## Single-Crystal Casting of Nickel-Base Superalloys by Directional Rapid Solidification

R. E. SHALIN - V. A. PANKRATOV - Scientific production amalgamation "VIAM" Moscow

## Abstract

Despite its technological difficulties, single-crystal casting is becoming a method of choice for the production of critical components, especially gas turbine blades. Its initial requirement is directional solidification (DS) to eliminate crystal nucleation before the dendrite front. Most DS processes, however, have a low temperature gradient on the isoliquidus front (G < 10 k/m or even 3-5 k/m), and are associated with several drawbacks: formation of a coarse dendritic structure, zone liquation, with freckles, porosity, etc. The quality of a DS alloy is also dependent on the solid-liquid phase interface.

The paper describes DS at different G and growth rate values of a Ni-base superalloy with melt physical characteristics-solidification interval  $T = 70^{\circ}$ C. It was found that high-gradient DS with fast, liquid-metal cooling, called rapid DS (RS), gave an excellent interface, and eliminated freckles and Mg surface carbides. Use of RS in a unit producing 144 turbine blades/day in three shifts is also reported. A 4 to 10-fold increase in service life was achieved.

## Riassunto

La fusione a cristalli singoli, seppur non priva di difficoltà tecniche anche notevoli, sta proponendosi sempre di più quale procedimento di elezione per la produzione di componenti critici, soprattutto le pale della turbina a gas. Il primo passo di detto procedimento è la solidificazione direzionale (SD), necessaria affinché sia eliminata la nucleazione del cristallo davanti alla fronte dendritica. Il maggior numero dei procedimenti SD, però, è caratterizzato da un gradiente basso di temperatura ( $G_L < 10 \text{ k/m}$  o persino 3-5 k/m) sulla fronte isoliquidus, per cui quest'ultimi presentano certi inconvenienti: formazione di una struttura dendritica grossolana, liquazione zonale con "freckles" (= lentiggini), porosità, ecc. Gli AA. descrivono i risultati della SD condotta a vari valori di  $G_L$  e di tasso di crescita su una superlega a base di Ni con intervallo tra caratteristiche fisiche della colata e la solidificazione  $\Delta T_O$  di  $70^{\circ}$ C. La qualità di una lega sottoposta alla SD dipende anche dall'interfaccia tra la fase solida e quella liquida. Dimostrano che la SD a gradiente alto, con raffreddamento rapido per mezzo di metallo liquido, da loro designato SD rapida (SR), porta alla formazione di un'ottima interfaccia, ed elimina sia le freckles sia i carburi a Mg di superficie. Riferiscono poi l'applicazione della RS su un'unità dotata di una capacità produttiva di pale 144 al giorno, ottenendo così un aumento di durata 4-10 volte maggiore.

## Introduction

There are at least three reasons why nickel-base superalloys possess the highest operating properties:

- they are free from the grain boundaries that form the weakest point in a superalloy with respect to high-temperature creep and thermal fatigue stresses;
- they are usually made from specially alloyed superalloys with high thermal stability;
- their crystallographic orientation can be optimally coordinated with axial and azimuthal stress vectors, so as to make use of the advantages associated with single-crystal anisotropy. Single-crystal casting is now being employed on an ever wider scale for critical components, such as gas turbine engine blades, despite the known difficulties in mastering this technological process. Formation of a single-crystal structure in a shaped casting first requires directional solidification (DS) to eliminate crystal nucleation before the front of growing dendrites or on the mould surfaces. The second requirement is the presence of a special starting zone for formation of an orientation crystal needed in the casting working part.

The temperature gradient on the isoliquidus front  $(G_L)$  and the crystal growth rate (R) are of prime importance in the DS method.

Most DS processes are low-gradient processes, since  $G_L$  does not exceed 10 K/cm, and may be only 3 to 5 K/cm.

The shortcomings of low-gradient DS are responsible for the low alloy cooling rate (V cooling =  $G_LRK/sec$ ) and the formation of a coarse dendritic structure, as the distance between the dendrite axes is determined by this rate in accordance with the exponential function:

$$\lambda = A (G_L \cdot R)^{-B}$$
 (1)

A structure of this kind is characterised by coarse precipitations of primary  $\gamma = \gamma'$  eutectic constituents, Mc carbides based on TiC, and the development of porosity.

In addition, low-gradient processes create the conditions for zone liquation along the melt interdendritic channels, which are enriched in readily fusible elements (aluminium, titanium, etc.).

This results in liquation defects: surface freckles, coarse surface  $M_eC$  carbides based on  $Ni_3W_3C$  (when alloys containing carbide are used).

The quality of a DS alloy is also greatly affected by the solid-liquid phases interface, as defined by the ratio of the GL and R values. Plane front solidification in particular occurs under a definite condition:

$$\frac{G_L}{R} \geqslant \frac{\Delta To}{D}$$
 (2)

This expression physically relates the solidification parameters GL and R to the melt characteristics solidification interval ( $\Delta$ To) and impurity diffusion coefficient (D) in the melt.

Violation of this condition, i.e.  $\frac{G_L}{R} < \frac{\Delta To}{D}$ , results in distortion of the plane front and the

formation of cell or dendritic interfaces. It may also contribute to the initiation of new crystallisation centres ahead of the growing dendrite front, as the result of concentrational overcooling.

Expression (2) can be rewritten to permit analysis of DS accompanied by concentrational cooling:

$$\frac{G_{L}}{R} = Kf \frac{\Delta To}{D} \tag{3}$$

where "Kf" is a coefficient defining the type of interface: Kf>1, corresponds to a plane front, at Kf<1, the plane front is distorted. Introduction of this criterion permits quantitative characterisation of the conditions under which cellular, dendritic and equiaxial interfaces are formed. The expression for Kf is readily obtained from (3):

$$Kf = \frac{G_L/R}{\Delta To/D} \tag{4}$$

This criterion is analytically connected with the expression for concentrational overcooling:

$$\Delta T k = \Delta T o \left( 1 - e^{-\frac{x}{K_f}} \cdot x \right) \tag{5}$$

where  $X = \frac{G_L \cdot x}{\Delta To}$  is the relative distance from the solidification front:

Irrespective of the variable distance "x" from the front, the characteristics of concentrational overcooling are obtained from (5) (fig. 1):

- maximum concentrational overcooling:

$$\Delta Tr_{\text{max}} = \Delta To \left[ 1 - K_f (1 - l_n K_f) \right]$$
(6)

- concentrational overcooling gradient

$$G_k = G_L \left( \frac{1 - K_f}{K_f} \right) \tag{7}$$

These characteristics have been employed to analyse the DS of a nickel-base superalloy with  $\Delta T_o = 70^{\circ}\text{C}$  at different  $G_L$  and R. DS of cylindrical specimens  $\emptyset$  15 mm was performed in thin-walled, extruded electrocorundum crucibles, using a wide range of solidification rates and temperature gradients.

Interface type was determined by examining the microstructure of solidified specimens. Kf,  $\Delta T_{kmax}$  and  $G_k$  were calculated from expressions (4), (6) and (7). The results are shown in Table 1.

TABLE 1 - Interface types under different solidification conditions

Solidification conditions  G <sub>L</sub> K/cm		Interface Type			
	R mm/min	Kf –	$\begin{array}{c} \Delta T_{kmax} \\ K \end{array}$	G <sub>k</sub> K/cm	
100	0.1	$8.6 \times 10^{-2}$	49	$1.06 \times 10^{3}$	cellular
100	1.0	$8.6 \times 10^{-3}$	66.5	$1.15 \times 10^4$	cellular
70	1.0	$6 \times 10^{-3}$	67.2	$1.16 \times 10^4$	cellular-dendritic
70	100	$6 \times 10^{-5}$	69.95	$1.17 \times 10^{6}$	dendritic
3	1.0	$2.6 \times 10^{-4}$	69.8	$1.15 \times 10^4$	dendritic
3	100	$2.6 \times 10^{-6}$	70	$1.15 \times 10^{6}$	equiaxial grains

It is clear that the interface type depends on Kf and the two overcooling characteristics  $\Delta T_{kmax}$  and  $G_k$  (fig. 2). A decrease in Kf reduces interface stability, while at low values DS is disturbed due to formation of equiaxial zones ahead of the solidification front. This is associated with either an increase in GL or a decrease in R, depending on the type of alloy involved.

Optimum kf values corresponding to stable formation of directional dendritic structures lie in the range 1 to  $6.10^{-4}$ . They can be obtained by using either high gradients and high solidification rates, or low gradients with low solidification rates. High-gradient processes are more effective, however, since they result in the stable production of directional structures at high cooling rates.

Augmentation of GL on the solidification front requires an increase in the heat flow from the solidified casting in the direction of the cooler. This can be achieved by increasing the cooler's heat emission and head. Our data indicate that the best way to increase GL is to use liquid metal cooling with easily fusible alloys for thin walled shell moulds filled with the melt. We found that the thermal flow density can be increased 3-4-fold by comparison with vacuum cooling, and 8-10-fold compared with vacuum cooling in moulding boxes with the filler.

The Fuko law shows that in a quasi-steady-state process, GL on the front increases in proportion to the thermal flow density. When liquid cooling only was used, an increase of up to 70 K/cm was achieved. Further increases were obtained when special shields were added. R was increased 3-5-fold and the cooling rate 20-30-fold. We have called this method rapid directional solidification (RS).

Quantitative metallography was employed to study the effect of the cooling rate on the microstructure of the nickel-base superalloy casting. Analytical relations corresponding to expression (1) can be written for  $\lambda$ , the size of  $\gamma-\gamma$  eutectic inclusions, and  $M_eC$  primary carbides:

$$\lambda = 620 \ (G_L \cdot R)^{-\frac{1}{3}}$$
 (8)

$$\lambda_{\gamma - \gamma'} = 20.8 \ (G_{\gamma - \gamma'} \cdot R)^{-0.26}$$
 (9)

$$\lambda M_e c = 4.2 (G_{MC} \cdot R)^{-0.26}$$
 (10)

These ratios are plotted in fig. 3. Alloy microstructures after DS at two cooling rates are illustrated in

fig. 4. Refining of the constituents of an alloy during RS results in increases in both strength ( $\sigma_B \sigma_{100} \sigma_-$ ) and ductility ( $\delta$ ,  $\phi$ ) (Table 2).

TABLE 2 - Nickel-base superalloy properties after casting by DS and RS methods

Costing			$\sigma_{\mathrm{B}}$	δ	φ	$\sigma_{100}$			$\sigma_{-1} \ (2 \cdot 10^7)$
Casting method	$G_L R$	λ	20°C	20°C	20°C	900°C	1000°C	20°C	900°C
	K/min	mkm	MPa	%	%	MPa	MPa	MPa	MPa
DS	2-4	400-500	850	9	8	380	186	180	260
RS	70-140	130-180	1100	11	15	410	225	290	300

RS eliminated both freckles and MeC type surface carbides. This was due to sharp decreases in both the area of the interdendritic channels (this being proportional to  $\lambda^2$ ) and the solid-liquid phase zone solidification time, namely:

$$\tau = \Delta \text{To } (G_{L} \cdot R)^{-1}$$
(11)

It must be pointed out that element dendritic liquation is slightly increased during RS (fig. 3). For the reasons stated above, however, this does not result in appreciable zone liquation.

Dendritic liquation growth depends on the cooling rate and is the function with the maximum importance. The cooling rate range investigated lies wholly within this ascent branch, where liquation increases with the cooling rate. Some decrease in the "B" coefficient in expressions (9) and (10) compared with the absolute value can be attributed to an increase in dendritic liquation during RS.

Special UVEK-8P units (fig. 5) have been devised for RS and are now being used on a wide scale. Their specifications are:

Mould heating temperature  $1500^\circ$  Crystallizer temperature  $700^\circ$  Solidification rate 1-20 mm/min Cruising velocity 170 mm/min Working medium: vacuum  $5.10^{-3}$  mm/Hg Dimensions  $7000 \times 5600 \times 4500$  mm Area occupied  $40 \text{ m}^2$ 

Each unit can solidify up to 12 blades at a time in a total operating cycle of 2 hours, giving a production efficiency of 144 blades/day in three shifts. One operator is required to supervise two units. An automatic control system has been developed for technological aspects of the RS process.

High-temperature corundum-mullite crucibles specially developed for RS casting are used for metal melting. They have a high heat resistance. The quick-change variants employed can withstand up to 30 melts followed by cooling practically down to R.T.

Special compositions were created when developing the RS casting technology, and the production parameter values of ceramic refractory cores and corundum-based, thin-walled shell moulds were worked out. A significant increase was obtained in the high-temperature strength of the mould and core materials during bending tests (Table 3).

TABLE 3 - Strength of core and mould materials

Product	Casting Method	o <sub>bend</sub> MPa		Deformation Onset Temperature		
		20°C	1350°C	1550°C	$\sigma_b = 0.2 \text{ MPa}$	$T_{max}$
core	ES	16-20	1.8-2.0	_	1400	1480
	RS	18	10	6	1520	1650
		25	12	8		
mould	ES	3.2	1.6	_	1400	1500
		4.0	2.0			
	RS	26	14	4	1560	1650
		28	18	6	1560	1650

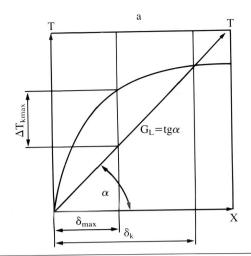
Increased refractoriness and high-temperature strength enabled the shell mould thickness to be reduced by 20-30%. This is of great importance in ensuring a high thermal flow density during cooling of the liquid metal.

RS was taken as the basic method when developing a technology for the single-crystal casting of turbine blades. The anisotropy of single crystals allows parts to be cast in this way with a given crystallographic orientation.

Mention may be made of the fact that both azimuthal and axial orientation with respect to the most loaded blade elements can taken into consideration for highly-loaded parts, such as turbine blades. Single-crystal casting using special seeds with a given orientation in the starting zone goes a long way towards meeting these requirements. This technology has been applied to RS with the UVEK-SP unit. The structure of the gating system and the DS regimens should ensure transfer of the orientation from seeding to the starting zone, and then to the casting. They are determined by the blade structure and must be calculated to suit each particular case. Typical RS-cast blades with directional (a) and single-crystal (b) structures are illustrated in fig. 6.

The output of finished parts by macrostructure can be varied from 60% to 90%, depending on the complexity of a given blade. A high-temperature melt treatment technology (HTMT) has been devised to increase the number of finished parts by single-structure (and hence the total output). Joint studies with the Ural Polytechnic have shown that HTMT ensures melt homogenization prior to solidification. This prevents dissociation of refractory particles and the formation of clusters, which can be regarded as solidification centres during cooling below liquidus and hence responsible for decreasing the stability of single-crystal DS. HTMT also frees an alloy from oxides and other refractory inclusions, as well as detrimental impurities. The 10-15% increase in output achieved with developed HTMT schedules fully offsets the technological complexity associated with its introduction.

Single-crystal casting on the UVEK-SP units has so far been mastered with respect to the manufacture of gas turbine blades. This technology has led to a 4-10-fold increase in the service life of such blades.



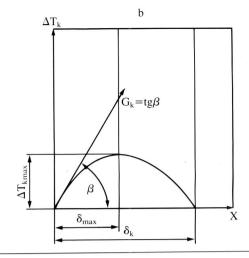


Fig. 1:

Dependence of interface configurations on " $K_F$ " criterion value in DS. a) Concentrational overcooling characteristics

b) Interface configurations

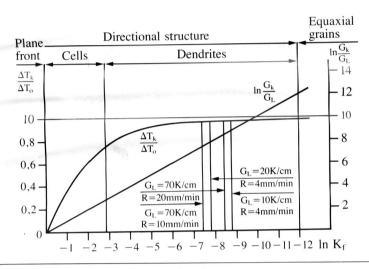


Fig. 2:

Dependence of interface configurations on concentrational overcooling characteristics and " $K_F$ " criterion in DS.

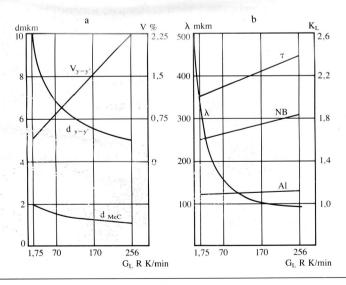
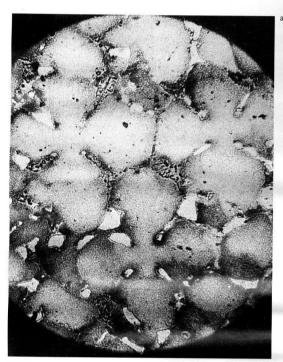


Fig. 3:

Dependence of nickel-base superalloy structural constituents dimensions on cooling rate in DS.



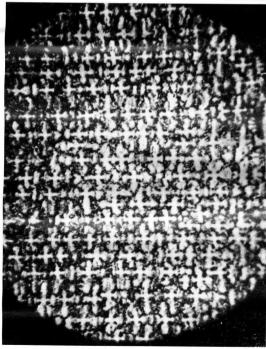


Fig. 4: Nickel-base superalloy microstructures after DS with cooling rates of 4 K/min (a) and 140 K/min (b).

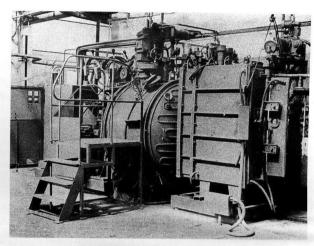
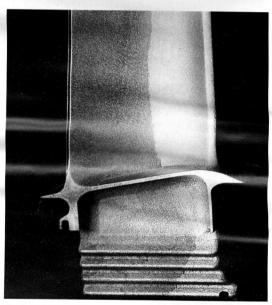


Fig. 5: 9BHK — 8/7 Installation for casting by RS method.



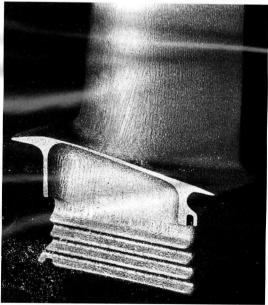


Fig. 6: Turbine blades cast by RS. a) with directional structure b) with single-crystal structure