed without stopping the equipment and to lower copper content in the scrap.

Conclusions

Thereby, the technique of metallurgical expertise of the defects in metal products developed in the presented work allowed not only to determine the nature of these defects forming, but also made the base for improvement of manufacturing technologies. This information can be used for development of the systems for monitoring and control of metal product quality at all technological stages.

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Application of the gas-thermal coating for increase of service properties of the metallurgical equipment and metal products

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The material science of monometals in the development has come nearer to achievement of the certain natural limits, the basic resources are already developed, and we can’t expect the numerous innovative breaks in this area. The further progress in the theory and technologies of metal materials will be connected, first of all, with application of composites, including — layered ones.

In layered composite materials we can observe possible overlapping of various physical, chemical and mechanical properties, including such properties whose combination basically can’t be realized in traditional homogeneous materials. The reserves incorporated in the concept of association of various materials in a uniform composition, are huge and, in a sense, only start to be revealed. One of the basic directions of realization of the given concept is creation of materials with the modified superficial layer. In most cases destruction of a material begins from a surface: both superficial destruction (deterioration) and volumetric destruction (formation of cracks). Thus, at the decision of a problem of increase of reliability of a material, it is necessary to strengthen a superficial layer first of all. The surface of a detail is a critical part in system “detail – environment” (corrosion destruction, including high-temperature destruction), therefore updating of a superficial layer becomes the most effective approach to the decision of one of the most important applied problems of material science — maintenance of compatibility of a material with a working environment.

The technology gas-thermal sputtering is still one of most technically and economically accessible methods of updating of superficial layers of metal products and billets [1]. Various kinds of processing of billets with gas-thermal coatings (GTC) — thermal, plastic, mechanical and so forth, — allow to improve essentially characteristics of a coating or even to lead to creation of essentially new composition. In the Moscow institute of steel and alloys the researches on influence of various factors on physicomechanical and operational properties of coatings have been carried out, and large scientific and practical experience on drawing and processing of coatings has been accumulated for solving of various applied problems. It is shown [2], that such deficiencies of coatings as high porosity, low adhesion and durability of coupling between particles of a coating can be eliminated by heat treatment and metal forming.

The technology to control the service characteristics of a coating has been developed. The various combination of heta treatment procedures (temperature, holding time, atmosphere) and plastic deformation (a rolling, rotblasting, processing by brushes, etc.) of coatings has been considered. The relationships connecting procedures of examined processes with physical and mechanical properties of coatings and operational properties of products with coatings are received. The wide range of materials for coatings (Al, Zn, Ni, Ni-Cr, different steel grades, etc.), applied on steel and copper substrate has been investigated.

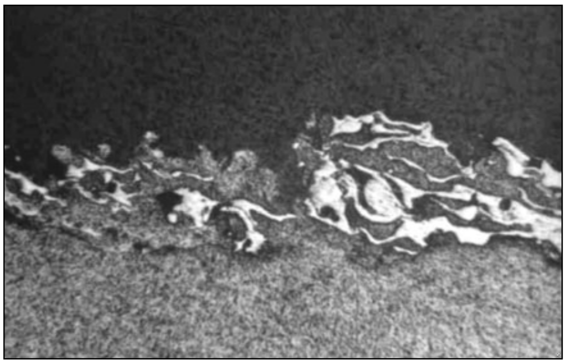

Fig. 1. Nickel aluminide layer on the surface of aluminium billet