OXIDATION RE-MELTING OF THE CR13NI1 CAST STEEL BY MEANS OF OXYGEN-BLOWING

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OXIDAČNÍ PŘETAVOVÁNÍ CR13NI1 OCELI NA ODLITKY S DMÝCHÁNÍM KYSLÍKU

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Abstrakt

Vysokolegované korozivzdorné 13% chromové oceli s přísadou 1% niklu, odolné vůči kavitaci, představují dlouhodobě provozně ověřený materiál, který má trvalé místo v konstrukcích čerpadel, kompresorů a vodních turbín Kaplanových, Francisových, Peltonových, a v konstrukcích hydraulických systémů. K stálému používání ocelí Cr13Ni1 patří jejich dobré slévárenské vlastnosti, nižší cena a pro menší vodní díla také vyhovující mechanické, korozní a kavitační charakteristiky. Trvalý zájem o použití oceli Cr13Ni1 a snaha vyrábět z ní odlitky přetavováním vratného materiálu, s dobrými mechanickými vlastnostmi a bez slévárenských vad, především bez bublin, kterými jsou přetavby nejčastěji provázeny, vedly k proměření aktivit kyslíku v charakteristických fázích oxidačních přetaveb těchto ocelí. Proměření aktivit kyslíku proběhlo ve vztahu ke změnám ve složení lázně a k dosahovaným mechanickým vlastnostem. Základním cílem příspěvku je informovat o výsledcích provozního výzkumu oxidačních přetaveb oceli Cr13Ni1 s naznačením možností jak technologii přetaveb účelně optimalizovat.

Abstract

Corrosion-resistant 13% Cr and 1% Ni cast steels are used to produce castings for the Kaplan, Pelton and Francis turbines. These cast steels also have a satisfactory cavitation resistance. The oxidation re-melting process, where oxygen is blown into the bath, was elaborated with the aim of reducing the gas content (hydrogen and nitrogen) in re-melted Cr13Ni1 steel scrap. This technology was tested on and described for twenty-five melts, with special regard to oxygen-blowing conditions, mechanical properties (verified by the impact test) of the cast steel, and suppressing the formation of gas holes in the castings. A basic electric-arc furnace was used in the oxidation re-melting process of steel scrap. The measuring of bath temperature and oxygen activity in the bath, and the parallel sampling for chemical analysis of the bath were realized in the following melt periods: (1) after melt-down and heating before oxygen blowing; (2) immediately after the end of oxygen blowing; (3) at the start of the reduction period, after preliminary deoxidation with Al, slag reduction and modification of chemical composition; (4) in the bath at the end of the reduction period; and (5) in the ladle,

after tapping and after SiCa deoxidation. Results of this measuring are given in Table I and the corresponding mechanical properties of and impact test results for re-melted steels are in Table II. The heat treatment of cast samples of re-melted corrosion-resistant steels was as follows: homogenisation annealing at 1150°C / (6÷20 h – as a function of the wall thickness of castings) / (air cooling) plus hardening at 940°C / (4÷6 h) / (air cooling) and tempering at 720°C / (4÷6 h) / (air cooling). The castings from re-melted steel of this type were without bubbles.

Key words: corrosion-resistant steel, castings, water turbines, oxidation re-melting, oxygen-blowing condition, oxygen activity, mechanical properties, re-melting technology optimizing

1. Introduction

High-alloy corrosion-resistant 13% Cr and 1%Ni cast steels, which are also resistant to cavitation, represent a material of long-tested operation. It has come to occupy a permanent place in the design of pumps, blowers and water turbines (of the Kaplan, Francis and Pellton types) as well as in the design of hydraulic systems. These Cr13Ni1 cast steels are used in the production of parts of medium or larger mass, of the order of so-called small units of tonnes (< 5 t) in spite of the fact that Cr13Ni4 to Cr13Ni6 cast steels with molybdenum addition have been developed, which exhibit better mechanical properties and better weldability [1 to 7].

The on-going application of the Cr13Ni1 cast steels is related to their better foundry-engineering properties, lower cost and, in case of smaller hydroelectric stations, their satisfactory mechanical, anti-corrosion and cavitation characteristics, even in cases when dynamically highly stressed castings are concerned [8].

The constant interest in the application of the Cr13Ni1 cast steel, efforts to make from it castings via re-melting the scrap, its good mechanical properties and the absence of foundry defects (in particular bubbles, which very frequently accompany re-melting) have led to the measurement of oxygen activities in the characteristic phases of oxidation re-melting of the above type of cast steel. The activities were measured in relation to the changes in bath composition and the mechanical properties achieved.

The basic aim of the paper is to provide information about the results of field research into the re-melting of Cr13Ni cast steel and to suggest some possibilities of meaningfully optimizing the technology of re-melting.

2. Melting and measuring

The measuring was performed on regular melts from electric-arc basic furnaces of a medium charge mass of 6.9 ± 0.5 t, with the melt being oxidized with gaseous oxygen. A total of 25 melts were chosen for a detailed examination of oxidation re-melting.

The primary charge consisted of ca 85 wt.% (only % in the following) of high-alloy chromium cast steel scrap of the type of Cr13Ni1, the rest was unalloyed steel scrap with a low content of phosphorus and sulphur. The charge composition was chosen such that after melting the chromium content in the melt was about 11.5 % and the nickel content about 1% and that of carbon about 0.35% (these are weight percentages). Pulverized coke and electrode fragments were used to carburize the charge.

After melting the charge the bath was heated to an average temperature of 1614 ± 60°C, after which oxygen began to be blown. Steel tubes of ¾" (some 19.1 mm) in diameter,

protected at the end with fireclay tubes, were used. Oxygen was blown into the bath under a pressure of ca 1 MPa for a period of 7 to 10 minutes, during which the carbon content in the bath dropped below 9.1%. Under these conditions the temperature of the bath increased to 1780 ± 39°C, when oxygen ceased to be blown.

When oxygen blowing was over, a reduction mixture of silicochromium (SiCr) – (4.0 ± 1.7 kg/t), pulverized ferrosilicon (FeSi) – (2.5 ± 1.6 kg/t), silicocalcium (SiCa) – (1.5 kg/t) and aluminium chips (Al) – (1.1 ± 0.2 kg/t) was added into the bath. The reduction mixture was left to work for about 10 minutes, after which the bath was stirred and the slag was skimmed and a new one was prepared from lime and fluorite. At the same time, preliminary deoxidation with aluminium forged onto a rod took place – Al – (0.6 kg/t).

The chemical composition of the bath during the reduction period was modified as needed, using silicochromium (SiCr) – (1.4 ± 0.7 kg/t), ferrosilicon (FeSi) – (1.2 ± 0.4 kg/t), low-carbon ferrochromium (FeCr) – (46.2 ± 16.4 kg/t), silicomanganese (SiMn) – (1.9 kg/t), ferromanganese (FeMn) – (3.4 ± 0.9 kg/t), and pure nickel (Ni) 0 (0.5 kg/t). Final deoxidation with silicocalcium (SiCa) – (2 kg/t) took place in the ladle at a temperature of 1619 ± 24°C.

Oxygen activity in the bath was measured with combined TSO-3 probes, which enable simultaneous measurement of both the bath temperature and the oxygen activity. The chemical composition of the bath was established with the aid of emission spectral analysis. The measurements were realized during these characteristic phases of melting:

(1) – subsequent to the charge being melted, prior to blowing oxygen into the bath,
(2) – after the end of oxygen blowing into the bath, before skimming the oxidation slag,
(3) – at the beginning of reduction period after preliminary deoxidation with aluminium and after the formation of reduction slag,
(4) – in the furnace towards the end of reduction period and before tapping,
(5) – in the ladle subsequent to tapping and precipitation deoxidation with silicocalcium.

Mechanical properties, specifically yield strength (R_e), tensile strength (R_m), ductility (A_5), thinning (Z) and notch impact strength (KCU 3) at a temperature of 20°C were established on test rods prepared from separately cast bodies of $60x60x200$ mm^3, which had been heat treated together with castings in annealing foundry furnaces. Heat treatment consisted of homogenization at 1150°C / (6 to 20 hours, depending on the weight and wall thickness of the casting) / air, then quenching at 940° (4 to 6 hours) / air (cast steel Cr13 Ni can be quenched during cooling in air) and tempering at 720°C / (4 to 6 hours) / air. The sequence of individual phases of melting, mean values and standard deviations of individual measured and examined quantities are given in Table I.

3. Results of measuring and their evaluation

Melting and oxygen blowing: Melt interval $\Delta t_{1,2} = (1)-(2)$

The average value of decarburization speed follows from the difference in the carbon content at the end of oxidation (2), i.e. towards the end of oxygen blowing, and after melting (1), i.e. before oxygen blowing. It follows from the data in Table I that on the average it is 0.613 % C/h, which is a sufficient guarantee of the re-melting bath being degassed adequately, i.e. the content of hydrogen and nitrogen in the bath being reduced. During oxygen blowing the speed of decarburization will in fact reach its maximum, the value of which can be estimated with the aid of the mean blowing time, which is 8.5 min. According to the data in Table I the maximum speed of decarburization is ca 2.0 % C/h. These parameters simultaneously characterized the

castings produced from this series of 25 melts, which did not contain any foundry defects of the type of cavity, i.e. bubbles. Passing into the slag in this period are manganese, silicon, phosphorus and chromium, whose concentration in the bath drops by 2.62% Cr to a concentration of 8.68 % Cr, which is about 77% of its content in the bath after melt-down. The concentration of nickel in the bath is slightly increased.

Table I Arithmetic means and standard deviations of metallurgical data in individual melting phases (25 industrial melts) chemical composition is in [wt.%]

Phase i	Δt_i [min]	$\Sigma \Delta t_i$ [min]	T [°C]	$a_{[O]}$ [ppm]	C	Mn	Si	P	S	Cr	Ni	Al_c
(1)	30.2	30.2	1614	133	0.36	0.39	0.11	0.022	0.021	11.30	0.90	-
	20.6	20.6	60	93	0.10	0.06	0.07	0.005	0.007	1.16	0.13	
(2)	27.4	57.6	1780	837	0.08	0.27	0.02	0.019	0.021	8.68	0.90	-
	11.3	27.2	39	387	0.03	0.06	0.02	0.005	0.007	0.97	0.18	
(3)	39.1	96.7	1740	325	0.09	0.54	0.18	0.022	0.021	10.21	0.90	0.0117
	10.7	27.3	33	116	0.02	0.15	0.11	0.005	0.005	1.25	0.13	0.0208
(4)	24.5	121.2	1660	110	0.09	0.60	0.28	0.028	0.026	13.34	0.94	0.0082
	12.7	33.2	32	43	0.02	0.12	0.18	0.004	0.004	0.66	0.08	0.0241
(5)	23.7	144.9	1619	58	0.10	0.70	0.30	0.025	0.020	12.87	0.91	0.0019
	12.3	31.2	24	27	0.02	0.09	0.13	0.004	0.005	0.54	0.11	0.0018

Note: the first line gives the arithmetic mean, the second line the standard deviation

The changes are further accompanied by a marked increase in the temperature and activity of oxygen in the bath, namely from an average value of 133 ppm of oxygen to 837 ppm, which is an approximately six-fold increase.

Slag replacement and beginning of reduction period: Melt interval $\Delta t_{2,3} = (2) – (3)$

After the reduction of chromium from the slag by means of a reduction mixture formed by SiCr, FeSi, SiCa and Al, and subsequent partial skimming of the oxidation slag, after the formation of new slag from lime and fluorite, and after preliminary deoxidation of the bath with aluminium the activity of oxygen in the bath is markedly reduced (about 2.6 times). The chromium content in the bath increases to an average value of 10.21% Cr, which is about 90% of the chromium content after the charge melt-down. The permanent loss of chromium that accompanies oxygen blowing into the bath thus amounts to 1.09% Cr on the average.

In the bath the development of slag reduction mode is coupled with increased concentrations of carbon, manganese, silicon, phosphorus and nickel, and the bath temperature decreases. The total aluminium content in the samples taken from the bath has no standard statistical distribution, it is accompanied by a large scatter in the values of Al-concentration, and its melting loss makes 81% of the added amount of 0.6 kg/t.

Reduction phase: Melt interval $\Delta t_{3,4} = (3)-(4)$

The chemical composition of the bath was modified by adding ferro-alloys and a small amount of pure nickel. Diffusion deoxidation process had a significant role in that the oxygen activity dropped to 110 ppm (i.e. to ca one third of the value at the beginning of this period). Manganese, silicon and phosphorus contents increased (in the case of phosphorus via its return from the slag) and there was also a slight increase in the content of sulphur, with the bath temperature decreasing markedly (by 80°C). The melt loss of the aluminium added for preliminary deoxidation in the amount of 0.6 kg/t is 86% at the end of the reduction phase in the bath prior to tapping, it has no standard statistical distribution and is characterized by a large scatter of values.

End of the reduction period and melt tapping into ladle: Melt interval $\Delta t_{4,5} = (4)-(5)$

Following the furnace tapping and the precipitation deoxidation in the ladle (adding 2 kg SiCa/t into the ladle) oxygen activity in the bath decreases to ca 53% of the value from the end of reduction period in the furnace prior to tapping. Both tapping and precipitation deoxidation with SiCa are accompanied by increased aluminium melt loss which, in relation to its amount of 0.6kg/t added into the furnace for preliminary deoxidation amounts to 96.8 ± 3.0%. The total aluminium content Al_c in finished cast steel is very low, a mere 0.0019%. This shows that cast steels produced by this technology will be prone to the secondary austenitic grain getting coarser during holding time at quenching, normalization and, above all, homogenization temperatures [9].

Mechanical properties

These properties were established for finished cast steel and they are given in Table II. From the values given in the Table it follows that in oxidation re-melting the ratio of tensile strength to yield strength achieved is 0.68, with a ductility of 22% and excellent contraction of 62%. Also, notch impact strength KCU 3 of this cast steel, established on separately cast bodies processed together with castings in foundry annealing furnaces, is adequately high and sufficiently resistant to brittle fracture. If we consider that the critical value of notch impact strength, which signals the danger of brittle fracture, is about 25 Jcm^{-2}, then the lower value of the interval < x ± 2s > has for the set of test specimens according to the data in Table II a value of 26 Jcm^{-2}. It is known that 95 % of all values of the respective quantity are to be found in the interval of normal statistical distribution < x ± 2s > [10]. It can thus be stated that the above operation-tested technology of oxidation re-melting with oxygen blowing ensures, if properly adhered to, a statistically significant reserve in toughness against brittle damage, good strength and plastic properties. It also provides an adequate guarantee that castings will be free of bubbles, which frequently accompany the re-melting of these cast steels without oxygen blowing.

Table II Mechanical properties of Cr13Ni1 cast steel (25 industrial melts)

Property	Yield strength R_e [MPa]	Tensile strength R_m [MPa]	Ductility A_5 [%]	Contraction Z [%]	KCU3 20 ºC [J/cm²]
arithmetic mean x	459.1	671.5	22.24	62.16	79.71
statistical deviation s_x	45.8	48.8	4.01	4.27	27.01

4. Technological implications

The choice of the conditions for blowing oxygen into baths of 13%Cr cast steels with 1%Ni can greatly affect the economy of oxidation re-melting since this process is characterized by a high melt loss of chromium due to its passage into the slag. After the end of oxygen blowing not all the chromium can be reduced back from the slag and chromium is then also skimmed as part of the oxidation slag. It is therefore necessary that blowing oxygen into the bath be accompanied by as small a melt loss of chromium as possible. The relations between the concentrations of chromium and carbon and the bath temperature prior to the beginning of blowing that meet the above requirement and which served as approximate values for the choice of the conditions for oxygen blowing in the case of the melts under examination are given in Table III (see also fig.1). The relations mentioned above were calculated as given in [2].

Table III Relation between chromium and carbon contents in the bath in [wt.%] and bath temperature when blowing oxygen at a pressure $p(O_2) = 0.1$ MPa and a concentration of 1% Ni

Content of element	Bath temperature [°C]				
	1600	1650	1700	1750	1800
Cr [wt.%]	C [wt.%]	C [wt.%]	C [wt.%]	C [wt.%]	C [wt.%]
6.0	0.223	0.158	0.113	0.078	0.058
7.0	0.263	0.183	0.133	0.093	0.071
8.0	0.303	0.213	0.153	0.108	0.078
9.0	0.343	0.243	0.173	0.123	0.093
10.0	0.383	0.273	0.198	0.143	0.103
11.0	0.428	0.308	0.223	0.158	0.118
12.0.	0.473	0.338	0.248	0.178	0.133
13.0	0.518	0.378	0.273	0.198	0.148
14.0	0.563	0.413	0.303	0.223	0.163

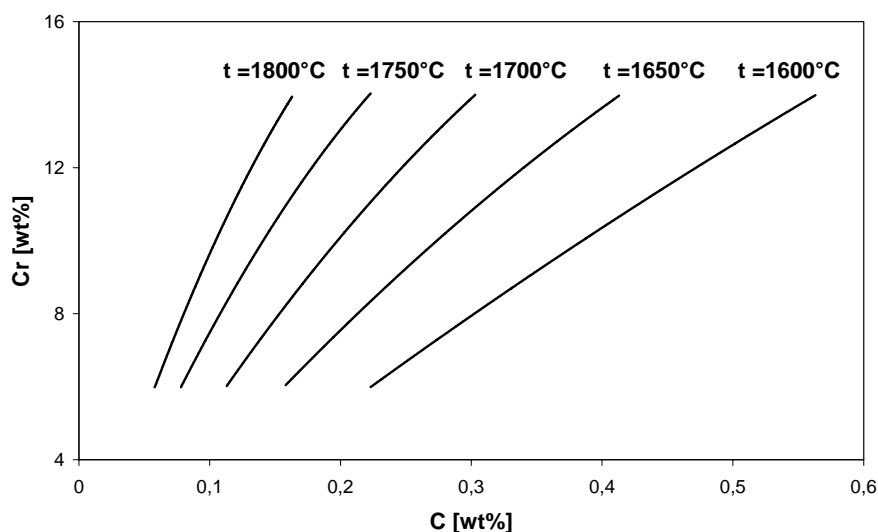Note: data in the Table have been calculated as reported in [2]

Fig.1 Relation between chromium and carbon contents according to Table III

Mathematical-statistical processing of the data measured in phase (2), i.e. paired values of carbon and chromium concentrations in the bath subsequent to the completion of oxygen blowing by the least squares method yielded the equation

$$[\%C] = 0.00293[\%Cr]^{1.1513} \qquad (1)$$

with the correlation coefficient $r = 0.4561$, which for 23 degrees of freedom of the set under examination is significant on a level of statistical significance better than 0.05 [10].

Equation (1) can retrospectively be used to assess the extent to which the theoretically calculated relations between Cr and C concentrations and temperature T after oxygen blowing given in Table III are reflected in actual relations. For example, for an average (post oxygen-blowing) temperature of 1780°C and a chromium concentration of 8.68 % (Table I) we obtain by

equation (1) a corresponding carbon concentration of 0.0771% C, the upper limit being 0.1295% C and the lower 0.0460% C. By interpolating the values in Table III for a chromium concentration of 8.68% Cr and a temperature of 1780°C we obtain a carbon concentration of 0.1002%. It is evident that this value of carbon concentration is in the interval of experimentally established concentrations of the same element in the bath for oxygen blowing, which is < 0.0460; 0.1295>, and is close to the experimentally established value of carbon concentration 0.0771.

For the mean bath temperature at the beginning of oxygen blowing 1614°C and the initial chromium concentration 11.30 % the corresponding equilibrium concentration of carbon in the bath should by equation (1) be 0.1149%, but the actual average carbon concentration in the bath is 0.36 % (melt phase (1) – Table I). In the set of melts under examination the blowing of oxygen into the bath begins at an average value of the excess in carbon concentration in the bath: 0.36 – 0.11 = 0.25%C above its equilibrium concentration, which is also a guarantee of faster oxidation of carbon than chromium.

However, it is necessary to take notice of the behaviour of phosphorus in the bath in the individual melting phases. It can be seen from Table I that in phase (2), i.e. after the completion of blowing oxygen into the bath, before the reduction of chromium from slag and before skimming the slag, the content of phosphorus in the bath is on the average lower than its concentration in the bath after melt down, i.e. in phase (1). The reduction in phosphorus concentration during oxygen blowing (that is when the activity of oxygen in the bath increases) makes on the average ca 14% of its mean concentration in the bath after the beginning of melting. At the beginning of the reduction period (i.e. in phase (3), subsequent to the reduction of chromium from slag and the following skimming of this oxidation slag, after preliminary deoxidation with aluminium and after the reduction slag has been formed), when the activity of oxygen decreases, we can witness an increase in the concentration of phosphorus in the bath, practically to the value of phosphorus concentration in the bath after the melting of the main charge. In the remaining melt phase (4) and following the tapping (5) the content of phosphorus in the cast steel continues increasing, in proportion to the decrease in the activity of oxygen in the bath. The relation between oxygen activity and phosphorus concentration in the bath can be expressed for the individual melt phases (1) to (5) using the data in Table I by the power function

$$[\%P] = 0.0395 \left(a_{[O]ppm} \right)^{-0.107}, \tag{2}$$

with the correlation coefficient r = 0.8903, which is statistically significant on a significance level better than 0.05.

It must therefore be borne in mind that when the above technology of oxidation re-melting of Cr13Ni1 cast steel is used, the bath cannot be dephosphorized. On the contrary, there is a tendency towards gradually increasing phosphorus concentration in finished cast steel.

If the above technology is maintained, the activity of oxygen in the bath is controlled in all the melt phases, i.e. in phases (1) to (5), by a parameter formed from the product of the concentrations of carbon, silicon, manganese and chromium in the bath. For the arithmetic means of oxygen activities and concentrations of C, Si, Mn and Cr in Table I from 25 melts of this cast steel the following equation holds with much probability:

$$a_{[O]} = 12.28 \left([\%C][\%Si][\%Mn][\%Cr] \right)^{-1.318}, \tag{3}$$

with the correlation coefficient r = 0.9950, which, even if we assign to the data sets in Table I merely three degrees of freedom $\nu = n - 2$, n = 5, is significant on a statistical significance level α better than 0.001 (the critical value of correlation coefficient for $\alpha = 0.001$ and $\nu = 3$ is r = 0.9912). The thermodynamic justification of the preceding equation can be given using parallel-running re-oxidation reactions of the above elements, i.e. C, Si, Mn and Cr in the bath.

The conditions for oxygen blowing into the re-melting bath of Cr13Ni1 cast steels, which is the relations between the concentrations of carbon and chromium and the bath temperature established according to Table III for a pressure of blown oxygen of 1 MPa can be regarded as appropriate to ensure the least chromium melt loss possible.

5. Conclusion

The paper comprises the results of measuring and monitoring the process of 25 oxidation re-melts of 13% chromium cast steels with 1% nickel and the mechanical properties of finished cast steel for the production of water turbine castings. Melting took place in a 7-tonne electric basic arc furnace, and the following knowledge was obtained:

For the above conditions of melting and oxygen blowing at a pressure of ca 1 MPa into the bath it is advisable to use a bath temperature of 1620°C at the beginning of oxygen blowing, and to choose the relation between the chromium and the carbon concentration in the bath according to the relation:

$$[\%C] > 0.00293[\%Cr]^{1.51} + 0.30 \qquad\qquad (4)$$

where [%C] and [%Cr] are the carbon and the chromium concentrations (in wt.%) in the bath. After the beginning of melting the chromium concentration in the bath should be approximately in the middle of the interval 9.5 to 13.0 % Cr.

Blowing gaseous oxygen into the bath under these conditions is coupled with an increase in the bath temperature by about 170°C, a loss of about 1.1% chromium in the bath due to its subsequent removal via the oxidation slag, and a 0.28% decrease in carbon content.

The reduction period is characterized by a relatively high thermodynamic oxygen activity in the bath, which is markedly reduced only by the deoxidation of SiCa in the ladle after furnace tapping. In other cases the basic choice of blowing conditions can be recommended, i.e. carbon and chromium concentrations and bath temperature in accordance with Table III.

Strength and plastic properties of finished, homogenization-annealed and alloyed cast steel are in the case of the technology described above satisfactory. The notch impact strength of individual melts gives a good guarantee against brittle fracture of this cast steel after its final heat treatment. Blowing gaseous oxygen reduces to a minimum the tendency towards bubble formation in the castings.

Acknowledgement

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