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Precipitation kinetics in austenitic 18Cr-30Ni-Nb cast steel

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Abstract

The study presents the results of investigations on the precipitation kinetics in austenitic 18%Cr-30%Ni cast steel stabilised with an addition of 1.84 wt% niobium. Phase analysis of isolates extracted from the alloy subjected to annealing within the temperature range of 600–1000°C during 10–1000 h was made. The phase constitution of the isolates mainly comprised niobium carbides of the NbC type and complex chromium carbides of the $Cr_{23}C_6$ type. In specimens annealed within the temperature range of 700–900°C, a high-silicon G phase was additionally identified. The highest kinetics of the precipitation process was recorded after annealing at the temperatures of 800 and 900°C.

Keywords: Ni-Cr cast steel, Precipitation, Niobium carbide, G phase

1. Introduction

Niobium is introduced to austenitic cast steel assigned for operation at high temperatures mainly to improve its creep resistance.

The presence of niobium in cast steel also reduces the carburising effect in this alloy when assigned for operation at the carburising-oxidising atmospheres [1]. The change in the cast steel structure caused by partial or total replacement of the complex chromium carbides of the $M_{23}C_6$ type with simple MC type carbides may be but does not necessarily have to be favourable for other mechanical properties [2-4]. The changes in the properties are the consequence of changes not only in the type of the carbides but also in their morphology. Moreover, during annealing, the NbC carbides undergo transformation into a high-silicon G phase. Investigating the kinetics of this transformation in stabilised austenitic steel, Powell has proved that its rate reaches the highest level within the temperature range of 750-800°C [5]. It is accompanied by the precipitation of chromium carbides of the $M_{23}C_6$ type, due to carbon being liberated from the NbC carbides. On the other hand, Soares et al. [6,7] have observed that in the case of centrifugally cast HP steel with an addition of 1.97wt% Nb, the NbC carbides are the least stable at the temperature of 900°C.

The aim of the present study has been an assessment of the kinetics of the precipitation process taking place in 18%Cr-30%Ni cast steel with an addition of 1.84 wt% niobium during its annealing.

2. Experimental

The chemical composition of the examined cast steel is given in Table 1. Melting was carried out in an induction furnace with acid lining. The liquid metal was gravity poured into open moulds made from a chemo-setting sand mixture [8].

Table 1.
Alloy composition (wt%)

C	Si	Mn	Cu	Al	P	S	Cr	Ni	Nb	Ti
0.30	1.82	0.96	0.22	0.02	0.015	0,01	18.2	29.3	1.84	0.06

The cast steel specimens cut out from the test ingots were subjected to annealing in the atmosphere of air, successively at the following temperatures: 600, 700, 800, 900 and 1000°C. The time of annealing was 10, 100, 500 and 1000 hours. The specimens were next cooled in air and machined to the dimensions of Ø10×30 mm.

For determination of the phase composition, isolates extracted from the specimens before and after annealing were prepared using an electrolyte of the following composition: 5 g $C_2H_2O_4$, 200 cm^3 HCl, 1000 cm^3 H_2O [9]. A schematic representation of the stand for electrolytic extraction is shown in Figure 1. Table 2 shows fractions of the phase constituents isolated from an austenitic matrix after conversion into weight percent values.

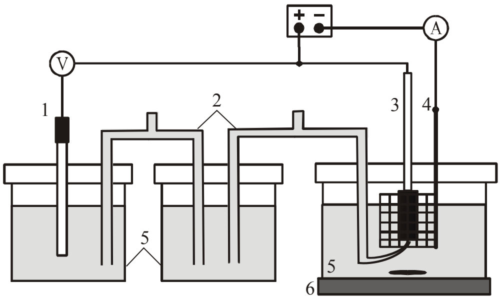

Fig. 1. Diagram of the measuring system for electrochemical dissolution; 1 – calomel electrode, 2 – salt-bridge, 3 – sample, 4 – platinum electrode, 5 – electrolytes, 6 – electromagnetic stirrer [9]

The phase constituents in isolates were identified on a Philips X'Pert PRO diffractometer (CoK_α radiation, voltage of 30 kV, current of 40 mA) within an angular range 2Θ from 20 to 120°.

The microstructural examinations of cast steel were carried out on cross-sections of the circular specimens under a Nikkon optical microscope. Surface distribution of elements was examined under a JEOL scanning microscope with EDX attachment. The surfaces of the specimens were mechanically ground with abrasive paper and diamond paste. They were next polished on a cloth impregnated with Al_2O_3 slurry. Etching was made with a reagent of the following composition: 3g $FeCl_3$, 10 cm^3 HCl, 90 cm^3 C_2H_5OH.

3. Results

The as-cast steel structure is shown in Figure 2. It is composed of an austenitic matrix with carbide precipitates in interdendritic spaces. Niobium carbides are structured in a typical way assuming the form of the, so called, Chinese script. Chromium carbides complete the network of the precipitates. The segregation of alloy chemical constituents, chromium and silicon mainly, is visible.

Table 2.
Weight fraction and phase composition of isolates

Temperature °C	Phase composition	Annealing time, h	Isolate fraction wt%
as-cast	NbC, $M_{23}C_6$	-	2.21
600	NbC, $M_{23}C_6$	10	3.62
		100	3.39
		500	3.72
		1000	4.24
700	NbC, $M_{23}C_6$ $Ni_{16}Nb_6Si_7$	10	4.18
		100	4.15
		500	4.01
		1000	4.36
800	NbC, $M_{23}C_6$ $Ni_{16}Nb_6Si_7$	10	4.66
		100	4.10
		500	4.39
		1000	4.79
900	NbC, $M_{23}C_6$ $Ni_{16}Nb_6Si_7$	10	4.31
		100	4.47
		500	4.43
		1000	4.82
1000	NbC, $M_{23}C_6$	10	2.97
	NbC, $M_{23}C_6$, $Ni_{16}Nb_6Si_7$	100	3.01
		500	2.46
	NbC, $M_{23}C_6$	1000	2.37

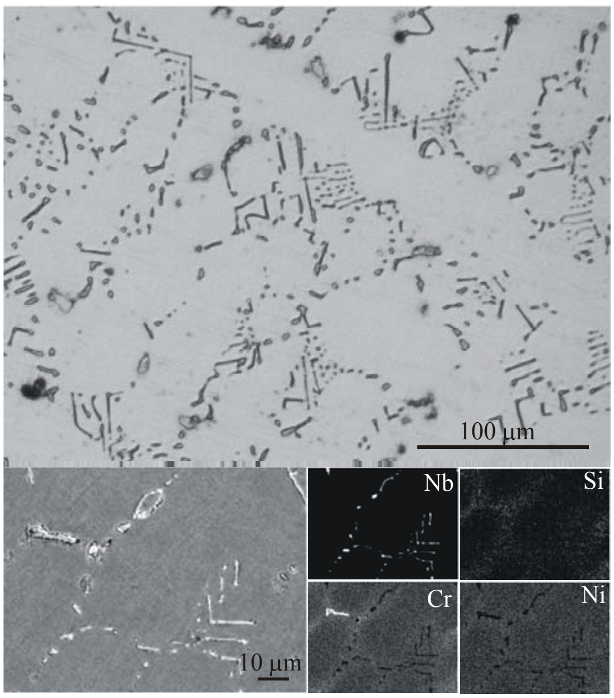

Fig. 2. Microstructure of alloy in as-cast state and distribution of alloy chemical constituents

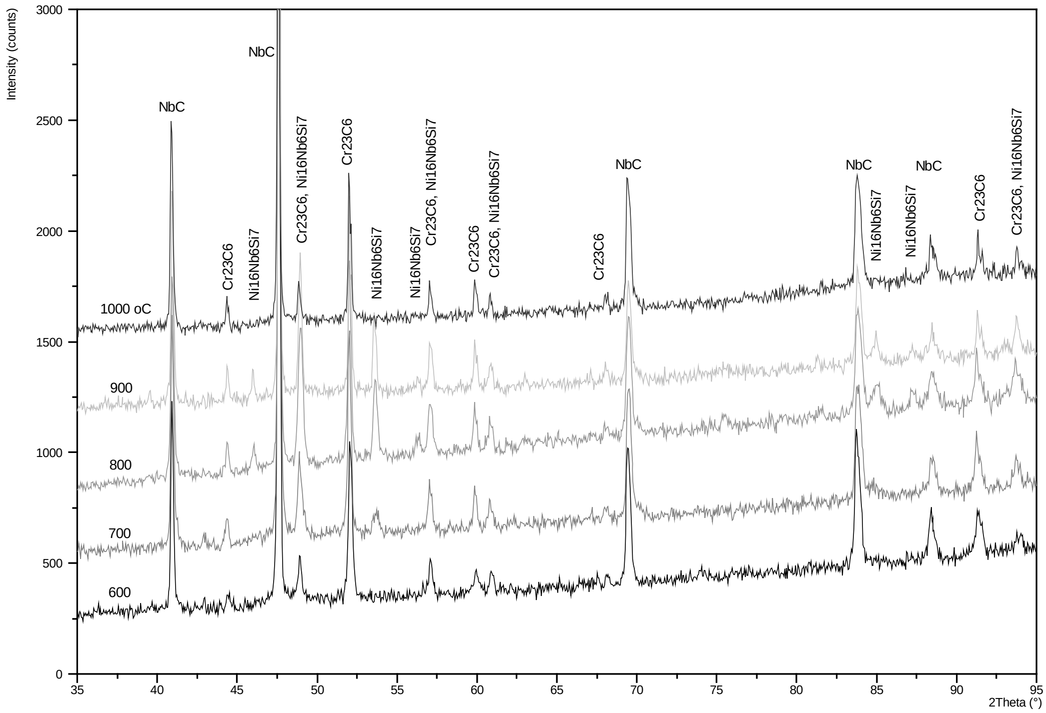

Fig. 3. X-ray diffraction spectra of isolates after 1000 h annealing at temperature 600, 700, 800, 900 i 1000^{o}C

The X-ray diffraction of the isolate of the alloy in as-cast state has proved the presence of two phase constituents, i.e. NbC and $Cr_{23}C_6$ carbides. The total percent content of these constituents in alloy was 2.21 wt% - see Table 2.

Examples of diffraction patterns of the examined alloy after 1000 h annealing within the examined range of temperatures are shown in Figure 3. The phase composition of isolates obtained from all the specimens is given in Table 2. The simple NbC carbides and the complex $Cr_{23}C_6$ chromium carbides were identified for all the annealing times and temperatures. In specimens annealed at the temperature of 700-900^{o}C, also the presence of the $Ni_{16}Nb_6Si_7$ phase, in literature called G phase, was identified. Very small reflections from the G phase were also recorded in specimens annealed at the temperature of 1000^{o}C after 100 and 500 h annealing. The most numerous and most intense reflections originating from the above mentioned phase were found in the diffraction patterns of specimens annealed at the temperatures of 800 and 900^{o}C.

An analysis of the weight percent fractions of the isolates shown in Figure 4a indicates that the quantity of the extracted phases changes in specimens annealed at different temperatures. It increases with the temperature of up to 900^{o}C, to drop quite obviously at 1000^{o}C. This relationship is recurrent in each time interval of the annealing process.

On the other hand, in terms of the process time, one can see that when the state of equilibrium is reached after 10 h annealing, due to the removal of segregations typical of the as-cast state, the amount of the isolate obtained from the specimens annealed at the temperatures of 700, 800, 900^{o}C remains at a relatively constant level until the time of 500 h. It increases after 1000 h annealing. For the specimen annealed at 600^{o}C, this tendency of the increase starts as early as after 500 h, but - contrary to other specimens, when the temperature is 1000^{o}C, the amount of the isolate starts decreasing as early as after 500 h annealing - Fig4b.

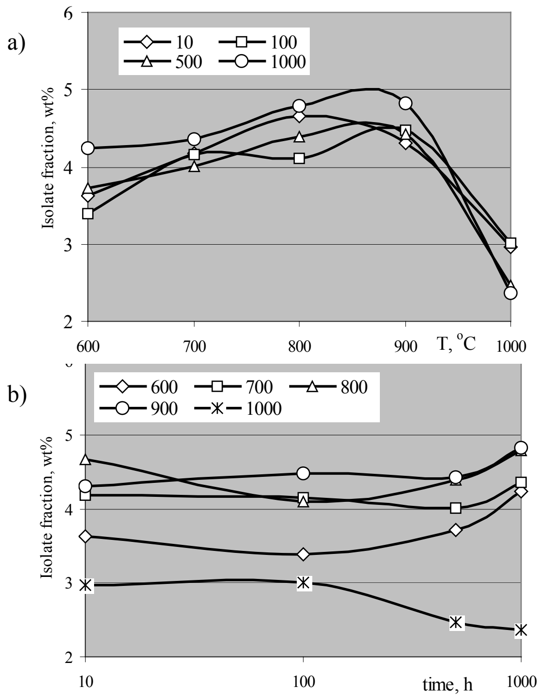

Fig. 4. Weight percent fraction of isolates vs a) temperature and b) time of cast steel annealing

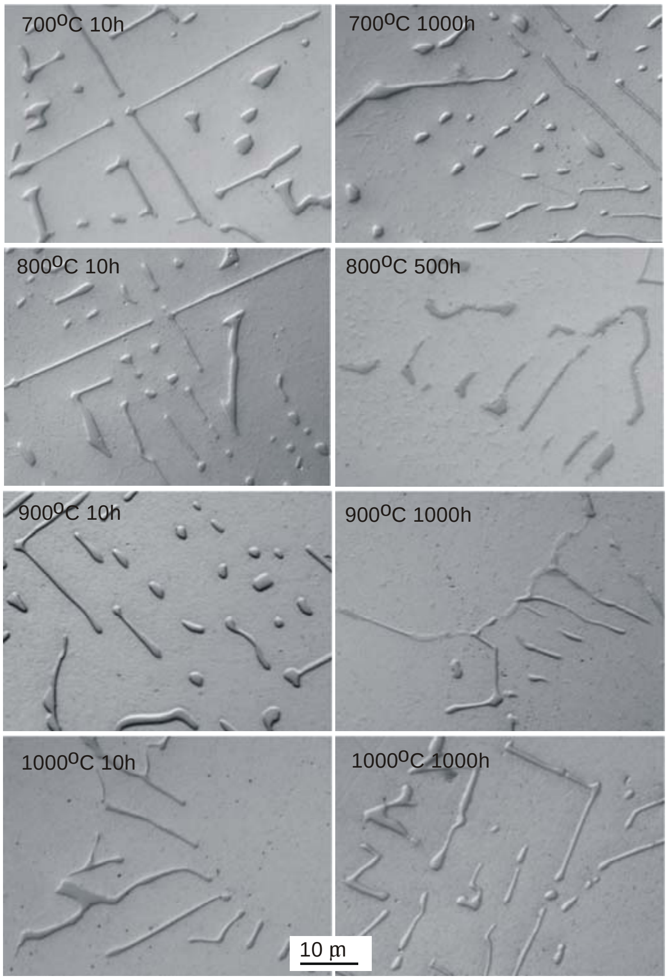

Fig. 5. Microstructure of annealed cast-steel, Nomarski Contrast

The smallest amount of the isolate was obtained from the specimens annealed at the temperature of 1000^{o}C; slightly more was obtained from those annealed at 600^{o}C. For the remaining temperature ranges, these amounts were comparable.

The structure of the annealed cast steel is shown in Figure 5 on the example of some selected specimens. In the structure of the alloy after annealing at the temperature of 600^{o}C no changes in the morphology of the precipitates have been observed. The first secondary precipitates appeared after annealing at the temperature of 700^{o}C. These are the single precipitates present near the primary ones. The first symptoms of the transformation of some NbC carbides are also visible. After annealing at the temperature of 800^{o}C, as early as after 500 h, a distinct transformation of all the primary precipitates was observed along with a high-rate process of the secondary precipitation in alloy matrix. The transformation of NbC carbides is also seen in the structure of the cast steel after annealing at the temperature of 900^{o}C, but it is not accompanied by so intense effect of the secondary precipitation as in the case of the 800^{o}C temperature. In specimens of the alloy annealed at 1000^{o}C no traces of the NbC carbides transformation or of the presence of the secondary precipitates in alloy matrix have been observed.

4. Summary

The metallographic characteristic and the phase identification indicate that precipitation processes in the gravity cast austenitic 18%Cr-30%Ni steel with an addition of 1.84% niobium assume a particularly intense course within the temperature range of 800-900^{o}C. The said processes are mainly those of the secondary precipitation within alloy matrix and phase transformation of NbC carbides into a high-silicon $Ni_{16}Nb_{6}Si_{7}$ phase.

Isolation of phase constituents enables quantitative assessment of the precipitation processes and confirms the metallographic observations. The results obtained on the isolates indicate that within the examined time interval the quantitative content of the respective phases increases in cast steel with longer time of annealing.

At the temperature of 1000^{o}C, the number of the precipitates did not increase, on the contrary, for times of annealing longer than 100 h the amount of the isolate decreased. This effect is probably due to the dissolution of $Cr_{23}C_{6}$ carbides in austenite.

Acknowledgements

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