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Crystallization of GX2CrNiMoCuN 25-6-3-3 Grade Alloy Cast Steel and its Microstructure in the As-cast State and After Heat Treatment

T. Wróbel [a,*], P. Jurczyk [a], C. Baron [a], P. Nuckowski [b]

[a] Silesian University of Technology, Department of Foundry Engineering, Towarowa 7, 44-100 Gliwice, Poland
[b] Silesian University of Technology, Materials Research Laboratory, Konarskiego 18a, 44-100 Gliwice, Poland
* Corresponding author. E-mail address: tomasz.wrobel@polsl.pl

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Abstract

The paper presents the results of research conducted in the field of crystallization and microstructure of duplex alloy cast steel GX2CrNiMoCuN 25-6-3-3 grade. The material for research was the above-mentioned cast steel with a chemical composition compliant with the relevant PN-EN 10283 standard, but melted at the lowest standard allowable concentration of alloying additives (some in short supply and expensive), i.e. Cr, Ni, Mn, Mo, Cu and N. The analysis of the crystallization process was performed based on the DTA (Derivative Thermal Analysis) method for a stepped casting with a thickness of individual steps of 10, 20, 40 and 60 mm. The influence of wall thickness was also taken into account in the cast steel microstructure testing, both in the as-cast state and after solution heat treatment. The phase composition of the cast steel microstructure was determined by using an optical microscope and X-ray phase analysis. The analysis of test results shows that the crystallization of tested cast steel uses the ferritic mechanism, while austenite is formed as a result of solid state transformation. The cast steel under analysis in the as-cast state tends to precipitate the undesirable σ-type Fe-Cr intermetallic phase in the microstructure, regardless of its wall thickness. However, the casting wall thickness in the as-cast state affects the austenite grain size, i.e. the thicker the casting wall, the wider the γ phase grains. The above-mentioned defects of the tested duplex alloy cast steel microstructure can be effectively eliminated by subjecting it to heat treatment of type hyperquenching.

Keywords: Duplex stainless cast steel, Microstructure, Crystallization, DTA, Heat treatment

1. Introduction

The interest in using the cast steel defined as a foundry alloy of iron and carbon with a content of C ≤ 2.1% by weight and other elements, not subject to plastic processing in the technological process for industrial applications according to various sources [1, 2] dates back 200 years. It should be noted here that the lack of technological shaping of cast steel by plastic forming distinguishes this material from similar ones, because of the chemical steel composition. Therefore, the shape of the cast steel part is reproduced by casting the liquid alloy into a sand or metal mold, where the process of its solidification and cooling take place. However, subsequent steps of the technological line often include the heat treatment of steel castings using the same principles and processes as for steel.

Table 1.
The chemical composition of tested duplex cast steel GX2CrNiMoCuN 25-6-3-3 grade

Elements content, wt.%										
C	Si	Mn	Cr	Mo	Ni	Cu	Nb	N	P	S
Tested duplex cast steel										
0.026	0.40	0.55	24.60	2.61	5.15	2.79	0.05	0.12	0.015	0.006
According to PN-EN 10283										
≤ 0.03	≤ 1.00	≤ 1.50	24.50 – 26.50	2.50 – 3.50	5.00 – 7.00	2.75 – 3.50	-	0.12 – 0.22	≤ 0.035	≤ 0.025

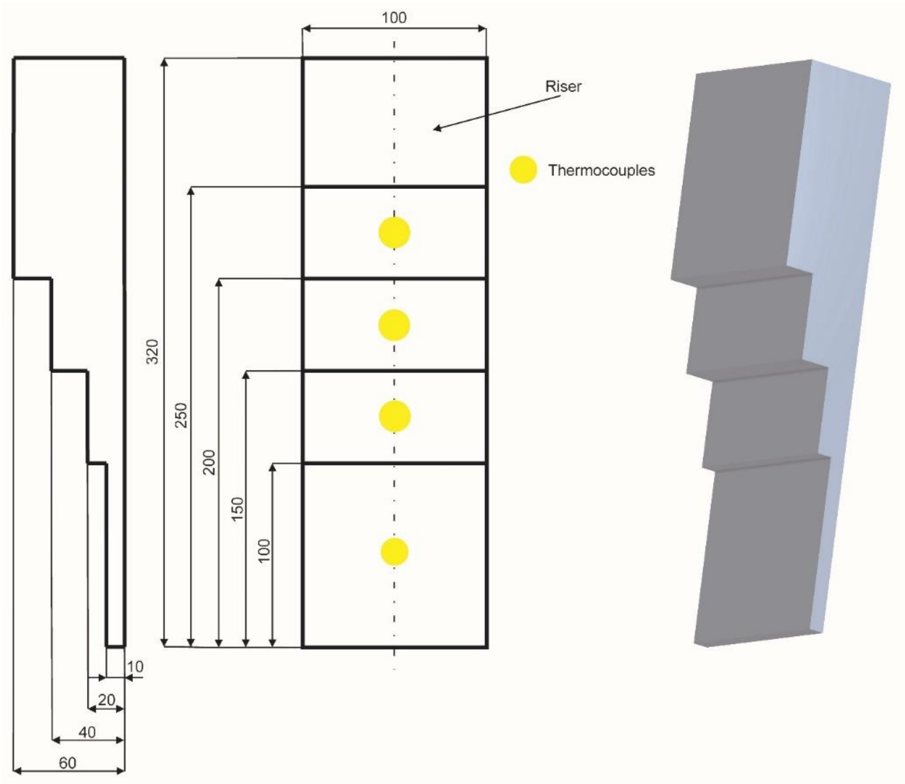

Fig. 1. Scheme of the stepped test casting

When using the above cast steel definition, it should be noted that in addition to the two basic components, i.e. Fe and C, its chemical composition comprises other elements added in a certain concentration, which determines whether the material belongs to the group of unalloyed, the so-called carbon or alloy cast steels. Among the alloy cast steels, one of the most important groups are corrosion-resistant cast steels. Their main alloying additives include Cr, Ni and Mo. Special attention in this group, because of their utility properties, should be paid to alloy cast steels with a ferritic-austenitic microstructure, commonly referred to as duplex cast steels [3 - 14]. When reviewing the historical outline of duplex cast steel presented in articles [3, 4], it can be considered the “youngest” in the group of corrosion-resistant alloy steels. As a result, there is a potential for research and development work covering several aspects of both their chemical composition and the manufacturing process of the final cast.

Therefore, the aim of the article was to describe the crystallization process and the final phase composition of the microstructure in the as-cast state and subsequent heat treatment of duplex cast steel GX2CrNiMoCuN 25-6-3-3 grade, with a chemical composition compliant with the requirements of the relevant PN-EN 10283 standard, but melted with the lowest allowable concentration of alloying additives (some in small supply and expensive), i.e. Cr, Ni, Mn, Mo, Cu and N.

2. Range of studies

The chemical composition of the duplex cast steel under analysis determined using an optical LECO GDS500A glow discharge spectrometer, is shown in Table 1. In spite of the limitation to the so-called “lower limit of the range” concentration for alloy additives, i.e. Cr, Ni, Mn, Mo, Cu and N, the chemical composition of the cast steel under analysis complies with the requirements of the relevant PN-EN 10283 standard (Tab. 1).

Figure 1 shows a scheme of the stepped test casting with the thickness of individual steps 10, 20, 40 and 60 mm, respectively, along with the arrangement of Pt-PtRh10 thermocouples. The cast steel under analysis was melted in an electric induction furnace. The pouring temperature was 1590°C. The mold was made of molding sand prepared on the basis of quartz sand grains in the Alphaset technology. The analysis of the crystallization process was performed using the DTA (Derivative Thermal Analysis) method based on temperature changes determined using the CrystalDigraph-PC recorder during the liquid cast steel solidification and then its cooling in the solid state. The recorded cooling curves T=f(t) allowed us to calculate the dT/dt crystallization curves.

Since the range of the research included both the as-cast and heat-treated state, the solution heat treatment was performed using a LINN HIGH TERM VMK-1600-G resistance electric chamber furnace. Soaking temperature of 1080°C for 900 s and cooling in water at 25°C were used. The selection of heat treatment process parameters results from the analysis of authors' previous papers [11 and 13].

Microscopic metallographic examinations were performed using a NIKON LV150N upright optical microscope, using grinding and polishing for metallographic microsections followed by electrolytic etching. The Mi19Fe etching reagent with the following composition was used: 3g$FeCl_3$, 10 cm^3 HCl and 90 cm^3 C_2H_5OH, used with the following electrolytic etching parameters: voltage 15 V and etching time 30 s. As part of the tests using an optical microscope, a measurement of γ grain size and a quantitative analysis of the share of ferrite and austenite and possibly other phases in the microstructure of duplex cast steel was carried out using the image analysis NIS-Elements v. F3.0 software.

In addition, X-ray diffraction analysis was performed using an XPertPro X-ray diffractometer by PANALYTICAL. Measurements were carried out in the angular range of 2θ from 10 to 120° with a recording step every 0.05°, and a counting time of 50 s. Filtered X-rays from a cobalt anode X-ray tube were used. The phases were identified based on the data contained in the ICDD (International Centre for Diffraction Data) database.

In addition, hardness testing for individual steps of the step casting in the as-cast state and after heat treatment was performed using the Brinell method with the KABID PRESS B4CS hardness tester and an indenter using a sintered carbide ball 10 mm in diameter and loaded with a force of 29420 N.

3. Study results and discussion

Figure 2 shows cooling curves recorded for individual steps of stepped casting. It was found that due to the different cooling kinetics for castings with different wall thicknesses, for variants 10, 20 and 40 mm, not all significant thermal effects originating from phase transformations occurring in the cast steel GX2CrNiMoCuN 25-6-3-3 grade solidified in the mold were recorded. Considering the above-mentioned, the DTA analysis was performed descriptively for a casting with a wall thickness of 60 mm (Fig. 3 and 4). The liquidus temperature was found to be 1476°C, while the crystallization end temperature was 1396°C. In addition, a thermal effect caused by the solid state transformation α→γ was identified. It took place at 1243°C.

In general, the experimentally determined values of characteristic temperatures are consistent with those determined by the calculation method using the Thermo-Calc Software, which is presented in detail in article [13] (Tab. 2). Some slight differences may result from the variable kinetics of the actual solidification process, not taken into account by the numerical calculation model. However, the DTA analysis shows no thermal effects from $M_{23}C_6$ carbides and σ phase. This may result from the sensitivity of the measurement method used for phases that occur in smaller amounts in the casting structure. The thermal effects resulting from the precipitation of the σ phase are shown in article [7]. It presents the results of the study on the crystallization mechanism for a similar duplex cast steel grade that contains a greater amount of the above-mentioned intermetallic Fe-Cr phase in the as-cast state.

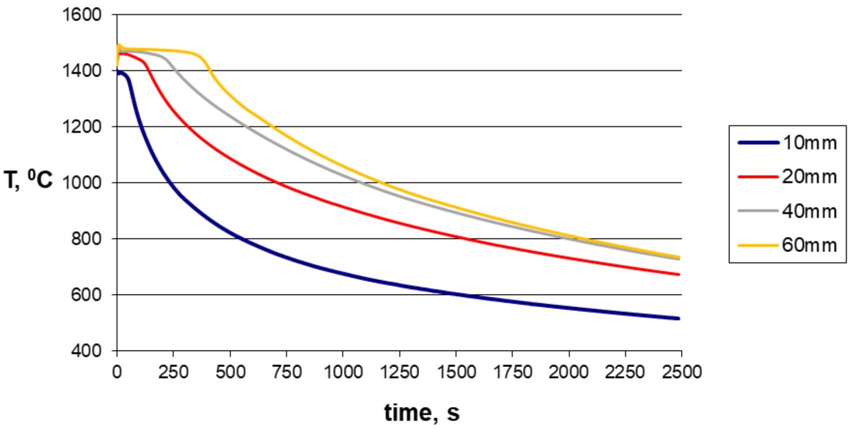

Fig. 2. Cooling curves of the stepped test casting of cast steel GX2CrNiMoCuN 25-6-3-3 grade at the thickness of individual steps 10, 20, 40 and 60 mm

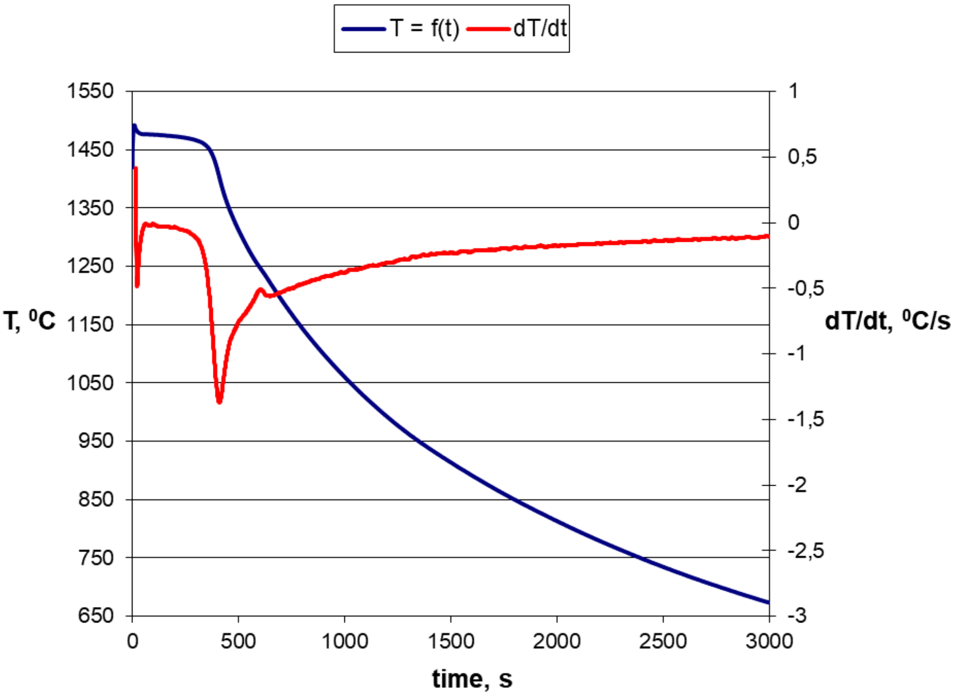

Fig. 3. Cooling and crystallization curves of the cast steel GX2CrNiMoCuN 25-6-3-3 grade test casting at the wall thickness 60 mm

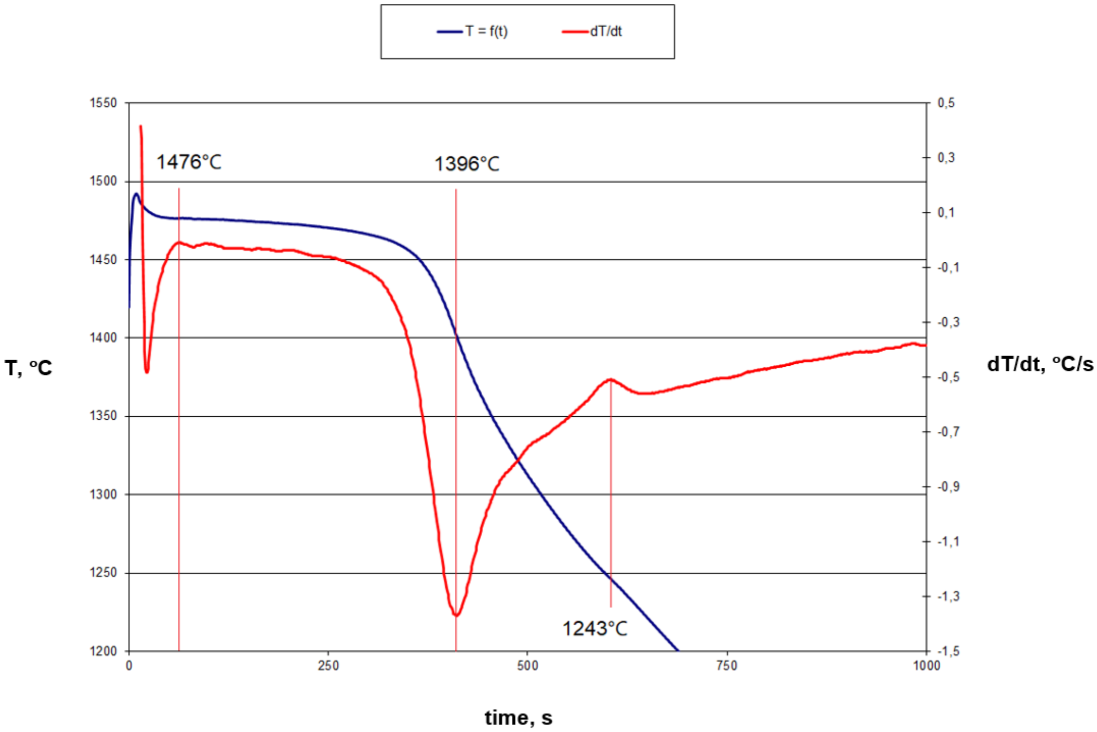

Fig. 4. Magnification for the cooling and crystallization curves of the cast steel GX2CrNiMoCuN 25-6-3-3 grade test casting at the wall thickness 60 mm

Table 2.
Temperatures characteristic for the process of primary and secondary crystallization of cast steel GX2CrNiMoCuN 25-6-3-3 with chemical composition according to Tab. 1

Phase transformation	**According to**	
	The real experiment (Fig. 4)	Thermo-Calc Software [13]
T (liquidus), °C	1476	1480
T (solidus), °C	1396	1373
T ($\alpha \rightarrow \gamma$), °C	1243	1307
T ($\alpha/\gamma \rightarrow M_{23}C_6$), °C	-	933
T ($\alpha \rightarrow \sigma + \gamma_{(secondary)}$), °C	-	820

The results obtained in the crystallization tests of GX2CrNiMoCuN 25-6-3-3 grade cast steel were verified based on the analysis of the microstructure of castings with a wall thickness of 10, 20, 40 and 60 mm in the as-cast state shown in Figures 5 to 8. It was found that in the as-cast state, regardless of the thickness of the casting wall, the microstructure of the cast steel GX2CrNiMoCuN 25-6-3-3 grade consists of ferrite α, austenite γ and intermetallic phase σ located on γ grain boundaries. However, the casting wall thickness affects the grain size, which for the cases under analysis can be expressed by the width of the austenitic phase. The measurements performed show that for a wall thickness of 10, 20, 40 and 60 mm, the average width of austenite grains is approximately 16, 17, 20 and 25 µm, respectively. In addition, the qualitative analysis of the microstructures examined, shows no significant effect of the wall thickness on the amount of the σ phase. This finding is confirmed by the X-ray diffractograms of the castings under analysis presented in Figure 9. Only peaks originating from α ferrite and γ austenite were identified. Therefore, it can be concluded that the amount of the σ phase in the castings under analysis with different wall thicknesses is much smaller than calculated for the model solidification conditions using the Thermo-Calc Software package (16.4% according to [13]), while its amount does not exceed 4%, i.e. is below the phase identification threshold determined with an X-ray diffractometer.

Whereas, the performed quantitative analysis was used to determine the effect of a wall thickness on the amount of ferrite α, austenite γ and σ phase. Based on obtained results was concluded, increasing a wall thickness slightly increases the amount of α and σ and decreases the amount of γ. The measurements show that for a wall thickness of 10, 20, 40 and 60 mm, the average amount of α/γ/σ is 52/46/2, 55/41/4, 55/41/4 and 59/37/4, respectively. In general, the similar tendency of the influence of the wall thickness on the phase composition in duplex cast steels is also presented in the papers [6 and 14].

In addition, the presence of the σ phase in such a small amount does not significantly affect the hardness of the tested cast steel GX2CrNiMoCuN 25-6-3-3 grade. The measurements performed show that for a wall thickness of 10, 20, 40 and 60 mm, the average hardness in the as-cast state is 229 (±5), 241(±9), 229 (±7) and 217 (±7) HB, respectively.

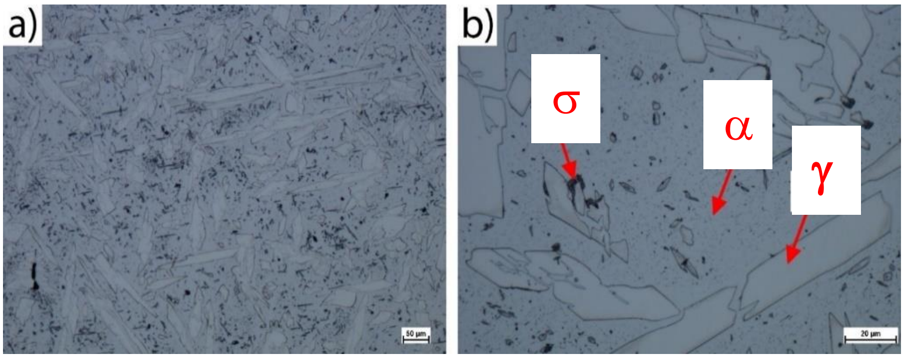

Fig. 5. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel in as-cast state at the wall thickness 10mm: a) mag. 100x, b) mag. 500x, α ferrite, γ austenite and intermetallic σ phase

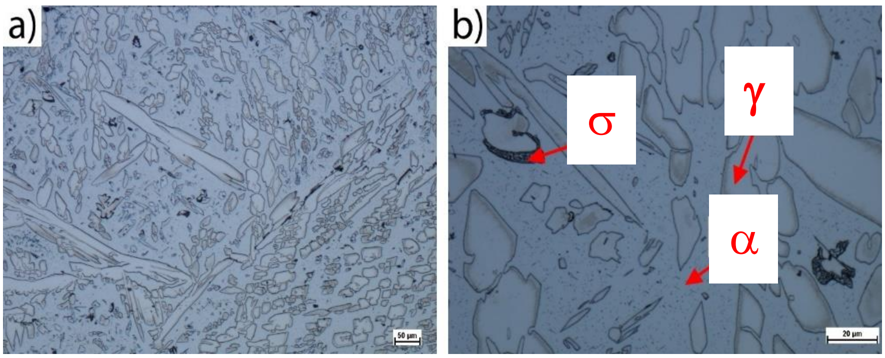

Fig. 6. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel in as-cast state at the wall thickness 20mm: a) mag. 100x, b) mag. 500x, α ferrite, γ austenite and intermetallic σ phase

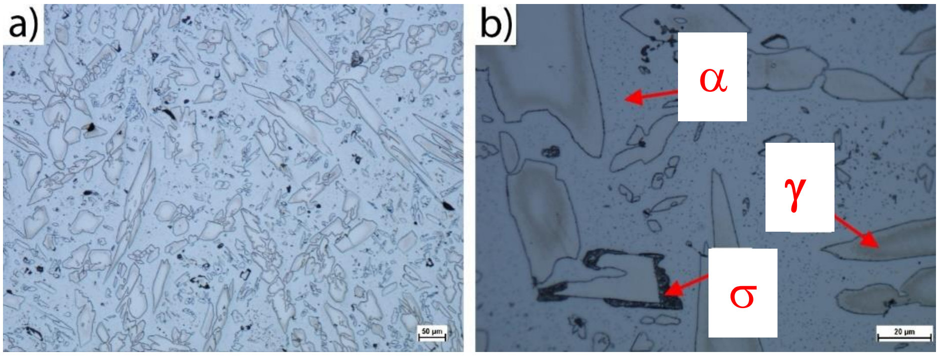

Fig. 7. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel in as-cast state at the wall thickness 40mm: a) mag. 100x, b) mag. 500x, α ferrite, γ austenite and intermetallic σ phase

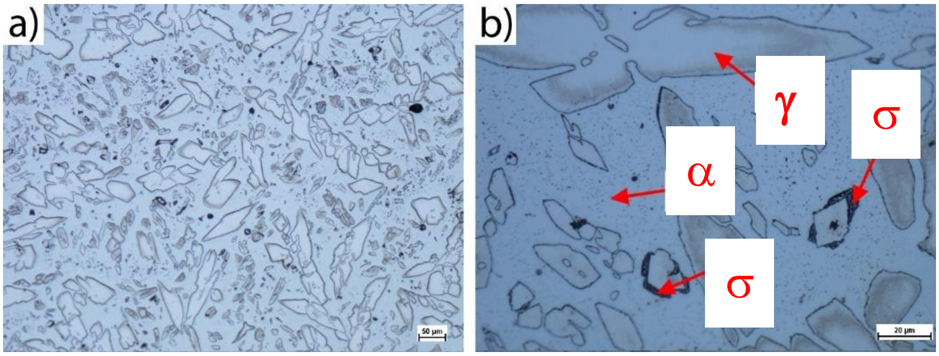

Fig. 8. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel in as-cast state at the wall thickness 60mm: a) mag. 100x, b) mag. 500x, α ferrite, γ austenite and intermetallic σ phase

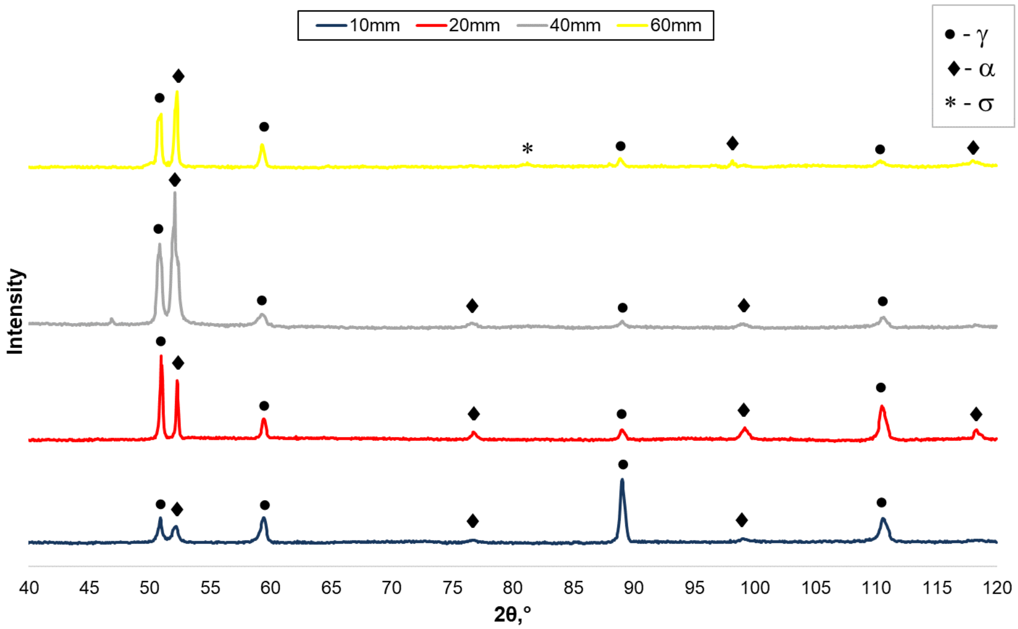

Fig. 9. X-ray diffractograms of tested GX2CrNiMoCuN 25-6-3-3 cast steel in as-cast state at the wall thickness 10, 20, 40 and 60mm

In spite of small amounts of σ phase found in the microstructure of the tested cast steel GX2CrNiMoCuN 25-6-3-3 grade, its complete elimination is necessary to obtain the utility properties required, as described in detail in articles [3, 5, 6 and 11-13]. Considering the above-mentioned, the tested castings were subjected to heat treatment of type hyperquenching from 1080°C.

This procedure allowed us to obtain a two-phase α+γ microstructure free of σ phase precipitates (Fig. 10 to 13). In addition, this type of heat treatment allowed us to homogenize the microstructure in relation to the casting wall thickness. The measurements performed show that for a wall thickness of 10, 20, 40 and 60 mm, the average width of austenite grains is approximately 10, 13, 14 and 13 μm, respectively.

By contrast, figure 14 shows X-ray diffractograms for the tested castings after hyperquenching, proving that their microstructure consists of α ferrite and γ austenite.

Moreover based on the results of the quantitative analysis was concluded, the used heat treatment uniformed the phase composition on the wall thickness of the casting. Therefore after hyperquenching in studied duplex cast steel the average amount of ferrite α equaling 52% and austenite γ equaling 48%. Additionally, it should be noted that the above-mentioned content of α and γ in the microstructure of studied duplex cast steel with the chemical composition according to Table 1 and after hyperquenching from temperature 1080°C is similar for calculated based on formulas (1-4) presented in papers [6, 9 and 11]:

$$Cr_{eq} = wt.\%Cr + 1.73wt.\%Si + 0.88wt.\%Mo = 27.6wt.\% \quad (1)$$

$$Ni_{eq} = wt.\%Ni + 24.55wt.\%C + 21.75wt.\%\ N + 0.40wt.\%Cu = 9.5wt.\% \quad (2)$$

$$\alpha = 4.010Cr_{eq} - 5.600Ni_{eq} + 0.016T_p - 20.93 = 53.8\% \quad (3)$$

$$\gamma = 100 - \alpha = 46.2\% \quad (4)$$

where:
Cr_{eq} - equivalent content of chromium, Ni_{eq} - equivalent content of nickel; T_p – hyperquenching temperature in °C; α – percentage amount of ferrite in microstructure of duplex cast steel; γ – percentage amount of austenite in microstructure of duplex cast steel;

In addition, after the hyperquenching of the tested cast steel GX2CrNiMoCuN 25-6-3-3 grade, no significant changes in the hardness of the castings were found as compared to the as-cast state. The measurements performed show that for a wall thickness of 10, 20, 40 and 60 mm, the average hardness after heat treatment is 229 (±5), 217 (±8), 217 (±7) and 207 (±4) HB, respectively.

Summing up, the cast steel GX2CrNiMoCuN 25-6-3-3 grade with a developed chemical composition crystallizes using the ferritic mechanism, while austenite is formed as a result of transformation in the solid state. The presented crystallization mechanism confirms the conclusions and observations developed by the author of the paper [7] based on testing duplex cast steel with a similar chemical composition. The cast steel under analysis in the as-cast state tends to precipitate the undesirable σ-type Fe-Cr intermetallic phase in the microstructure, regardless of its wall thickness. However, the casting wall thickness in the as-cast state affects the austenite grain size, i.e. the thicker the casting wall, the wider the γ phase. The above-mentioned defects of the tested duplex alloy cast steel microstructure can be effectively eliminated by subjecting it to heat treatment of type hyperquenching. The hyperquenching from 1080°C made it possible to eliminate the σ phase precipitates as well as to homogenize the microstructure and significantly reduce its sensitivity to the casting wall thickness. Another way to reduce the amount of the σ phase precipitated in duplex cast steel castings is knocking them out of the mold after they reach a temperature of approx. 1000°C and then subjecting them to air cooling, as presented in papers [6 and 13].

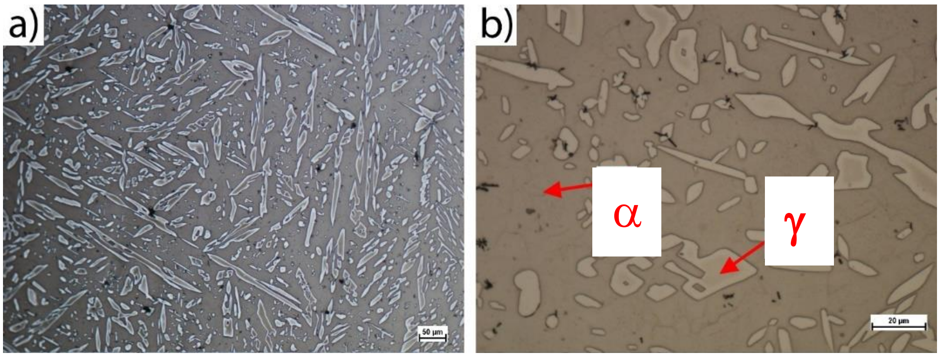

Fig. 10. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel after hyperquenching from 1080°C at the wall thickness 10mm: a) mag. 100x, b) mag. 500x, α ferrite and γ austenite

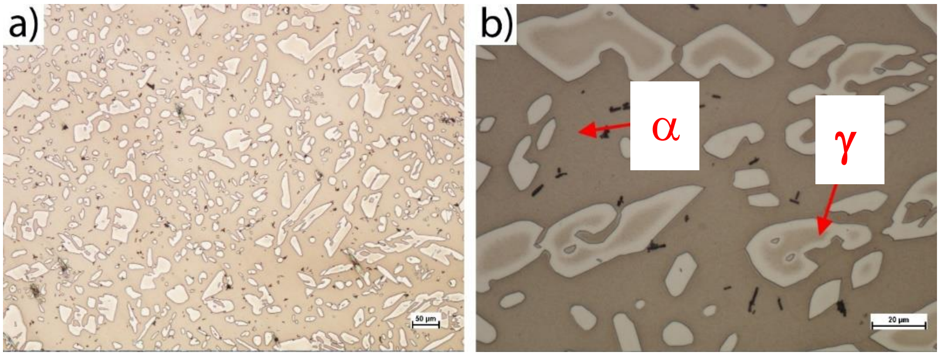

Fig. 11. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel after hyperquenching from 1080°C at the wall thickness 20mm: a) mag. 100x, b) mag. 500x, α ferrite and γ austenite

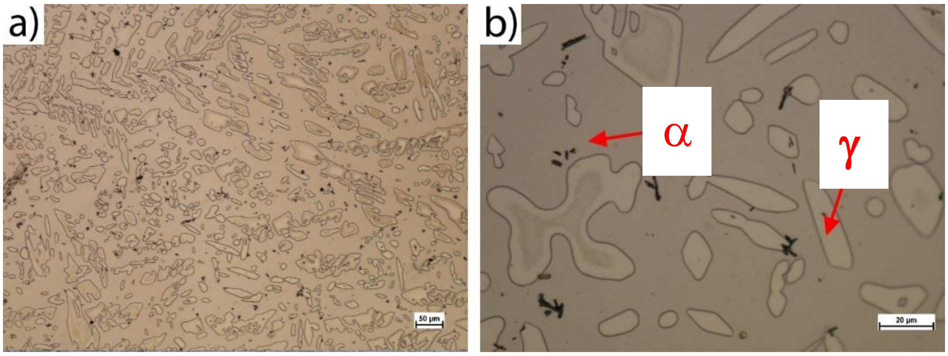

Fig. 12. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel after hyperquenching from 1080°C at the wall thickness 40mm: a) mag. 100x, b) mag. 500x, α ferrite and γ austenite

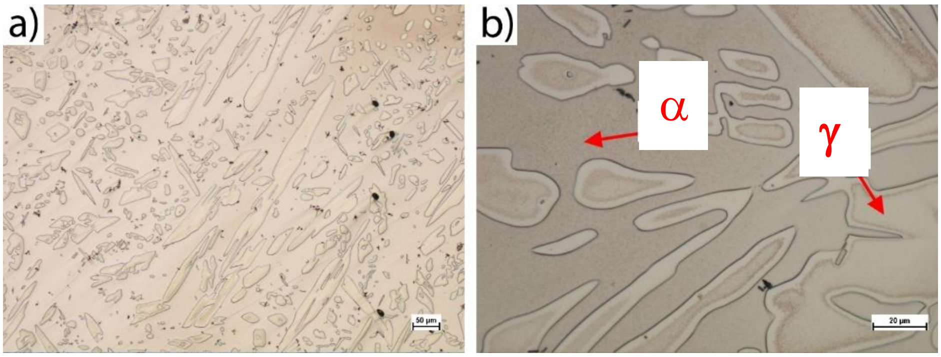

Fig. 13. Microstructure of tested GX2CrNiMoCuN 25-6-3-3 cast steel after hyperquenching from 1080°C at the wall thickness 60mm: a) mag. 100x, b) mag. 500x, α ferrite and γ austenite

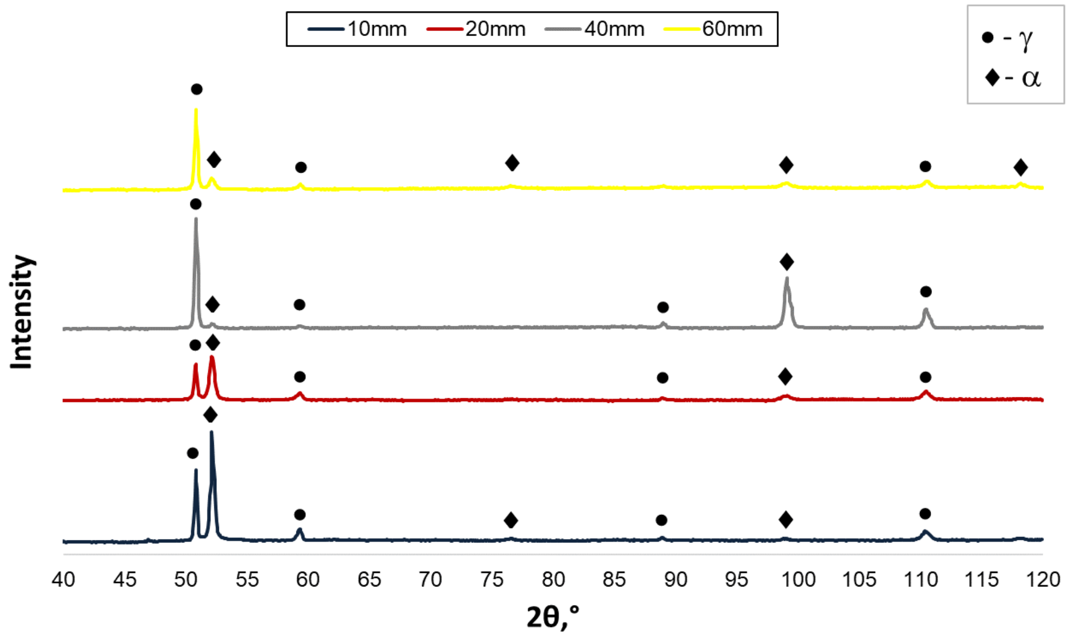

Fig. 14. X-ray diffractograms of tested GX2CrNiMoCuN 25-6-3-3 cast steel after hyperquenching from 1080°C at the wall thickness 10, 20, 40 and 60mm

4. Conclusions

The analysis of results obtained leads to the following conclusions:

1. The duplex alloy cast steel GX2CrNiMoCuN 25-6-3-3 grade with a chemical composition under analysis crystallizes using the ferritic mechanism, while austenite is formed as a result of transformation in the solid state.
2. The duplex alloy cast steel GX2CrNiMoCuN 25-6-3-3 grade with a chemical composition under analysis tends to precipitate the undesirable σ-type Fe-Cr intermetallic phase in the microstructure, regardless of the casting wall thickness.
3. Raising the wall thickness of the duplex alloy cast steel GX2CrNiMoCuN 25-6-3-3 grade casting with the chemical composition under analysis favors the thickness of austenite grain in the as-cast state.
4. For duplex alloy cast steel GX2CrNiMoCuN 25-6-3-3 grade with the chemical composition under analysis, heat treatments such as hyperquenching from 1080°C make it possible to completely eliminate the σ phase precipitates and homogenize the microstructure as well as to significantly reduce its sensitivity to the casting wall thickness.

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