

# Undercooling of superalloy melts: basis of a new manufacturing technique for single-crystal turbine blades

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## Abstract

The solidification of an undercooled superalloy melt has been investigated to advance a new technique for single-crystal (SC) turbine blade manufacture, the so-called autonomous directional solidification (ADS) technique. A special SiO<sub>2</sub>-free ceramic shell coated inside with a nucleation-inhibiting amorphous layer was used for the mould material. A small longitudinal temperature gradient ensured the alignment of the dendritic structure. A maximum undercooling of about 80 K could be achieved for the superalloy CMSX-6. It was found that above a critical undercooling of about 30 K the dendritic structure changed into a fine equiaxed grain structure. The size of the equiaxed grains was comparable with the secondary arm spacing of the dendrites. Within specimens with a large longitudinal temperature gradient, a transition between the fine-grained and the dendritic structure was observed. In these regions equiaxed grains are present side by side with primary dendrite trunks. Thus the temperature distribution within the undercooled superalloy melt has to be controlled carefully in order to produce an SC microstructure. Nevertheless, it could be shown that it is possible to produce SC superalloy turbine blades by the ADS technique.

## 1. Introduction

Directional solidification (DS) and in particular single-crystal (SC) solidification of turbine blades is usually achieved by using the Bridgman technique [1], where a complex vacuum casting furnace is necessary to optimize the microstructure. Moreover, a time- and energy-intensive processing has to be performed [2]. In order to investigate an alternative method for directional and/or SC casting, the directional solidification of an undercooled melt as proposed by Lux *et al.* [3] was taken up [4]. The improvement of this method results in a new technique for producing SC superalloy turbine blades [5].

The so-called autonomous DS (ADS) process exhibits several advantages compared to the conventional technologies. For example, the complex equipment of the Bridgman process can be simplified, owing to the lack of relative motion between the sample and the heater. The baffle, insulating the cooling chamber from the hot part of the furnace, as well as the withdrawal device can be omitted. Moreover, because of the rapid solidification, the ADS samples have reduced microsegregation which results in improved mechanical properties [3, 6]. Finally, processing times for the ADS technique are significantly shorter than for the Bridgman process.

## 2. Experimental procedure

For the experimental investigations, the low density SC nickel-base superalloy CMSX-6 [7] was chosen (Table 1). The shell moulds were made of an SiO<sub>2</sub>-free, Al<sub>2</sub>O<sub>3</sub> ceramic, coated inside with a nucleation-inhibiting amorphous layer. A ceramic plug at the bottom of the shell mould realized a heat insulation between the melt and a water-cooled chill plate. Turbine blade geometries of different sizes (up to 15 cm in height) as well as cylindrical geometries (15 mm in diameter) were cast.

The superalloy melt was poured into the shell mould without any purification. The mould was preheated up to the casting temperature of the superalloy. After pouring the temperature was kept constant for several minutes to reduce pour turbulence and to ensure a steady state temperature distribution. After this, the controllable heating device was switched off and the melt allowed to cool down according to the declining furnace temperature. Because of the specially prepared shell mould, the nucleation is delayed, leading to a substantial undercooling. The longitudinal temperature gradient within the undercooled melt (ensured by the water-cooled chill plate at the bottom of the shell mould) was varied by using ceramic plugs of different heights. The nucleation occurs at the coldest position

TABLE 1. Nominal composition of the superalloy CMSX-6 (weight per cent)

Cr	9.6-10.3
Al	4.7-4.9
Ti	4.6-4.8
Ta	1.8-2.2
Mo	3.0
Co	5.0
Mn	<0.01
Fe	<0.8
C	<0.006
S	<0.05
Ni	Balance

within the foot of the sample. The experimental set-up is schematically illustrated in Fig. 1(a).

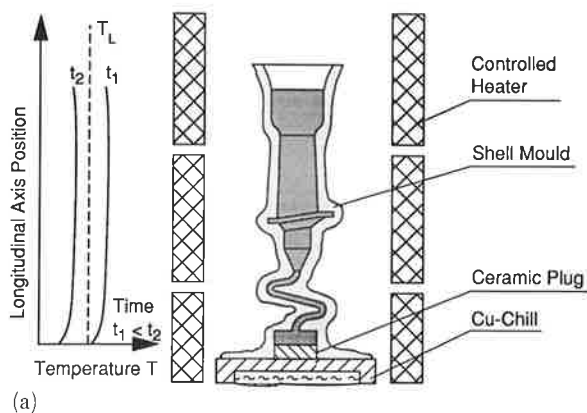
The temperature distribution, as well as the appearance of undercooling and recalescence, was measured by several thermocouples at different positions. The microstructures of the castings were characterized by microsection examination and Laue backscattering. Deviations from the mean crystallographic orientation were investigated by  $\gamma$  ray diffraction analysis. Rocking curves give the intensity distribution of the 468 keV peak of radioactive  $^{192}\text{Ir}$  vs. scattering angle and were measured along the axis of the specimens.

### 3. Results and discussion

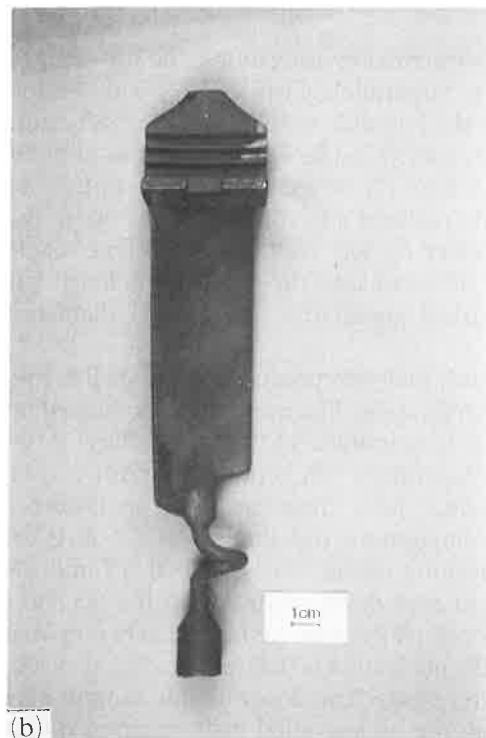
SC turbine blades up to 15 cm in height (Fig. 1(b)) as well as SC cylindrical geometries thus have been produced from the undercooled melt. The crystallographic orientations of the SCs were determined by the Laue backscattering method. Rocking curves of  $\gamma$  ray diffraction analyses with small half-width values and deviations of less than  $5^\circ$  from the crystallographic orientation revealed the excellent quality of the specimens [8]. Primary and secondary dendrite arm spacing, investigated at several longitudinal and transversal sections, appear to be smaller than those achieved by conventional DS-SC processes.

In specimens undercooled less than about 30 K a fine dendritic structure was formed, whereas with a higher undercooling an equiaxed grain structure appeared. This is undesirable but nevertheless an important result. Figure 2 shows a totally grain-refined cylindrical bar, with the temperature distribution along the axis just before the nucleation occurs. The maximum undercooling of about 80 K at the bottom of the sample as well as the shape of the curve have been determined by fitting the experimental values.

With a high undercooling at the bottom of the shell mould and a sufficient temperature gradient within the bulk of the melt, a transition between the fine-grained and the dendritic structure is observed. Figure 3 shows a partly grain-refined sample with the corresponding temperature fit. At the bottom of the bar the critical undercooling is exceeded, whereas the main part of the specimen has been moderately undercooled and thus a fine directional solidified dendritic structure is found. Figure 4 shows an enlarged view of the transition region. It can be seen that the origin of the primary dendrite trunks is within the equiaxed zone. The equiaxed grains are present side by side with primary dendrite trunks. Secondary arms develop further towards the end of the transition region. It should be noted that the size of the equiaxed grains is comparable with the secondary arm spacing of the dendrites.



(a)



(b)

Fig. 1. (a) Schematic illustration of the ADS process with a typical temperature distribution and (b) an SC turbine blade of about 15 cm, produced by this technique.

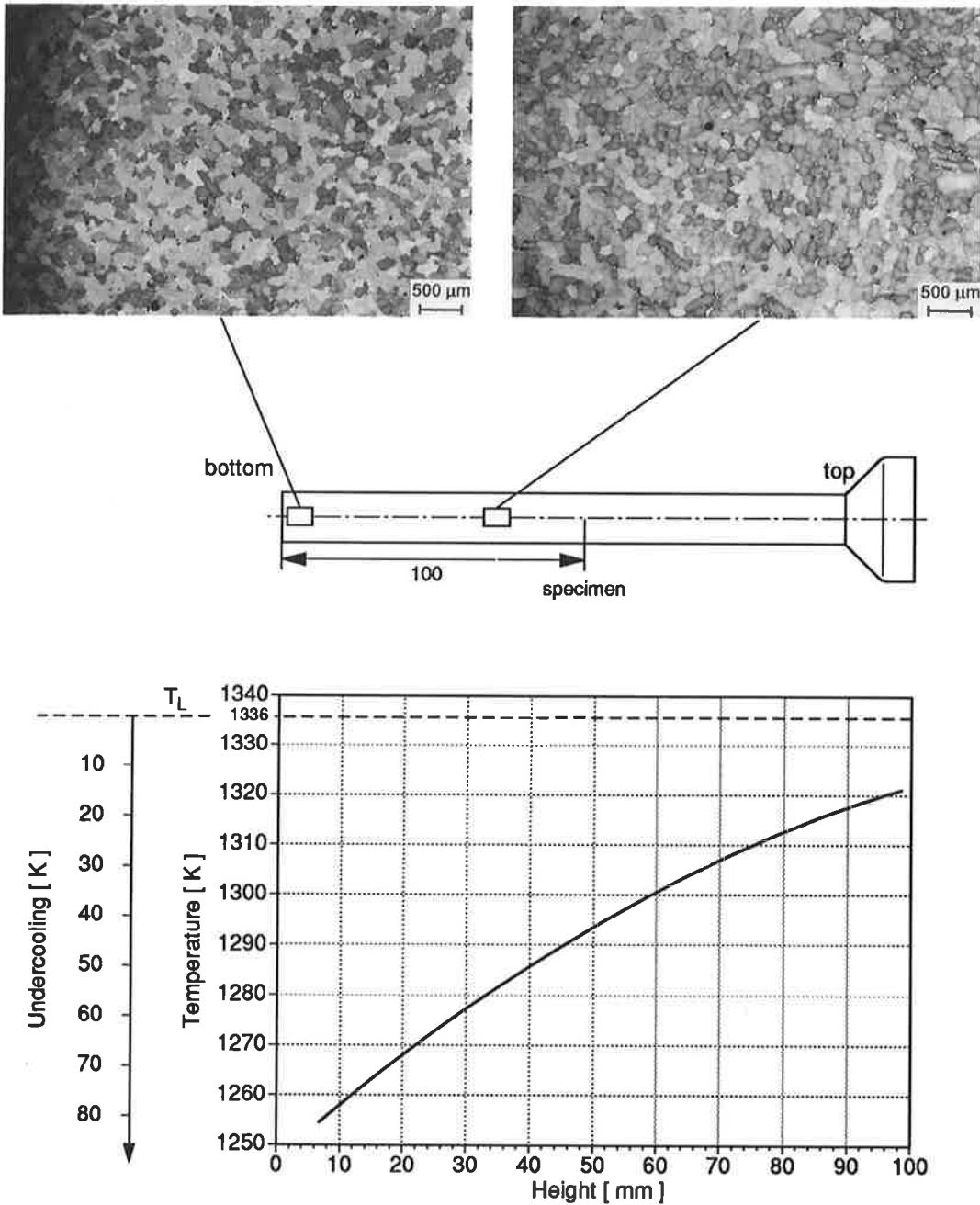


Fig. 2. The longitudinal sections are taken at the marked positions within a totally grain-refined cylindrical sample. In the lower part of this figure the temperature distribution along the sample axis just before the nucleation occurs is given. The maximum undercooling of about 80 K at the bottom of the sample as well as the shape of the curve have been determined by fitting the experimental values.

Grain refinement in undercooled alloy melts has been investigated experimentally by researchers [6, 9–13]. The critical undercooling for this phenomenon is reported to be 150–200 K. Hence there should be another reason for the grain refinement in the undercooled superalloy CMSX-6. However, the existence of

primary dendrite trunks within an equiaxed grain structure seems to exclude heterogeneous nucleation as the only mechanism for the grain refinement and the microsections do not indicate a dendrite break-off in association with strong convective flows.

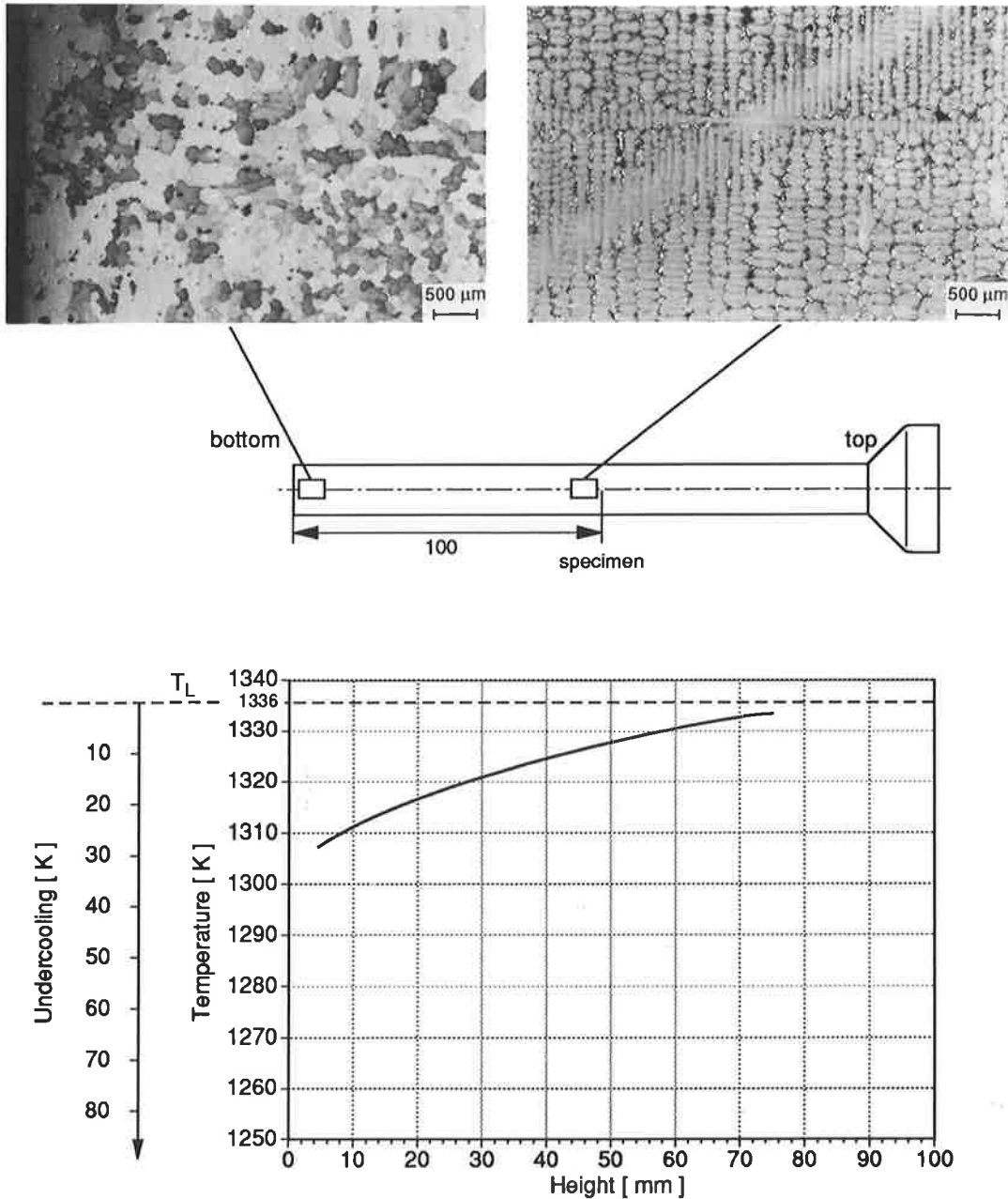


Fig. 3. Partly grain-refined sample with the corresponding temperature fit. At the bottom of the bar the critical undercooling is exceeded, whereas the main part of the specimen has been moderately undercooled and thus a fine DS dendritic structure is found. As determined by Laue backscattering the dendritic part of the specimen is a  $\langle 100 \rangle$ -oriented SC.

#### 4. Conclusions

In the light of the results presented, the temperature distribution within the superalloy melt has to be controlled carefully in order to produce an SC microstructure. Undercooling higher than the limit for the appearance of equiaxed grains must be avoided. To

obtain a directional SC microstructure a small longitudinal temperature gradient is needed. In spite of these difficulties, it was possible to produce an SC superalloy turbine blade by this new technique.

The transition from equiaxed grained to DS dendritic structure within one sample has been presented. In the transition region equiaxed grains are



Fig. 4. Enlarged view of the transition region from equiaxed grained into dendritic structure. It can be seen that the origin of the primary dendrite trunks is within the equiaxed zone. Secondary arms develop further towards the end of the transition region.

present side by side with primary dendrite trunks. Secondary arms develop further towards the end of the transition region.

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