STRUCTURE OF THE NI-BASE SUPERALLOY IN 713C AFTER CONTINUOUS CASTING

F. Zupanič, T. Bončina, A. Križman

University of Maribor, Faculty of Mechanical Engineering, Slovenia
Smetanova 17
SI-2000 Maribor, Slovenia

Abstract

In this work a Ni-base superalloy IN 713C was continuously cast in the form of rods with a circular cross section under a protective argon atmosphere using a vertical continuous caster. The structure of the rods cast at different casting parameters was investigated using different microstructural characterisation techniques. It was found that superalloy IN 713C could be continuously cast over a wide range of casting parameters. The continuously cast rods possessed a clean surface and excellent internal integrity without non-metallic inclusions and porosity. The microstructure consisted, regardless of the casting parameters used, of γ-grains, interdendritic γ/MC eutectic and coherent γ' precipitates distributed uniformly within the γ-matrix. The size, distribution, morphology and orientation of microstructural constituents depended strongly on local solidification conditions, which were greatly influenced by the nozzle/mould set up and the alternating drawing mode of continuous casting. The results of the work strongly suggest that IN 713C could also be continuously cast successfully using large-scale industrial equipment.

Superalloys 2000
Edited by T.M. Pollock, R.D. Kissinger, R.R. Bowman,
K.A. Green, M. McLean, S. Olson, and J.J. Schirra
TMS (The Minerals, Metals & Materials Society), 2000

Introduction

Continuous casting offers several advantages over the conventional casting of semi-finished metal products including lower production costs and higher productivity rates, as well as better surface quality and the excellent internal integrity of the products [1]. It has been widely used in the production of almost all alloys, but not yet in the manufacture of vacuum melted cast Ni-base superalloys [2]. It has the potential to replace conventional casting of remelting stick, which has high cost and labour intensive stages in the production of superalloys, although a large capital investment is unavoidable at the beginning. A further drawback for the introduction of continuous casting in the manufacture of superalloys arises from a lack of knowledge about the behaviour of particular superalloys when continuous cast. In order to answer the latter question, we initiated an investigation with the following objectives:

1) Evaluation of the castability of selected Ni-base superalloys in continuous casting using laboratory scale equipment

2) Finding a correlation between the adjustable casting parameters and the as-cast microstructure

3) Optimisation of the continuous casting parameters to obtain the best combination of production rate, process reliability and product quality

4) Determining the influence of the as-cast microstructure on the subsequent heat treatment stages, final microstructure and mechanical properties of the continuously cast products

5) Obtaining mostly unidirectional crystallisation of the Ni-base superalloys in a continuous caster.

So far, we have concentrated mainly on the first three objectives and have continuously cast with success three Ni-base superalloys. The aim of the present work was to carry out a characterisation of macro- and microstructure to evaluate the influence of casting parameters on the formation and development of microstructure during continuous casting. This information is critical in further optimisation of the casting parameters to obtain the desired goals.

The nickel-base superalloy IN 713C has been used for decades. Its application is in the form of precision cast parts for hot-end turbocharger wheels. It is interesting to note that the required properties are already attained normally in the as-cast condition, and therefore no heat treatment is necessary. The influence of casting parameters on the as cast microstructure of IN 713C was thoroughly investigated by Bhambri et al [3]. One of the most important reasons to experimentally continuously cast IN 713C lies in the fact that the annual production rate of this alloy is relatively high. In addition, the alloy possesses considerable high-temperature ductility and strength [4], which are usually an important factor in reliable continuous casting.

Experimental procedure

Continuous casting experiments were carried out using a pilot scale set-up consisting of a vacuum induction melting furnace (Leybold Heraeus IS 1.5) and a vertical continuous caster (Technica Guss; now SMS Meer, Demag Technica). Figure 1 a shows schematically the most important parts of the equipment. The alloy was melted in an alumina crucible. It was separated from the water-cooled Cu-Be mould by a ZrO_2 inlet nozzle. It is important to notice that the inner diameter of the ZrO_2 inlet nozzle (9.7 mm) was slightly smaller than the inner diameter of the Cu-Be mould (10 mm). It will be shown later that this feature greatly influenced the appearance of the rod and the formation of macroscopic in-homogeneities.

Table I Chemical composition of the investigated alloy IN 713C*

Fe	Cr	Al	Ti	Nb	Mo	C	Ni
1.42	13.15	6.04	0.78	2.11	4.19	0.15	Rest

* all compositions in this work are given in weight percents unless otherwise stated

Table II Conditions of continuous casting experiments

No. of a casting condition	#1	#2	#3	#4
Length of draw [mm]	5	7	8	8
Relative velocity of the drawing stroke [%]	30	35	35	35
Resting time [s]	0.15	0.15	0.15	0.10
Length of the reverse stroke [mm]	0.30	0.30	0.30	0.30
Relative velocity of the reverse stroke [%]	10	10	10	10
Casting speed [mm/s]	8.33	10.17	11.17	12.33
Melt temperature [°C]	1450	1420	1400	1420
Water flow rate [l/min]	20	20	30	30
Inlet water temperature [°C]	30	38	38	37
Outlet water temperature [°C]	30	43	43	43

In a typical trial around 14 kg of IN 713C, the composition of which is given in Table 1, was melted in the induction furnace in a vacuum of approximately 10^{-2} mbar. After heating up to 1420 °C the melt was continuously cast under a protective argon atmosphere at 1.030 bar through the Cu-Be mould, in which solidification occurred. The solid rod was pulled out of the mould using the predetermined "alternating drawing mode" (Fig. 1 b).

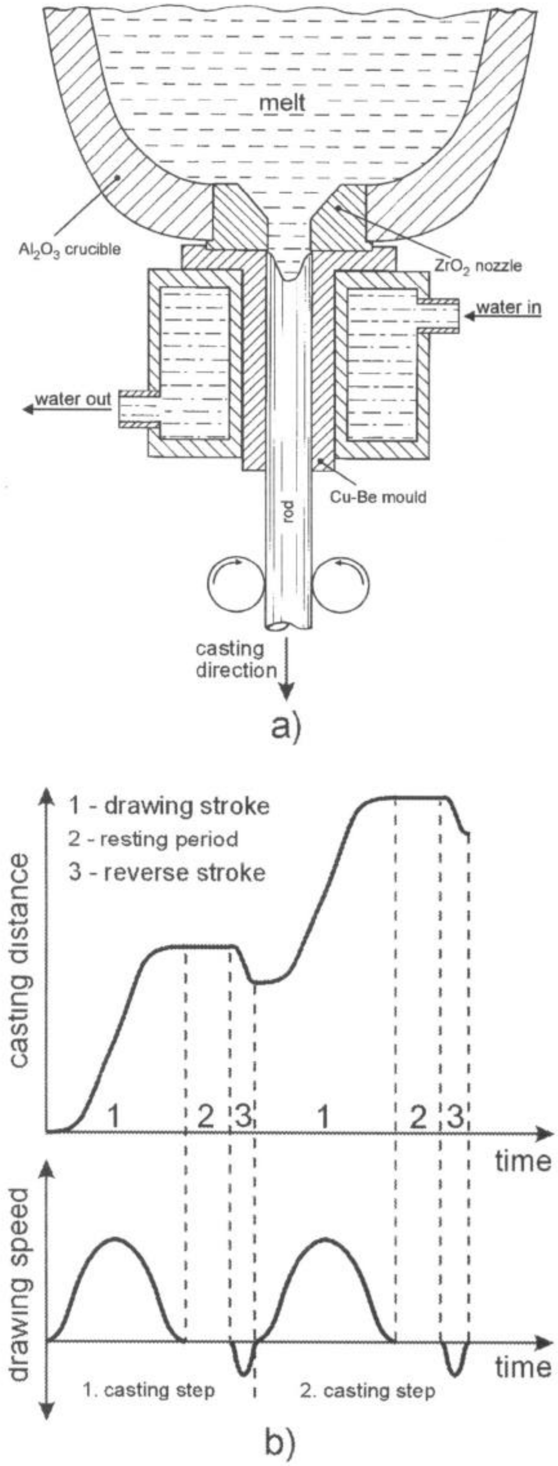

Figure 1: Schematic presentation of the continuous casting set-up (a); and the characteristics of the alternating drawing mode (b)

It consisted of three sequential stages: the drawing stroke, the resting period and the reverse stroke, which formed a casting step. It can be seen that during the drawing and the reverse stroke, the velocity increased from rest to the maximum velocity and decreased back to rest. During the reverse stroke the rod was moved in the direction opposite to the casting direction. This drawing mode is usually used when an alloy is continuously cast for the first time because it allows the most reliable continuous casting. Besides, the reverse stroke eliminates possible sticking between the bar and the Cu-Be mould.

During the trial, the length of draw was changed from 5 to 8 mm, the resting time between 0.10 and 0.15 s, but the length of the reverse stroke was always 0.3 mm. Different combinations of these parameters gave an average casting speed ranging from 8.33 to 12.33 mm/s. Details are given in Table II.

The continuously cast rods were prepared using standard metallographic procedures and investigated by different microstructural characterisation techniques, such as light (LM), scanning (SEM) and transmission (TEM) electron microscopy, and energy dispersive X-ray spectroscopy (EDS).

Results and discussion

Surface appearance of continuously cast rods

Continuous casting of the superalloy IN 713C took place without any breakouts of the rod or other deviations. Figure 2 shows some continuously cast rods. Their surface is bright, with a characteristic metallic shine. It is obviously free of thick oxide layers. Therefore subsequent grinding of the rods or even shot blasting, which are typical steps in the present production of remelting sticks, is unnecessary. Periodic markings on the surface are the consequence of the "alternating drawing mode". This is confirmed by the fact that the distance between them coincides nicely with the length of the casting step; which is defined as the difference between the length of draw and the length of reverse stroke. The diameter of the continuously cast rod at these markings is around 9.7 mm and thus smaller than in the other parts of the rod. It is suggested that there must be some relationship between it and the diameter of the inlet nozzle, which is also 9.7 mm.

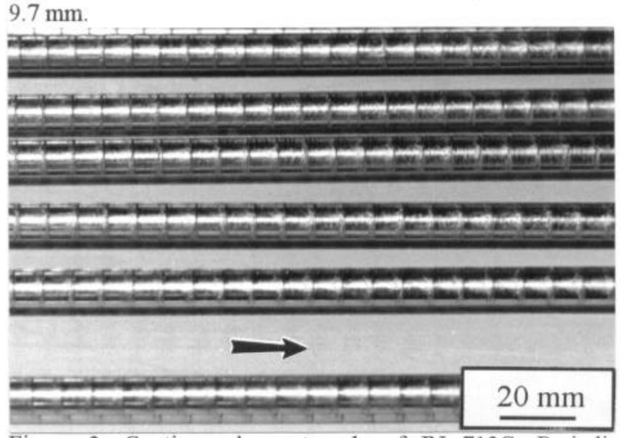

Figure 2: Continuously cast rods of IN 713C. Periodic markings are a result of the "alternating drawing mode"

Microstructural constituents in the continuous cast IN 713C

Metallographic analysis revealed that irrespective of the casting conditions the microstructure of the alloy consists of γ-grains with dendritic growth morphology, the MC/γ eutectic and coherent γ' precipitates distribution in the γ matrix are more or less uniform. On the other hand, it was observed that the size, distribution, morphology and orientation of these microstructural constituents depend on casting conditions. The γ/γ' eutectic, which was observed by Bhambri et al [3] in directionally solidified IN 713C, is not present in continuously cast rods. As a result of the alternating drawing mode a periodic pattern has formed with a wavelength almost equivalent to the length of a casting step (Fig. 3).

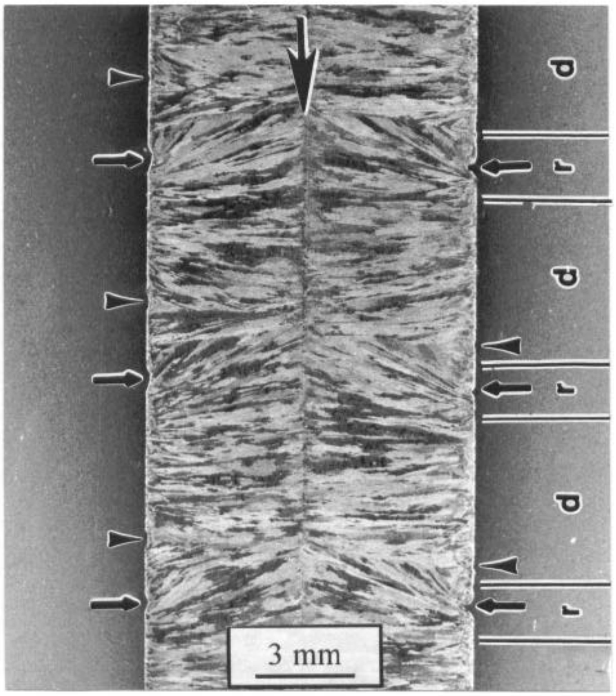

Figure 3: Macrostructure of a continuously cast IN 713C superalloy (r – the resting period and the reverse stroke, d – the drawing stroke). Casting conditions: the length of draw: 7 mm, the resting time: 0.15 s, the length of the reverse stroke: 0.3 mm, the average casting speed: 10.17 mm/s. The largest arrow indicates the casting direction

Macrostructure

Fig. 3 also shows the size and orientation of the γ grains. The γ grains are usually columnar and oriented in the radial direction. They must have formed on or very close to the mould wall and then grown toward the centre of the rod. In addition to columnar γ grains, a shallow layer of small equiaxed grains is present on the edge of the rod, and some equiaxed grains have also formed at the centre of the rod at the highest casting speed of 12.33 mm/s.

Within a casting step the orientation of γ grains changed from almost ideally perpendicular to the mould wall, towards orientations which were more closely aligned with the casting direction. The latter grains in fact grew in the opposite direction to the casting direction. It is likely that γ grains being perpendicular to the mould wall formed and grew during the drawing stroke, whereas the γ grains inclining to the cast direction grew during the resting period and the reverse stroke.

The periodic markings observed on the surface of the rod could be seen on the longitudinal cross sections in the form of large intrusions. They were positioned next to γ grains that were inclined to the casting direction; this clearly indicates that they must have formed during the resting period and the reverse stroke.

Besides these large intrusions we also observed smaller and less periodic intrusions (indicated by arrows ►). They were usually positioned close to the transition from the resting/reverse stroke and the drawing stroke. Their probable origin will be discussed later.

The drawing stroke

Despite the fact that the drawing velocity was changing all the time throughout the drawing stroke, the microstructure formed during the drawing stroke exhibited the same features over the whole drawing length. At this stage of continuous casting the melt was able to come into direct contact with the water-cooled Cu-Be mould. The heat was mostly extracted through the mould wall. This facilitated crystal grains having an [100] axis perpendicular to the mould wall to outgrow other less favourably oriented grains. Despite Bhambri et al. [3] claiming that the solidification of IN 713C should start with the primary crystallisation of MC carbide, the solidified microstructure shows characteristics, which are typical for primary crystallisation of the γ phase. It seems that carbides are present mainly within the MC/γ eutectic constituent in the interdendritic region.

Figure 4 compares microstructures formed at different distances from the edge of the rod during the drawing strokes. It is evident that microstructure is becoming coarser when moving toward the centre of the rod; the primary and secondary dendrite arm spacings are increasing, as well as the size and the amount of MC carbides. Close to the edge of the rod the MC carbides appear as separate particles almost parallel relative to the dendrite trunks, whereas in the middle of the rod they are present as a constituent of large MC/γ eutectic pools in the interdendritic region.

Bhambri et al. [3] determined the empirical relationship between the secondary dendrite arm spacing λ_2 and the local solidification time t_f:

$$\lambda_2 = At^n \quad (1)$$

with $A = 6.79 \times 10^{-6}$ and $n = 0.43$. Using equation (1) and taking into account that the solidification range of IN 713C amounts to approximately 60 °C, we calculated local cooling rates on the basis of measured secondary arm spacings λ_2

(Table III). It can be seen that the local solidification rate $\dot{T}$ at a distance of 0.5 mm from the edge was 150-250 °C/s and ~20 °C/s at the centre of the rod. It is interesting to note that only minor differences were observed between different casting conditions.

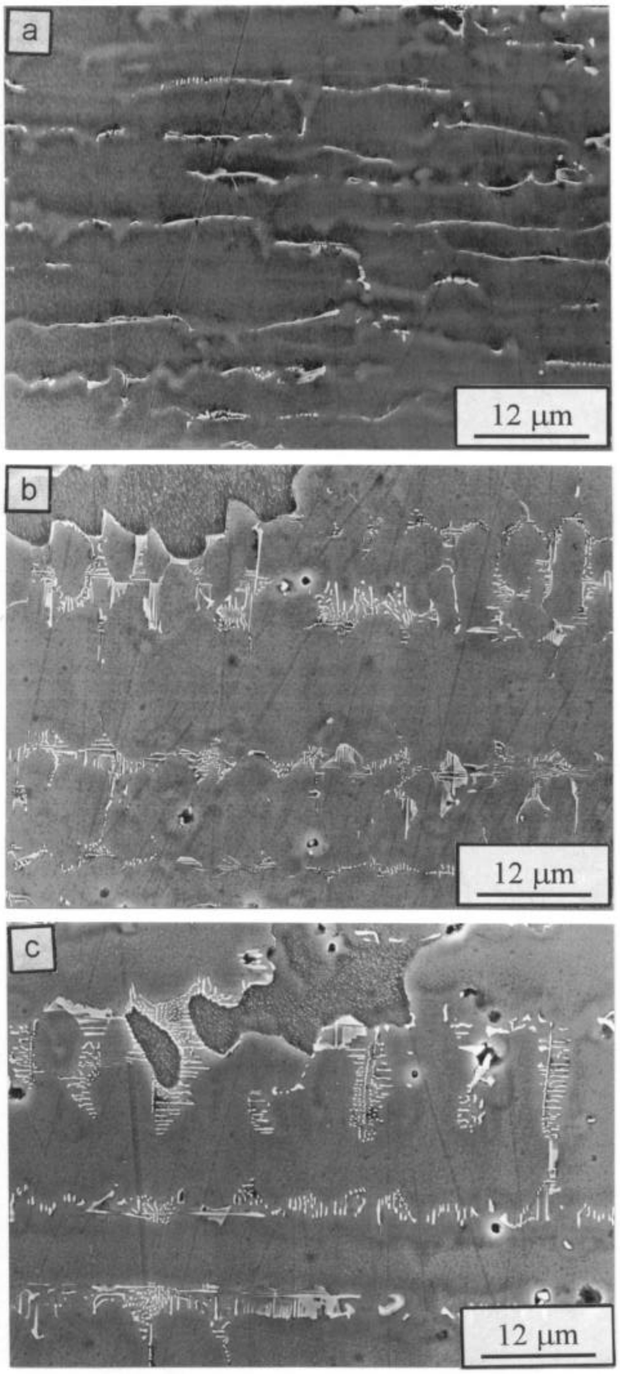

Figure 4: Microstructure of a grain growing perpendicular to the casting direction at different distances from the edge of the rod: a) 0.5 mm, b) 2.5 mm and c) 5 mm. (SEM, casting conditions #1). Dark γ-matrix, bright MC carbides

Table III: Secondary dendrite arm spacing λ_2 and estimated cooling rates $\dot{T}$

		#2			#4		
		0.5 mm	2.5 mm	5 mm	0.5 mm	2.5 mm	5 mm
perpendicular	λ_2 [μm]	4.5	6.6	10.6	3.7	6.1	10.1
grains	$\dot{T}$ [°C/s]	156	64	21	246	77	24
inclined	λ_2 [μm]	5.2	6.2	10.7	5.8	7.7	11.3
grains	$\dot{T}$ [°C/s]	112	74	21	87	45	18

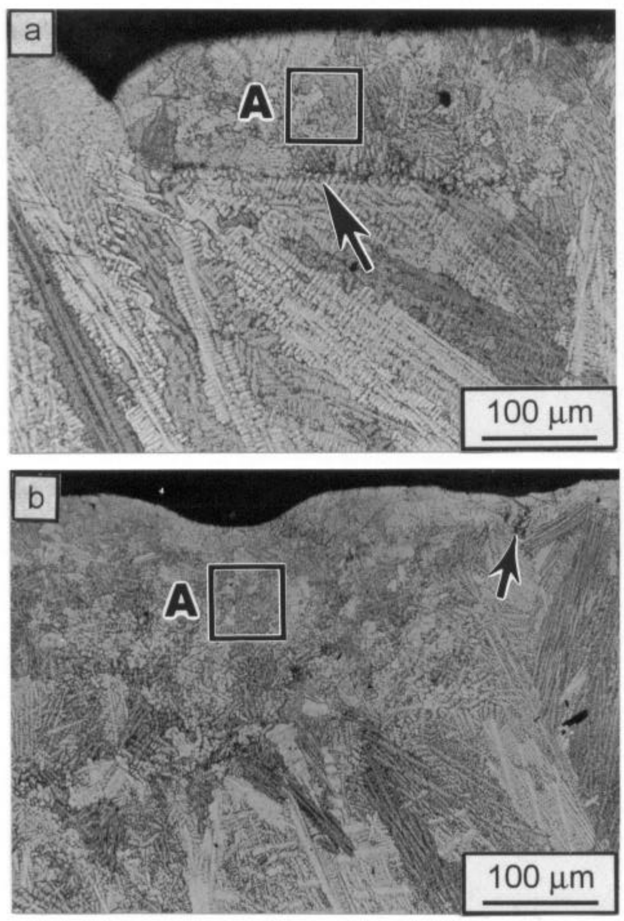

Figure 5: Light-optical micrographs of the region around a large intrusion, which have formed during the resting period and the reverse stroke a) casting conditions #1, b) casting conditions #4

The resting time and the reverse stroke

Less uniform microstructure has formed during the resting period and the reverse drawing stroke. Figure 5 shows this part of the rod at the smallest (conditions #1, 10.33 mm/s) and at the highest (conditions #4, 12.33 mm/s) casting speed. Just below the large intrusion in Fig. 5 a, we can observe that reorientation of γ grains (dendrite trunks) started to occur. This suggests that (1) in the course of the resting period and the reverse stroke the melt was not able to come into direct contact with the Cu-Be mould and (2) that the heat extraction direction had changed from being perpendicular to the mould wall towards the direction almost aligned with the rod axis. This could only happen if γ grains had started to grow into the inlet nozzle, which prevented dissipation of heat in the radial direction. Thus the relative amount of heat conducted in the axial direction increased considerably. In Fig. 5 a dendrite marked by the arrow is almost ideally aligned with the casting direction. It is approximately 150 μm from the rod edge. This clearly indicates that it must have grown along the inner wall of the inlet nozzle.

Primary and secondary dendrite arm spacing in the inclined grains are slightly larger than those of the perpendicular grains (Fig. 6 and Table III). It could therefore be inferred that cooling rates obtained during the resting time and the reverse stroke were smaller than those during the drawing stroke.

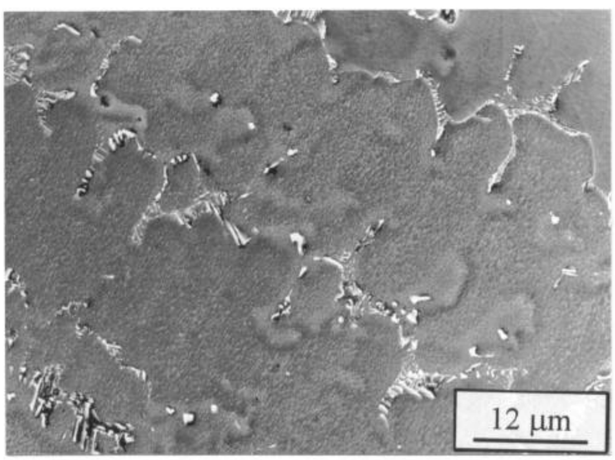

Figure 6: Microstructure of a grain growing inclined to the casting direction at a distance of 2.5 mm from the edge of the rod (SEM, casting conditions #1)

As a consequence of the growth of γ grains into the inlet nozzle and the fact that the inner diameter of the inlet nozzle was smaller than the diameter of the rod, a step has formed on the surface of the rod in the course of the resting time and the reverse stroke. At the initial stage of the following drawing stroke metallostatic pressure tried to force melt into the gap between the mould and the already solid shell. From Fig. 5 a it can be seen that the large marking is a result of incomplete filling of the mould; this means that the melt had solidified before completely filling the gap. With these processes, uniformly spaced large intrusions have formed. The microstructure formed in the gap consists of equiaxed γ grains with a much larger amount of MC/γ eutectic than in the other parts of the rod (Fig. 7 a). The latter observation indicates the melt which penetrated into the gap was already enriched by positively segregating elements; for instance Nb, C, Ti and Mo.

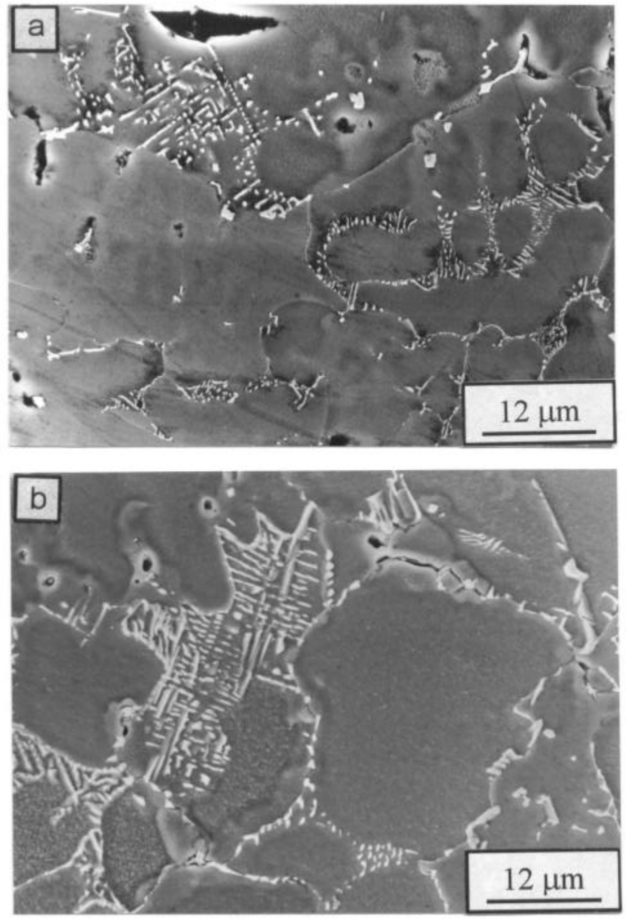

Figure 7: Scanning electron micrographs of a) the region marked by A in Fig. 5a, and b) the region marked by A in Fig. 5b

At casting speeds larger than 11.17 mm/s we were not able to observe dendrites growing parallel to the rod axis (along the inner wall of the inlet nozzle). On the other hand, the region of equiaxed grains next to large intrusions was considerably thicker than at casting conditions #1 and #2. This can be explained if we suppose that the temperature gradient in the liquid before the solid liquid interface increases with the growing casting speed. This would mean that at the beginning of the drawing stroke much hotter melt penetrates into the gap between the mould wall and the solid shell, causing partial remelting of the already solid rod in the vicinity of a large intrusion. Much coarser γ grains and γ/MC eutectic indicate smaller cooling rates compared to the casting conditions #1 and #2 (compare Fig. 7 a and b).

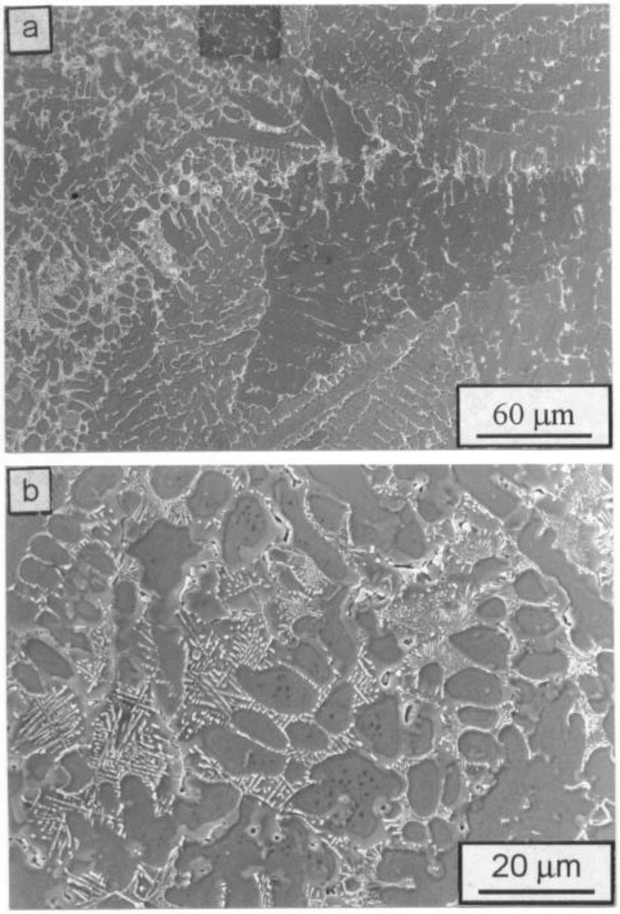

Figure 8: Scanning electron micrographs of a region under a shallow intrusion in Fig. 5a

Hot tearing

In Fig. 5 b we can observe a smaller intrusion, which is of the same type as those indicated by the smaller arrows in Fig. 3. Such intrusions are usually positioned at the beginning of the drawing stroke. With the small intrusion, a larger amount of MC/γ eutectic is present than elsewhere. This eutectic-rich band protrudes some 0.5 mm from the surface of the bar. It is believed that it forms because of hot tearing – formation of cracks by separating grains along their grain boundaries in the mushy zone. The occurrence of hot tearing can be explained as follows. Stresses arising from the acceleration of the rod during the initial stage of a drawing stroke resulted in a stretching of the thin solid shell and the separation of γ grains along the grain boundaries in the crack sensitive mushy zone. The melt enriched with the positively segregating elements filled the crack, and a eutectic band following the initial crack path has formed. Under the casting conditions applied, the appearance of hot tearing did not cause breakouts, but it was observed that the length of the cracks was increasing with casting speed, indicating that there exists a maximum casting speed, above which the reliability and safety of the continuous casting of IN 713C may be seriously endangered. The possibility of increasing production rate without decreasing safety and reliability would probably be a decrease of the acceleration at the beginning of the drawing stroke. This and other possibilities are to be investigated in our future work.

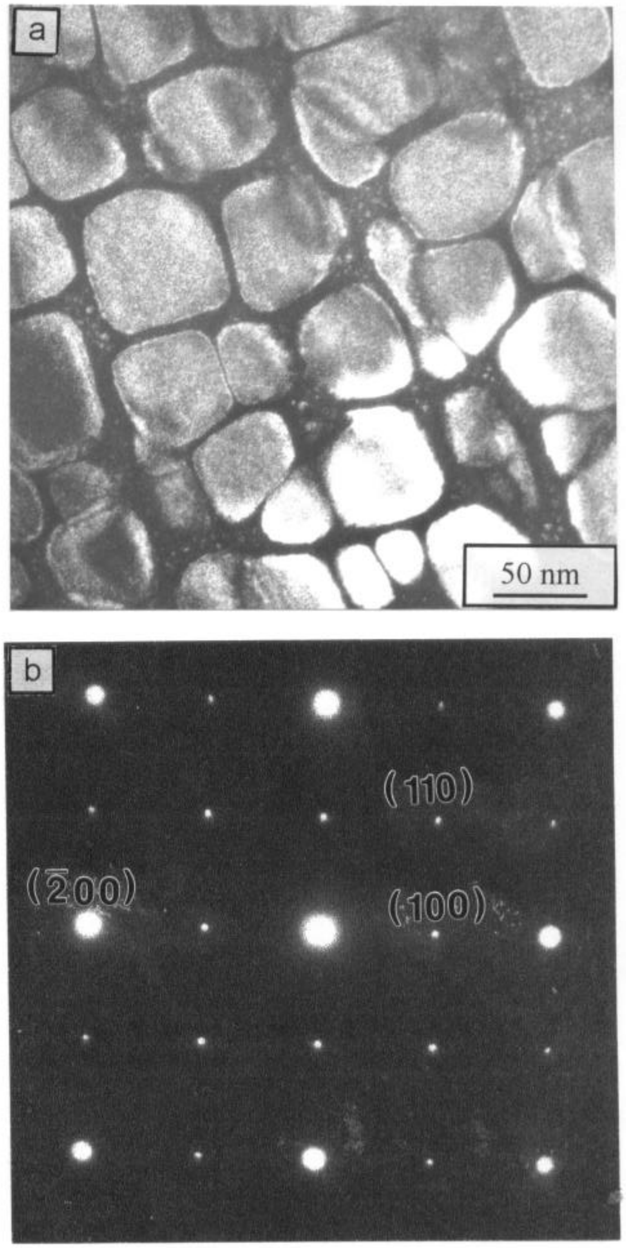

Figure 9: Transmission electron micrograph (a); and its selected area diffraction pattern (b). Small spots: a γ' precipitate, large spots: γ matrix.

Precipitation of γ'

The precipitation of γ' precipitates took place during cooling at temperatures under the γ'-solvus. So far, only a preliminary investigation has been carried out. The results indicate that duplex-sized γ' precipitates are present in the γ matrix (Fig. 9a). The average size of the larger population is around 60 nm, and the average size of the smaller population is around 5-10 nm. It is believed that the larger population has formed at higher temperatures and the smaller ones at lower temperatures. SADP (Selected Area Diffraction Pattern) of a large precipitate showed that it is coherent with the γ matrix (Fig 9 b), as usual in Ni-base superalloys [5]. Further work regarding this topic is in progress and will be reported later.

Conclusions

The results of the investigation show that a Ni-base superalloy IN 713C can be continuously cast over a wide range of casting parameters. The behaviour of IN 713C during the continuous casting of a small circular cross section in a vertical continuous caster strongly suggests that it would also be convenient for continuous casting using large-scale industrial equipment.

The continuously cast rods possessed a clean surface and excellent internal integrity. The microstructure of continuously cast rods consisted, regardless of the casting parameters used, of γ–grains, interdendritic γ/MC eutectic and coherent γ' precipitates distributed uniformly within the γ-matrix. The size, distribution, morphology and orientation of microstructural constituents depended strongly on local solidification conditions, which were greatly influenced by the nozzle/mould set up and the alternating drawing mode of continuous casting. The macrostructure changes periodically, and the wavelength of periodicity corresponds to the length of the casting step. According to the results of alloy characterisation some changes regarding the nozzle/mould set up and the alternating drawing mode will be necessary to attain all the goals of the investigation.

References

1. E. Herrmann, D. Hoffman (eds), Handbook on Continuous Casting, (Aluminium-Verlag Düsseldorf 1980), V-VI

2. "INVESTMENT CASTING - IMPROVED QUALITY FOR SUPERALLOY BAR STOCK - Ross & Catherall combines vacuum induction melting with continuous casting", Foundry Trade Journal, 173 (1999), 3555, B, 52 / 4

3. A.K. Bhambri, T.Z. Kattamis, J.E. Morral: "Cast Microstructure of Inconel 713C and its Dependence on Solidification Variables", Metallurgical Transactions B, 6B, (1975), 523-537

4. Alloy Digest, Data on world wide metals and alloys, Inconel 713C, February, 1959

5. J.R. Davies (ed.), ASM Specialty Handbook, Heat Resistant Materials, ASM International, (1997), Metallurgy, Processing and Properties of Superalloys, 221-254

Acknowledgements

Persons at the National Centre for HREM at Delft University of Technology are gratefully acknowledged for investigations by transmission electron microscopy. We also thank Derek Hendley from Ross & Catherall, Sheffield, UK for providing the alloy and useful advice regarding the melting procedure.