

Application of Silicon Carbide Chills in Controlling the Solidification Process of Casts Made of IN-713C Nickel Superalloy

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Abstract

The paper presents the method of manufacturing casts made of the IN-713C nickel superalloy using the wax lost investment casting process and silicon carbide chills. The authors designed experimental casts, the gating system and selected the chills material. Wax pattern, ceramic shell mould and experimental casts were prepared for the purposes of research. On the basis of the temperature distribution measurements, the kinetics of the solidification process was determined in the thickened part of the plate cast. This allowed to establish the quantity of phase transitions which occurred during cast cooling process and the approximate values of liquidus, eutectic, solidus and solvus temperatures as well as the solidification time and the average value of cast cooling rate. Non-destructive testing and macroscopic analysis were applied to determine the location and size of shrinkage defects. The authors present the mechanism of solidification and formation of shrinkage defects in casts with and without chills. It was found that the applied chills influence significantly the hot spots and the remaining part of the cast. Their presence allows to create conditions for solidification of IN-713C nickel superalloy cast without shrinkage defects.

Keywords: Chills, Silicon Carbide, IN-713C, Precision Casting, Shrinkage Porosity

1. Introduction

The presence of shrinkage defects is the reason for the deterioration of blades casts made of nickel superalloys and their mechanical properties. The analysis of blades casts revealed a presence of shrinkage defects, mainly shrinkage porosity in the hot spots - the airfoil and the root of blades [1]. The shrinkage porosity arises due to the lack of access of the liquid metal filling the interdendritic space during the solidification process. The loss of ability of feeding with liquid phase results in formation of shrinkage porosity between the dendrites branches [2].

Shrinkage defect in the hot spot of nickel superalloy cast manufactured with a use of the wax lost investment casting process can be eliminated by changing the feeding conditions and solidification process. The application of riser located over the hot spot results in shifting the shrinkage defect towards it [3]. Also modification of the gating system design through pouring the liquid metal into the cast area characterized by the largest thickness, creates conditions for proper feeding of the cast. During the manufacturing process of nickel superalloys blades casts with a use of the wax lost investment casting process, the location of pouring the liquid metal to the mould and position of the riser depend on cast shape and manufacturer guidelines. Therefore, risers and pouring with liquid metal can be applied

only in those areas of blade casts which will be further submitted for machining - area of shroud tip and root of the blade. Up to now, warming up of the ceramic shell mould and the gating system - used as a riser - has been the most commonly used method of controlling the cast solidification process [1]. The application of thermal insulating layer allows to warm the mould. In that way the rate of cooling and solidification decrease and the feed conditions for the cast improve. Controlling the solidification process of the blades casts higher than 200 mm, requires using a time-consuming method - application of several thermal insulating layers. It is often necessary to use the local cast cooling, which in case of using thermal isolation is difficult or impossible to realize.

The application of local cooling for the cast and decreasing the quantity of risers and amount of thermal insulating layers is possible by using chills. Currently, the chills are used to control the solidification process, mainly in case of the cast steel and cast iron castings [4]. In spite of many advantages they hasn't been used for manufacture of blades casts made of nickel superalloys (wax lost investment casting process) and characterized by equiaxed microstructure. On the basis of the literature analysis it was found that the graphite chills were employed only to create the single crystal casts. They allow to control the solidification front in the blade transition region between the airfoil and the blade root [5]. Therefore, it has been assumed that the application of chills will improve the manufacturing process of the blades casts. To achieve that, the authors made an attempt to develop the technology of manufacturing casts made of nickel superalloys by applying the wax lost investment casting process, with application of chills. The influence of chills on the kinetics of solidification process and the formation of shrinkage pores was presented in the paper.

2. The research methodology

Four wax models of plates casts (width of 50 mm, height of 100 mm and thickness of 5mm) were designed and manufactured - H1, H2, H3, H4 (fig. 1). In the middle part of the plates, the thickness was increased from one (H2, H3, H4) or both sides, over the length of 23 mm. Four wax models of casts and pouring cup were connected with the gating system into the wax pattern.

A wax pattern was used as a base for creating the multilayer ceramic shell mould. The first, approx. 1 mm thick, ceramic layer was obtained on the basis of corundum. Afterwards, the silicon carbide chills (50x23x8 mm) were placed on the layer of ceramic shell mould (fig. 2). Chills located on both sides were applied for the casts H1 and H2. In case of cast H3, chills only on one side were used. Cast H4 had no chills. It resulted in creation of solidification conditions for the cast with the shrinkage defect in the thickened part of the cast. Next layers of the ceramic shell mould were applied on the set, after attaching the chills. The ceramic shell mould with walls of approximate average thickness of 12 mm was made. After drying the mould, wax was melted out in the autoclave and during initial annealing. Subsequently, the thickness of layer of the ceramic shell mould above the chill, was mechanically reduced to approx. 2 mm (fig. 3).

The temperature measurement of the cooled element was carried out in order to determine the kinetics of solidification process. The measurement was conducted in the middle of the thickened part of the cast, at a height of 43.5mm above the cast base (fig. 1). The type B thermocouples (PtRh30 - PtRh6) with diameter of 0.2 mm were applied. In order to protect thermocouples against degradation, they were placed inside the uniform, bended, two-hole ceramic tubes with an external diameter of 3.5mm (fig. 3). The thermocouples junctions were located in one-hole ceramic jackets with an external diameter of 1,5 mm due to protect them against the influence of liquid alloy.

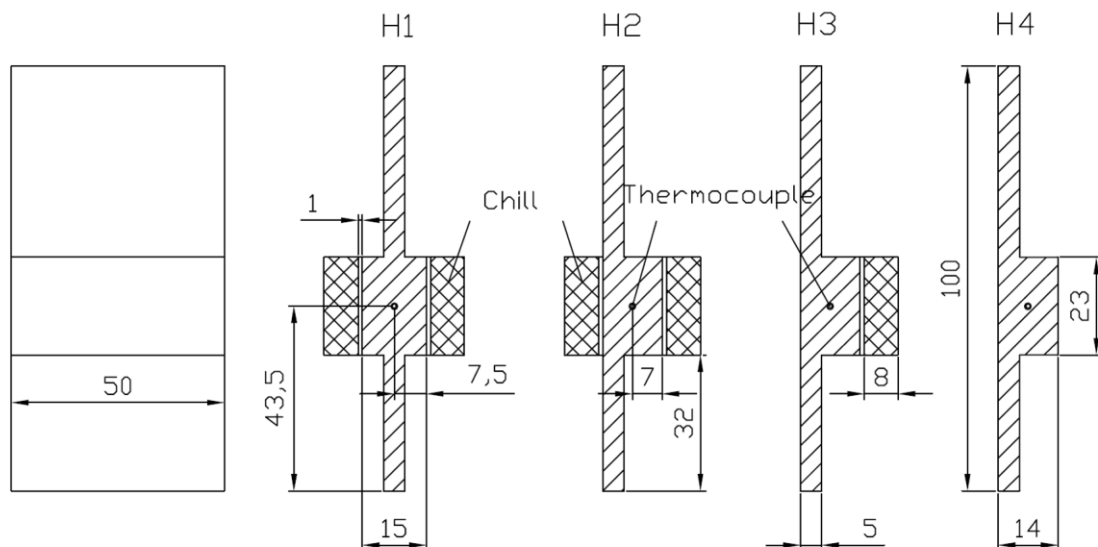


Fig. 1. Techniques of modifying the plate geometry for creating the hot spots as well as the location and size of chills.

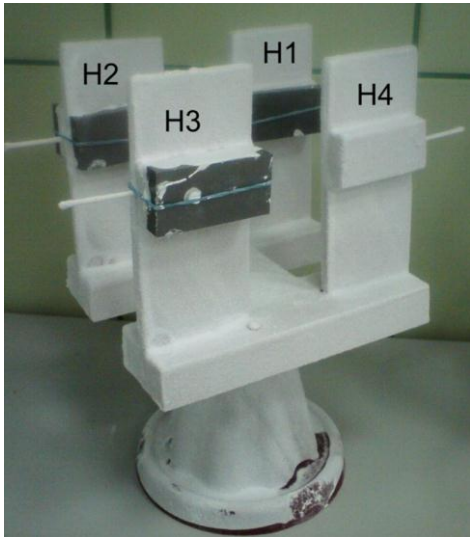


Fig. 2. The model set with chills located on the first layer of ceramic shell mould and ceramic shields of thermocouples

The solidification process of casts made of the IN-713C nickel superalloy (tab. 1) was performed in the VIMIC 2 E - DS/SC vacuum furnace manufactured by ALD Vacuum Technologies GmbH in the Research and Development Laboratory for Aerospace Materials at Rzeszów University of Technology. Charge weighting 4 kg was inductively melted in vacuum of the order 10^{-4} Pa. After the ceramic shell mould was placed in the heating chamber of the furnace it was annealed in the temperature of 1200 °C, during 2 h period. After pouring with IN-713C nickel superalloy at a temperature of 1470°C, the mould was shifted from the heating area to the cooling area of the furnace, in approx. 10 s. Afterwards the cooling chamber was vented in approx. 60 s.



Fig. 3. The ceramic shell mould with thermocouples, after pouring with IN-713C superalloy

Table 1.

Chemical composition of IN-713C superalloy

Elements content, in %wt.								
Cr	Mo	Ta + Nb	Al	Ti	B	C	Zr	Ni
12,5	4,2	2,0	6,1	0,8	0,012	0,12	0,10	The rest

The manufactured experimental casts were submitted to roentgenographic examination in Precision Foundry of WSK „PZL-Rzeszów” S.A. Areas with shrinkage defects were detected. The macroscopic examination of the surface of cast longitudinal section were carried out in order to verify the non-destructive tests and identify the shrinkage defects. Additionally, the GDS GD PROFILER HR spectrometer was applied for determining the chemical composition of IN-713C alloy on the surface of cast located under the chill. The kinetics of the cast solidification process was determined on the basis of experimentally obtained temperature distribution. Cooling rate of the cast were calculated using equation (1):

$$v_{ch} = \frac{dT}{dt} \quad (1)$$

where:

v_{ch} - cooling rate, °C/s

T - temperature, °C

t - time, s

3. Results of experimental research and their analysis

The authors used chills to control the solidification process of nickel superalloys casts. It has been assumed that the chill material should be characterized by:

- high oxidation resistance in the temperature up to 1450 °C,
- the value of thermal conductivity coefficient higher than the value for ceramic shell mould material,
- the value of linear expansion coefficient of the chill material which is close to the value for ceramic shell mould, in the whole temperature range,
- the lack of reaction with liquid metal and the ceramic shell mould.

Selection of chill material was done on the basis of own research and literature analysis. Silicon carbide in two forms - SiSiC and SSiC - was used as the chill material. The values of thermal conductivity coefficient for SiSiC and SSiC decrease with a rise of temperature. For the temperature of 1200°C its value is equal to approx. 32 W/mK (tab. 2). Whereas, in room temperature, the value of thermal conductivity coefficient for SSiC is higher than for SiSiC and is equal to, respectively, 124 and 100 W/mK. It was established that the thermal conductivity coefficient in the temperature of 1200°C is approx. 30 times higher than for ceramic shell mould. The analysis of literature data showed that SSiC is characterized by the most favourable thermal properties. Therefore, this material was used for manufacture of nickel superalloys casts using the wax lost investment casting process.

Table 2.

Thermal conductivity coefficient of materials of chills and ceramic shell mould depending on the temperature

Temperature	Thermal conductivity, W/mK		
	Ceramic shell mould [6]	SiSiC [7]	SSiC [7]
20	0.987	100	124
300	0.903	-	-
600	0.849	-	-
900	1.026	-	-
1200	1.29	32	33

The analysis of temperature measurements results allows to state that during filling the ceramic shell mould with liquid metal, the temperature decreases. It is a result of its contact with the inner surface of the mould. The temperature of liquid metal, which fills the mould in the thickened part of the cast, is equal to approx. 1400°C. After filling the ceramic shell mould, it was shifted from the heating area to the cooling area of the furnace in approx. 10 s. It was observed that the liquid alloy temperature had been decreasing till the equilibrium solidification temperature (liquidus point) was reached. It was established, on the basis of the literature analysis [8], that the temperature of the beginning of solidification process, depends on the cooling rate and the method of its determination. For IN-713C alloy it is in the range of 1336÷1343 °C. For the purposes of further analysis of results and calculations, it was assumed that the beginning of nucleation of γ phase determines the liquidus temperature of the alloy and is equal to 1340°C. Reducing the temperature below the liquidus point leads to nucleation of the γ phase in the alloy [9]. The cooling rate decreases intensively until it reaches positive value. In the time range from 30 to 55 s the temperature attain its minimum and the overcooling degree - both its minimum and maximum value. It is followed by a rise of alloy cooling rate up to 200 s. After 200 s the cooling rate of the cast diminishes. It is a result of eutectic transformation of ($\gamma + \gamma'$) alloy [10]. It was assumed in the paper that the temperature of the beginning of eutectic nucleation (approx. 1302°C) corresponds to the maximum cooling rate in a time range 50 ÷ 220 s. Further cooling and growth of overcooling degree leads to solidification of ($\gamma + \gamma'$) eutectic in the interdendritic spaces. End of solidification process is determined by the solidus temperature of the alloy - 1259°C. It corresponds to the maximum cooling rate in the time range from 200 to 250 s. Below the solidus temperature the separation of γ' phase is present [11]. Similarly to the previous cases, this transformation is characterized by intensive change of cooling rate and temperature. The heat of transition is released and the cast cooling rate becomes lesser. Temperature T_{solIS} determines beginning of the transformation and the solvus temperature which is fundamental for the thermal treatment process of IN-713C alloys. The end of transformation is determined by T_{solIE} temperature - the beginning of cooling process with constant velocity.

The process of pouring the ceramic shell mould and further cooling of liquid metal was conducted in vacuum up to 45 s. The heat exchange in the heating and cooling chamber of the furnace took place by radiation. After venting the cooling chamber of the

furnace with air in a time range from 45 to 120 s, the further heat exchange was realized through radiation and convection (fig. 5).

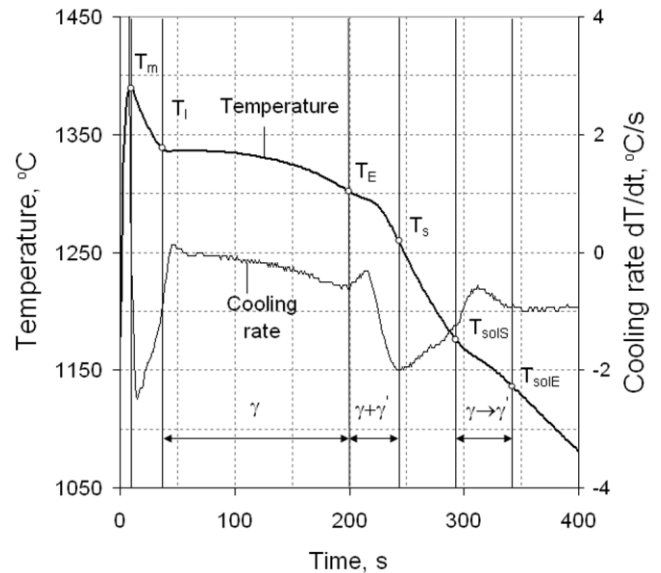


Fig. 4. The temperature and cooling rate in H4 cast (without chills) as a function of time, T_M - maximum temperature, T_L - liquidus temperature, T_E - eutectic solidification temperature T_S - solidus temperature, T_{solIS} - temperature of the beginning of γ' phase separation, T_{solIE} - temperature of the end of γ' phase separation

Application of chills in the hot spots of the casts influences significantly the temperature and the cooling rate (fig. 5). From the moment of mould pouring process to approx. 14 s, the cooling rate of the thickened part increases for casts H1, H2 and H3. The cooling rate of casts equipped with chills, reaches its minimum value after approx. 38 s. The cooling rate increases afterwards. Intensive heat abstraction from the liquid alloy through chills or phase transition occurring in chill material, are probable reasons of this phenomenon. Heat flow from the liquid metal to the chill causes its heating up above the value of service temperature - 1350°C. It is possible that phase transition of chill material proceeds in this temperature range. It is confirmed by the change in character of the cooling curve. Solidification heat is released in the nucleation temperature of the γ phase - it is accompanied by the intensive decrease of the cooling rate. It has been established that application of chills increases also the overcooling degree of the alloy.

A significant change in cooling rate for the thickened part of the cast occurs after approx. 70 s. It is probably caused by the location of thermocouple in its centre. In the first instance the external layer of the cast solidifies (area near the chill). After some time the chill influences the centre of solidifying cast - it results in the abrupt change of cooling rate. The further course of solidification process is the same as in a case of the cast without chills.

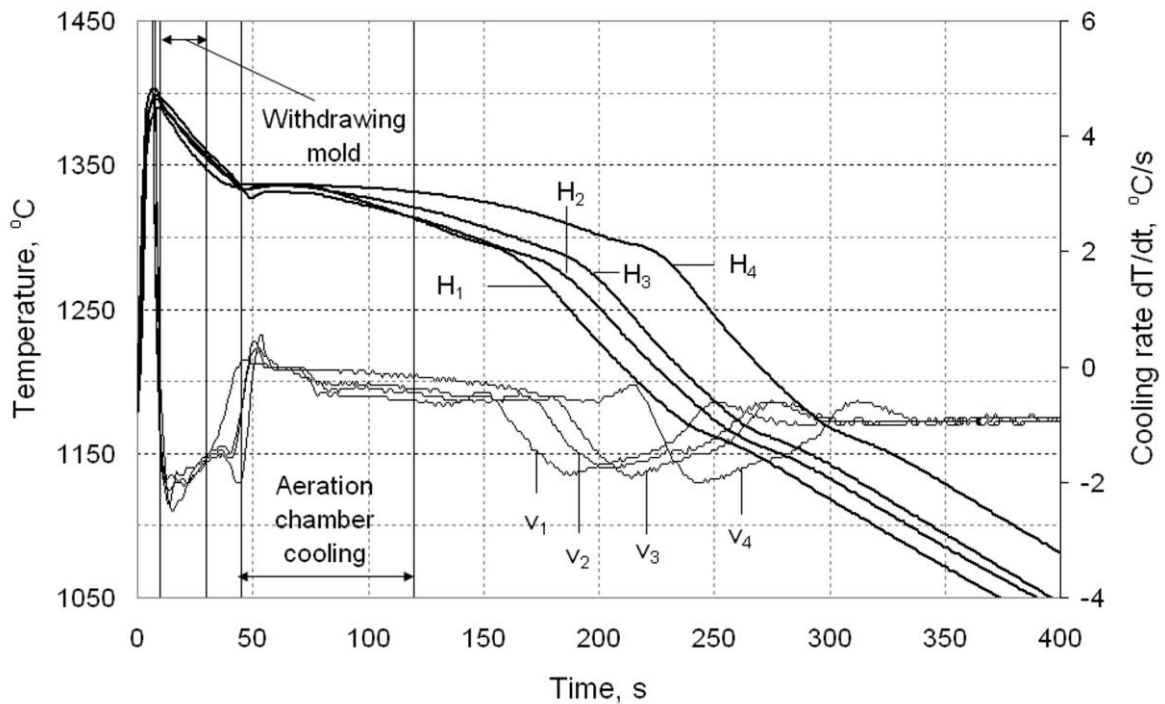


Fig. 5. The temperature - H_1 , H_2 , H_3 i H_4 and the cooling rate - v_1 , v_2 , v_3 , v_4 in the thickened part of the cast depending on the time and location of the chills

The duration of solidification process of the alloy was determined as a time difference between the beginning and the end of solidification process. The authors established the average cooling rate for the alloy - during the solidification process. It was calculated as a ratio of the difference between temperatures of the beginning and the end of solidification process and the solidification time. It has been established that the cooling rate and the duration of solidification process depend on the chills quantity and the cast shape (tab. 3). The highest average cooling rate (-0.6°C/s) was obtained for casts H_1 and H_2 - with two chills. Application of single chill causes the cooling rate of cast H_3 to decrease (to the value of -0.54°C/s). It lengthens also the solidification time.

Table 3.

The solidification time and the average value of cooling rate for the cast, depending on the type and amount of chills

The characteristics of measurement - quantity and type of chills	Solidification time, s	The average value of cooling rate during the solidification process, $^{\circ}\text{C/s}$
H_1	144	-0.6
H_2	162	-0.6
H_3	174	-0.54
H_4	208	-0.39

On the basis of the X-ray examination the authors detected no shrinkage defects in the thickened parts of casts equipped with

chills. In the cast without chills a shrinkage defect was found in the upper the thickened part of the cast.

Casts were submitted to macroscopic examination in order to verify the non-destructive testing and determine the types of shrinkage defects on the cast longitudinal section. The cast shape, the temperature measurement and the location of shrinkage defects were a basis for determining the course of cast solidification process. The analysed cast without chills - H_4 was divided into three characteristic feeding areas: the gating system l_w , the thickened part of the cast l_n and the end zone l_b (fig. 6a). The directional solidification process proceeds in the lower part of the cast as a result of influence of the so-called end zone l_b and the thickened part of the cast l_n which acts as a riser. This kind of conditions during solidification process makes the formation of shrinkage porosity in this area impossible.

Increasing the thickness of the middle part of plate cast results in non-uniform cooling and solidification as well as the formation of shrinkage defects. In the thickened part of the cast, a shrinkage cavity and shrinkage porosity arises (fig. 6b). It is caused by higher cooling rate and solidification rate for the plate cast in comparison to those values for the thickened part of the cast. As the feeding capability of hot spot by liquid metal diminishes the shrinkage cavity is formed.

The shrinkage cavity and porosity, which are localized in the thickened part of the cast, were removed through modification of solidification conditions for this part of the cast. Application of chills resulted in a rise of cooling rate of the thickened part of the cast - H_1 , H_2 and H_3 , in comparison with cast H_4 . Therefore, the thickened part of the cast solidifies in the first instance and afterwards - the regions which are influenced by the chills.

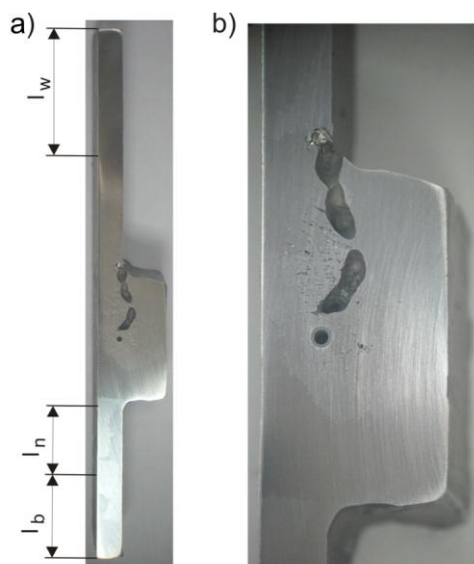


Fig. 6. The longitudinal section of cast and the cast partition into areas of influence (feed) of: the gating system - l_w , the thickened part of the cast - l_n , the end zone l_b (a) and cavity location and shrinkage porosity (b)

It was shown, on the basis of macroscopic examination, that there is a shrinkage porosity in case of casts equipped with chills, above the thickened part of the cast (fig. 7). In the analysed casts the operating range l_o of chills is outside the riser zone - gating system l_w (fig. 7a). The conditions leading to incorrect feed of

interdendritic spaces with liquid metal, coming from the gating system area arise between the influence area of the chill and the gating system. It has been determined that placing the chill on both sides of the thickened part of the cast leads to enlargement of the area of influence, in comparison to the system with one chill. Therefore, the region of shrinkage porosity in the plate cast volume is located at a larger distance from the thickened part of the cast (fig. 7a, b). Also the relative volume of those defects is smaller on the sections of casts with chills placed on both sides.

The method and sequence of pouring liquid metal to the ceramic shell mould has also an influence on its further temperature distribution. In the presented example the liquid metal with the lowest temperature value fills up the lower space of the mould cast. Therefore, it influences the directional solidification process of casts.

Chemical microanalysis didn't prove that there is a change of chemical composition of the IN-713C alloy on the cast surface. No presence of products of reaction of liquid metal with chill material were detected. The tight ceramic layer with a thickness of approximately 1mm prevents the reaction from occurring. The material of this ceramic layer must be characterized by high endurance and thermal conductivity. It should also adhere to the chill surface. Lesser adhesion leads to formation of gap which increase the thermal resistance and decrease the heat flow and the thermal efficiency of the chills. The conducted research led to the conclusion that this layer was of high quality - it adhered strongly to the chill. After melting and performing the preliminary annealing of the wax, no cracks were found.

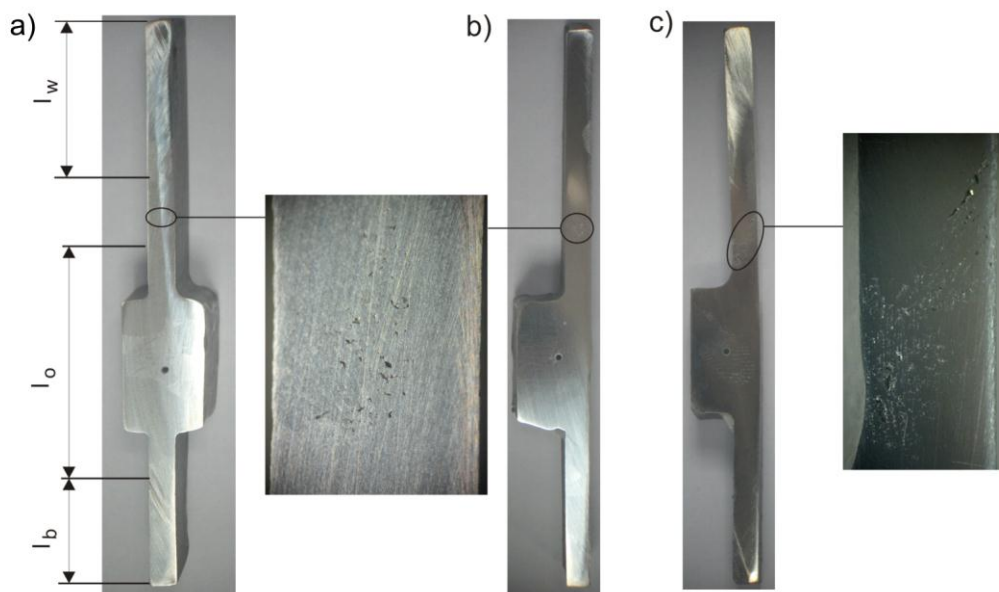


Fig. 7. Three areas of influence, originating from: the gating system - l_w , the chills - l_o and the end zone - l_b in the cast (a). Location of shrinkage porosity on the longitudinal sections of casts equipped with two-sided chills H1 (a) and H2 (b) and one-sided chill H3 (c)

4. Summary

The authors developed a methodology of solidification process control for casts made of IN-713C nickel superalloy with a use of external chills. The chills were made of silicon carbide - SiC. No change in chemical composition of IN-713C alloy on the surface of cast equipped with chill and no reaction of ceramic shell mould with chill material were detected. Basing on the analysis of results of temperature distribution measurement and the assessment of cast solidification process kinetics, it has been established that phase transitions are present during cast cooling. The approximate value of solvus temperature, the temperature at the beginning of solidification process of the γ phase and the eutectic, also at the end of solidification process were determined.

The cooling rate and duration of the casts solidification process depend on the quantity of applied chills and the shape of the thickened parts of the cast. The highest average value of cooling rate - approx. 0.6°C/s - was obtained for the casts with two-sided chills. The application of single chill reduces the cooling rate to the value of approx. -0.54°C/s and increases the solidification time. The cooling rate of the thickened part and the remaining volume of the cast determines the formation of shrinkage defects in those casts. By using chills, it is possible to control the solidification process of the thickened part of the cast and create conditions for proper feed of the remaining cast volume. No shrinkage defects in the areas with larger walls thickness were detected, despite the significant difference in thickness between them and the remaining part of the cast. Introduction of chill from one side of the thickened part of the cast allows to obtain the required cooling rate and create conditions for cast solidification without formation of shrinkage defects. Controlling the solidification process of the casts, characterized by complex shape, with a use of chills, can be insufficient. The application of additional element - thermal insulation of the ceramic shell mould - increases the effect of directional solidification of the casts.

It should be emphasized that the thermal conductivity of silicon carbide chills rises significantly with the decreasing temperature. Therefore, those chills can be utilized for manufacture of precision casts made of alloys which are characterized by lower solidification temperature in comparison to the nickel superalloys.

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