

# DIRECTIONAL SOLIDIFICATION OF CMSX-3 NICKEL BASED SUPERALLOY

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## Introduction

Single crystal nickel based superalloys still belong to the most widely used and reliable materials for the most exposed parts of gas turbines. This is so despite the fact that in the last few years an intensive development of intermetallic compounds, refractory metals and alloys, ceramics and various composite materials for high temperature applications is running.

Directional solidification (DS) is a technology, which enables to produce gas turbine blades and vanes with columnar crystal or single crystal structures. High temperature mechanical properties of single crystal nickel base superalloy castings are better than castings with polycrystalline structures [1]. The aims of producing the blades as single crystals are to eliminate grain boundaries that limit creep rupture properties and ensuring of [001] crystal orientation with minimum of Young's modulus that is suitable for thermal fatigue resistance. The final structure of the castings prepared by DS is influenced by the parameters of the process, therefore it is inevitable to map and control them.

## Theoretical background of directional solidification

The basis of DS process resides in indirection heat discharge during crystallization. The principle of DS is shown schematically in Fig. 1. The ceramic shell mould filled with molten alloy is withdrawn from heated furnace. The biggest cooling effect is oriented on to the bottom part of the mould where appropriate circumstances are created for the nucleus of solid phase and the solidification interface gradually proceeds vertically in its whole mould. The crystals will grow against the direction of the heat discharge.

The condition of the DS is that the solidification interface was macroscopically planar and perpendicular onto the direction growth of the crystal. In microscopical scale the morphology of solidification interface (plane, cellular, dendritic), and at the same time the morphology of the developed structure markedly depends on the thermal gradient  $G$  and solidification rate  $v$ . The alloy will solidify with plane front, if the following inequality is satisfied [2]:

$$\frac{G}{v} > \frac{\Delta T}{D_L} = \left( \frac{G}{v} \right)_c \quad (1)$$

where  $D_L$  is the liquid state diffusion coefficient,  $\Delta T$  is the melting range of alloy and  $(G/v)_c$  is the critical ratio of  $G/v$ . As  $G/v$  becomes progressively less than  $(G/v)_c$  a cellular and then dendritic solidification front develops. The primary dendrite arm spacing in the dendritic structure is directly dependent on the values  $G$  and  $v$ .

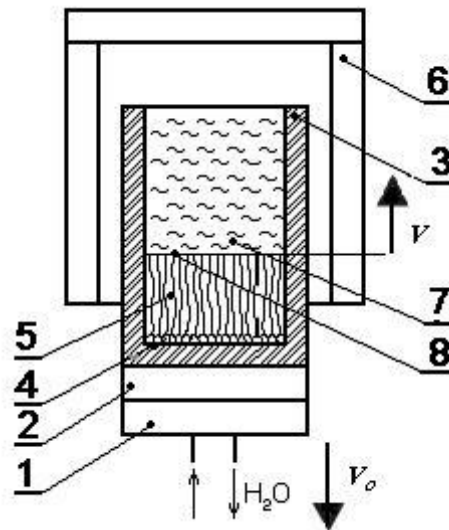


Fig. 1 The principle of the directional solidification process

1- mobile table, 2- water-cooled pad, 3- ceramic shell mould, 4- random oriented crystals, 5- columnar (or single crystal) structure, 6- hot chamber, 7- molten alloy, 8- solidification interface

### Applied process of directional solidification

At the vacuum metallurgy laboratory of the Faculty of Materials Science and Technology Trnava, SUT Bratislava, prof. Žitňanský produced an equipment by rebuilding a vacuum furnace (KOPP VS7) and worked out a method [3] with the help of which the first single crystal castings were produced as early as the mid 80'. The equipment was gradually innovated and the preparation of single crystal castings from several types of nickel based superalloys [4,5] was realised in it. On this experimental equipment cylindrical bars were made  $\phi$  12 x 80 mm from nickel based superalloy CMSX-3, the nominal chemical composition of which is given in Table 1.

Table 1. Nominal chemical composition of the CMSX-3 nickel based superalloy (wt.%)

Cr	Co	Mo	W	Al	Ti	Ta	Hf	Ni
8	4	0,5	9	5,7	0,7	5,7	0,1	Balance

Ceramic shell moulds on the base of  $Al_2O_3$  were used for the casting. Shell moulds were made by the help of wax patterns, a technology of carrying ceramic layers gradually on (Fig. 2). The helix in the ceramic mould acts as a filter and only permits a single grain to pass through. After melting out the patterns the moulds were burnt out. Fig. 3 records the out burnt ceramic shell mould. The mould was placed onto the table surface of the oven cooled by water; it was warmed up to the proper temperature and was filled with molten CMSX-3 alloy. The filled mould was withdrawn from the hot chamber with a constant withdrawing rate  $v_0$ . The schematic illustration of the arrangement of the DS process is shown in Fig. 4. Fig. 5 shows the castings produced by the applied DS process. Every casting contained 6 cylindrical bars  $\phi$  12 x 80 mm. Different techniques were applied for quality monitoring of the cylindrical bars prepared for nickel base superalloys [6] from macroscopic examination, chemical etching, Laue X-ray diffraction to metallographical examinations.



Fig. 2 Vax pattern



Fig. 3 Ceramic shell mould

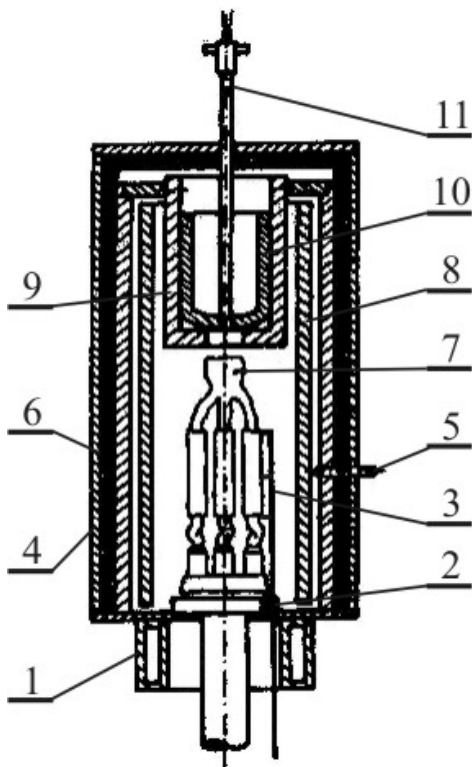


Fig. 4 The applied directional solidification process  
 1- cooler, 2- mobile table, 3- thermocouples,  
 4- covering of hot chamber, 5- measuring the  
 temperature of chamber, 6- thermal isolation,  
 7- ceramic shell mould, 8- heating unit,  
 9- support, 10- melting crucible, 11- measuring  
 the temperature of molten alloy

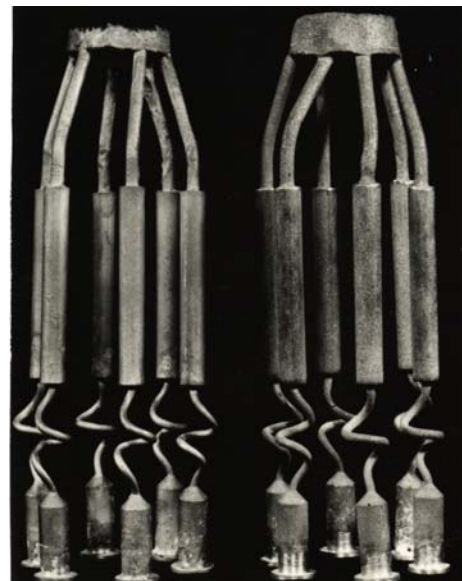


Fig. 5 Directionally solidified  
 castings from CMSX-3 nickel  
 based superalloy

## Measuring the thermal field in the process of directional solidification

Measuring of the thermal field response to setting the temperature along the axis of the cast part during the whole DS process. The temperature was measured in three points along the bar, measured from its bottom end  $L = 0$  mm - temperature 3,  $L = 40$  mm - temperature 2,  $L = 80$  mm - temperature 1. There were three thermocouples Pt30Rh-Pt6Rh placed into the shell mould in the above-mentioned positions in protective ceramic capillars (see Fig. 4). The measurement began at a time  $t = 0$ , when the feed of the table was switched on at a speed:  $v_0 = 2,86 \text{ mm}\cdot\text{min}^{-1}$ . It was finished after the mould was removed from the heated to the cooling zone. The whole measuring time lasted for 68 minutes.

Three cooling curves were created as a function of time,  $T = f(t)$ , from the received T-t points (Fig. 6). The change of the temperature along-the whole bar was obtained by parabolic interpolation of these three points as the temperature by such arrangements of the DS process is characterised by parabolic function [3]. During the process a thermal field was constructed on the casting from these operations – temperature T dependent on time t and the position on the bar L, i.e.  $T = f(t, L)$ . The thermal field of the casting from the alloy CMSX-3 is graphically represented in Fig. 7.

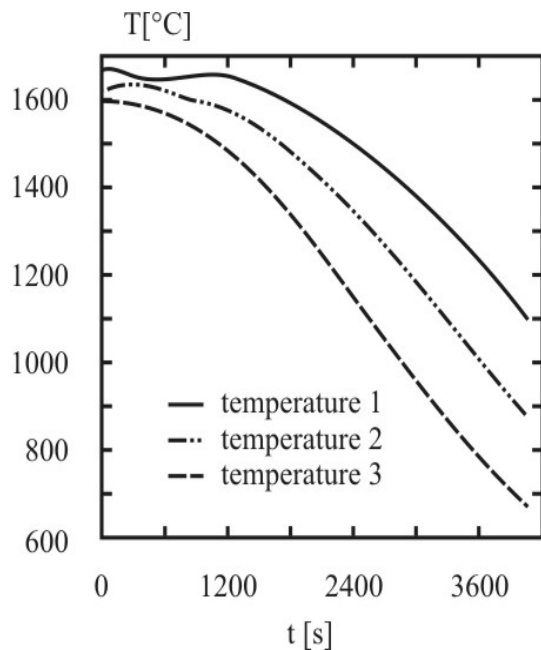


Fig. 6 Cooling curves in three points of the casting

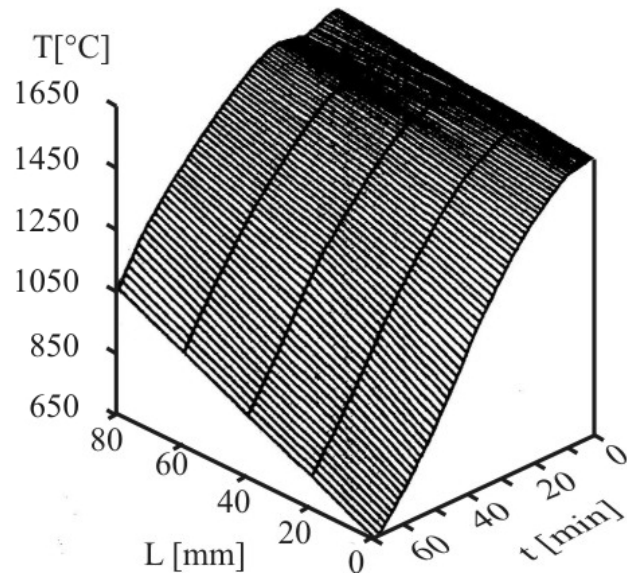


Fig. 7 Thermal field on the cylindrical casting of the CMSX-3 alloy

## Results and discussion

Thermal gradient  $G$ , solidification rate  $v$  and cooling rate  $v_T$  belong to the those main parameters which characterise the DS process. The temperature of solid-liquid (S-L) interface is  $1350 \text{ }^\circ\text{C}$  [7]. By analysing the thermal field for a chosen isotherm ( $T_{IS} = 1350 \text{ }^\circ\text{C}$ ) the following calculations were made in 26 points alongside the bar [7]:

- arrival time of isotherm  $T_{IS}$  into the appropriate point  $t$  [min]
- solidification rate (rate of S-L interface)  $v = \frac{dx}{dt}$  [mm·min<sup>-1</sup>] (2)
- cooling rate  $v_T = \frac{\partial T}{\partial t}$  [K·min<sup>-1</sup>] (3)
- thermal gradient  $G = \frac{\partial T}{\partial x}$  [K·mm<sup>-1</sup>] (4)

The determined values of the parameters, respectively their intervals represented by the applied DS process are shown in Table 2.

Table 2

The values of the crystallization parameters of the applied directional solidification process

Cooling rate $v_T$ [K·min <sup>-1</sup> ]	Solidification rate $v$ [mm·min <sup>-1</sup> ]	Thermal gradient $G$ [K·mm <sup>-1</sup> ]
16,5 - 12	4,9 – 2,7	5 – 3,7

Resulting from the analysis of the data we can state that by the applied process and by the withdrawal rate of mould  $v_o = 2,86$  mm·min<sup>-1</sup> the crystallisation process has begun at time  $t = 28$  min and was finished at  $t = 53$  min with a cooling rate 16,5 and 12 K·min<sup>-1</sup>. A faster cooling rate was around the bottom part of the casting (position  $L = 0$  mm), on the upper part of the casting the cooling rate was the slowest (position  $L = 80$  mm). The size of the thermal gradient and the solidification rate changed also according to the particular position along the stick. The quantitative dependence of the crystallisation parameters corresponds to data published earlier [8].

The registered values of the thermal gradient  $G$  and solidification rate  $v$  are data which can be found in conventional single crystal superalloy solidification range [9] and the following is applied for them:  $1,8 < G < 10,2$  K·mm<sup>-1</sup> and  $0,6 < v < 6$  mm·min<sup>-1</sup>. Exactly these two parameters of the DS process affect considerably the morphology of the structure of the produced single crystal castings.

The structure of the casting prepared by the DS process with the above-mentioned parameters is single crystal consisting of columnar dendrites the orientation of which is quasi equivalent with the progress of the thermal gradient. In Fig. 8 the microstructure of the transverse section of the directionally solidified bar is shown. The structure consists of dendrite crystals with four secondary arms. The average primary dendrite arm spacing was  $l = 225$  μm. Quested and McLean [10] were among the first who published the dependence between the formation of the particular morphological structure, the thermal gradient and the solidification rate (Fig. 9) in the case of nickel based superalloys. We have drawn our measured values  $G$  and  $v$  into that diagram (we considered average values  $G = 4300$  K·m<sup>-1</sup> and  $v = 6,3 \cdot 10^{-5}$  m·s<sup>-1</sup>). It can be seen from the position of the point that columnar dendritic structure is expected. We have proved this by metallographical analysis.

Changing the parameters of the DS process has a significant influence on the primary dendrite arm spacing. Quested and McLean indicated [10] that for DS polycomponent systems, like the nickel based superalloys, the model of Hunt [11] can be applied. On the basis of this model it can be concluded that the dendrite arm spacing  $l$  depends linearly on the

expression  $v^{-1/4} \cdot G^{-1/2}$ . This relationship is presented in Fig. 10 for three types of nickel based superalloys [10] which we completed with the measured values for CMSX-3.

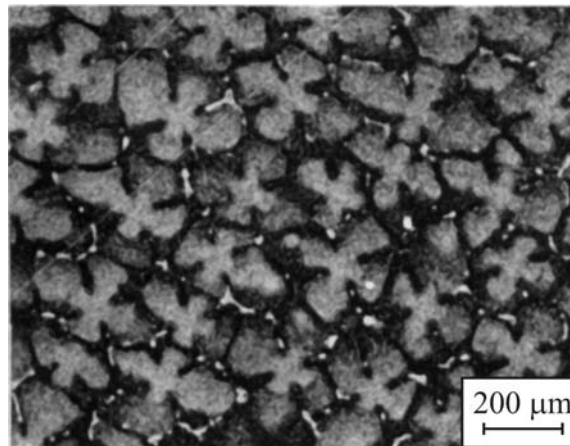


Fig. 8 Microstructure of directionally solidified CMSX-3 nickel based superalloy, transverse section, optical microscopy

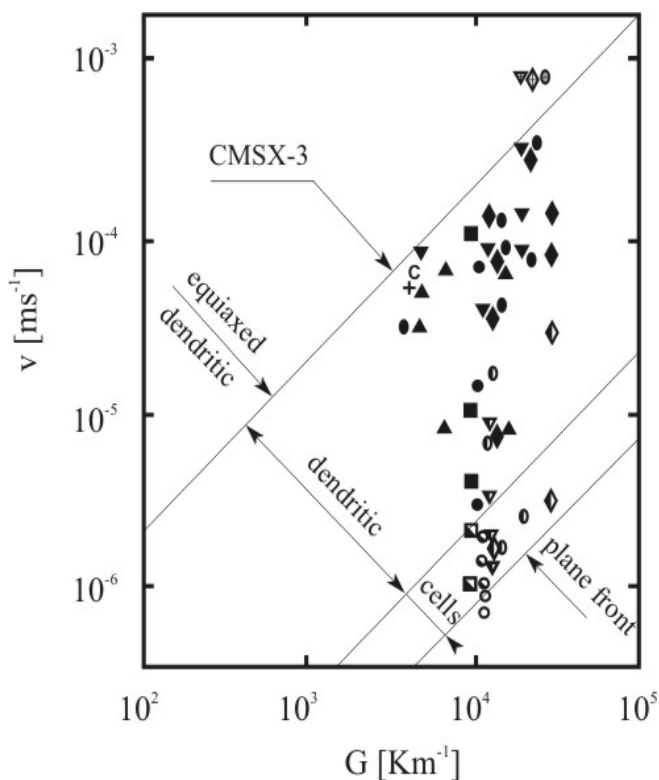


Fig.9 The solidification morphologies for directionally solidified superalloys plotted as function of solidification rate  $v$  and thermal gradient  $G$  [10]  
 $\nabla$  - MAR-M246,  $\circ$  - IN-713C,  
 $\square$  - MAR-M200  $\triangle$  - IN-100,  
 $\diamond$  - MAR-M002  $\circ$  - IN-738LC

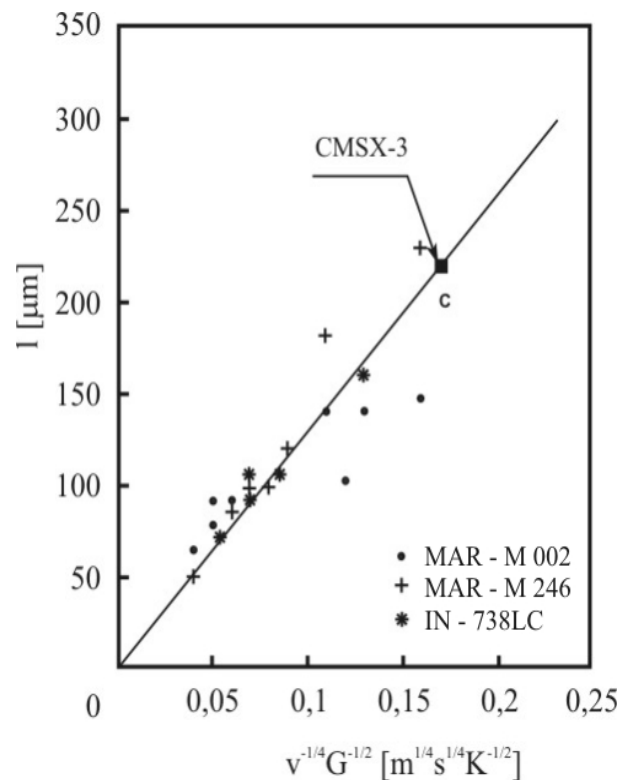


Fig. 10 Primary dendrite arm spacings as a function of  $v^{-1/4} \cdot G^{-1/2}$  for three directionally solidified superalloys [10]

The primary dendrite arm spacing can be reduced by increasing the thermal gradient as a result of intensive cooling. It is possible to utilise even the increase of the solidification rate by changing the withdrawing rate of mould, however the solidification interface might be destroyed easily and an equiaxed dendritic crystallization occurs in this case.

Generally it can be stated that a fine dendrite structure gives better mechanical properties at lower temperatures, however the castings prepared by DS are almost always used in heat treated conditions [12], which ensures the homogenisation of the microstructure and a structural state that leads to optimal properties at high temperature services.

## Conclusion

The applied parameters of the DS process allow the production of cylindrical castings with single crystal structure from the CMSX-3 alloy. The applied method of measuring the thermal field during the DS process enabled us to determine the thermal gradient  $G$  and the solidification rate  $v$ , which lead to the formation of columnar dendritic structure. The main quantitative parameter of the primary dendrite arm spacing structure depends linearly on  $v^{-1/4} \cdot G^{-1/2}$ . Heat treatment is needed to homogenise the microstructure and to ensure the optimal high temperature properties of the castings from CMSX-3 alloys.

## Acknowledgements

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