Effects of electron beam smelting on removal of inclusions in nickel-based superalloys

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Abstract. Electron beam smelting has been applied in refining nickel-based superalloys, which has been proved to be an effective method for removing inclusions. The inclusions are aggregated at the circular region (last solidified region) of the ingot surface periphery where the electron beam finally stayed by induced directional solidification after electron beam smelting. The relationship between the cleanness of nickel-based superalloys and the refining times was investigated. Based on the different refining times, the removal efficiency of inclusions was also discussed and the corresponding experiment was carried out to be compared. The results show that the area of the last solidified region on ingot surface decreases with the increasing of the refining times. The optimal refining time of nickel-based superalloys is considered in order to obtain a relatively high removal efficiency of inclusion and a low mass loss rate.

1. Introduction

The performance of nickel-based superalloys is frequently dependent upon both the number and the size of the inclusions presented in the matrix [1]. The previous investigations show that the impurities and inclusions have important impact on the properties of nickel-based superalloys [2-5]. The fatigue and fracture mechanical properties of the superalloys can be strongly affected by the ceramic and metal-reaction introduced inclusions during the primary melting process [6]. Therefore, the cleanliness of nickel-base superalloys is important. As a consequence of the inclusion reduction in the melting process, the low cycle fatigue life of nickel-base superalloys is greatly increased.

Once the chemical composition of the desired superalloy is determined, all kinds of raw materials for the desired composition are prepared into a master alloy ingot through the smelting process. The chemical composition must be strictly controlled, and the purity of the powder metallurgy (P/M) superalloy needs to be improved from the source for ensuring the excellent quality of superalloys, namely the smelting of master alloys. Vacuum induction melting (VIM) is the most mature process for melting P/M master alloy, but the master alloy could be contaminated by the refractory crucible during VIM. Consequently, the master alloy after VIM needs to be refined in order to improve the purity of P/M superalloy. Besides, the vacuum arc remelting (VAR) on the basis of VIM is used for
the secondary refining in Russia. In the United States the secondary refining by electroslag remelting (ESR) on the basis of VIM is carried out, and finally the third refining with VAR is conducted.

In order to remove impurities and inclusions from the superalloys [6, 7], electron beam smelting (EBS) is considered as a kind of effective way under a high temperature and high vacuum condition provided by electron beam equipment. In addition, the pollution from the crucible can be avoided by using water-cooled copper crucible during EBS process [8]. The electron beam button melting (EBBM) process has been widely used for assessing the content of inclusions in high strength alloys, notably of nickel-based superalloys [9, 10]. Various investigations have been carried out regarding the melting and solidification process during EBBM [11-17]. It has been proposed [12, 16, 17] that non-wetting inclusions such as Al₂O₃ rapidly segregate to the surfaces of the molten droplets during the fall from the electrode on to the melt surface, where thermocapillary (Marangoni) effects in moving them to the center of the button surface are involved.

In the present work, two different solidification methods were used for studying the effect of electron beam on removal of inclusions in nickel-based superalloy. Smelting with different smelting time duration with the same smelting power and different number of smelting cycles under the same smelting parameters were carried out. The effect of the last solidified region in the ingot was also investigated.

2. Experimental

Experiments with raw materials from nickel-based superalloys by implementation of vacuum induction melting (VIM) were conducted. The mass fraction of Ni, Cr, Co, W, Mo, Al, Ti and Nb in the raw materials was 56.218, 16.032, 12.999, 3.952, 4.115, 2.359, 3.612 and 0.713, respectively. The density of the raw material was 8.14 g/cm³. The bar was cut into the central part and edge part, as shown in figure 1. Figure 1(a), 1(b) and 1(c) present the microstructure of the central part. There was a shrinkage cavity in the central part. Al₂O₃, Al₂O₃-SiO₂ and Al₂O₃-SiO₂-CaO with irregular shapes were present in the central parts and their size is 50 μm, 30 μm and 40 μm, respectively. Inclusions were not found in the edge part.

The raw material of the four groups of experiments was about 500 g with the smelting power of 12 kW. During three of them the induced solidification was performed as the smelting power was turned down slowly and the power of electron beam was decreased gradually in a liner relationship to 0 kW for 5 min after smelting process and the smelting times were 0 min, 10 min and 30 min, respectively, with smelting power of 12 kW. During the other experiment the rapid solidification was held with the
smelting power turned down to 0 kW immediately after the smelting process, with the smelting power of 12 kW and smelting time of 30 min. The weight of the raw material was about 1500 g with smelting power of 15 kW and smelting time of 12.5 min. The ingot with the weight of 1500 g was smelted twice applying the following method. After the last solidified region of the ingot was cut off, the remaining part of the ingot was smelted for a second time with smelting time of 12.5 min and smelting power of 15 kW. The last solidified region of the ingot was also cut off, the remaining part of the ingot was smelted third time with smelting time of 12.5 min and smelting power of 15 kW.

Induced solidification was used for the second series of experiments with the same smelting power and different number of smelttings. The raw material was placed in the water-cooled crucible, after that the chamber was evacuated to a pressure less than $5 \times 10^{-2}$ Pa and the gun chamber was evacuated to a pressure less than $5 \times 10^{-3}$ Pa. Figure 2 is a schematic diagram of the Electron Beam Smelting (EBS) process. The surface of the melt was then irradiated by the electron beam with a circular scanning pattern to ensure it was homogeneously and steadily heated. Figure 3 shows the relative relationship between the smelting power and the smelting time during the EBS process. The EBS process was divided into three stages: melting stage, refining stage and solidification stage. The ingot was cut in direction along the diameter and through the center of the last solidified region, the cross-section was burnished and polished. The microstructure of ingot section was observed by SEM (Scanning Electron Microscope), EDS (Energy Dispersive Spectrometer) and tabletop microscope (TM3030 plus).

**Figure 2.** EBS schematic diagram.  
**Figure 3.** The relationship between smelting power and smelting time during EBS process.

### 3. Results and discussion

#### 3.1. Ingot morphology

Figure 4 shows the macro-morphologies of the nickel-based superalloy ingots after EBS with the smelting power of 12 kW. Figures 4(a), 4(b) and 4(c) present the morphologies of the ingots obtained by induced solidification, figure 4(d) shows the morphology of the ingot obtained by rapid solidification. The ingots are with heights of approximately 1.3 cm and with diameters of approximately 9 cm. The regions marked with a red line in the figures 4(a), 4(b) and 4(c), are named last solidified region. They are located near to the right edge of the ingot surface and this is the position that the electron beam finally stayed at the end of the experiment. Figure 5(a) and 5(b) present the detected by SEM and EDS surface morphologies of the last solidified regions of the ingot, obtained by EBS with power of 12 kW, smelting time 30 min and induced solidification. The former studies [18] have shown that there are large amounts of inclusions in the last solidified region. The species of the inclusions on the surface of the last solidified region are SiO$_2$-CaO and Al$_2$O$_3$-SiO$_2$-
CaO, which are the same as the raw materials. Foreign inclusions cannot be introduced to the molten pool, because the water cooled copper crucible is used during EBS. Thus, the inclusions in the last solidified region come from the raw material. It was found that the inclusions were enriched at the last solidified region. The surface area of the last solidified region is shown in table 1. The surface area of the last solidified is reduced with the increasing of the smelting time.

![Figure 4](image)

**Figure 4.** Morphology of superalloy ingots with smelting power of 12 kW: (a) smelting time 0 min, induced solidification; (b) smelting time 10 min, induced solidification; (c) smelting time 30 min, induced solidification; (d) smelting time 30 min, rapid solidification.

![Figure 5](image)

**Figure 5.** The microstructures of the last solidified region on the surface of the ingot with smelting power of 12 kW and smelting time of 30 min, induced solidification: (a) the edge of the last solidified region on the surface of the ingot; (b) the center of the last solidified region on the surface of the ingot.

| Smelting time/min | 0    | 10   | 30   |
|-------------------|------|------|------|
| Surface area of the last solidified region/cm² | 2.31 | 1.505 | 0.803 |
Figure 6 shows the macro-morphologies of nickel-based superalloy ingots after EBS with different smelting times of the smelting power of 15 kW and the smelting time of 12.5 min. The ingots are with height of approximately 1.6 cm and with diameter of approximately 11.5 cm. The surface area of the last solidified region is shown in table 2. The surface area of the last solidified is reduced with the increase of smelting times.

![Figure 6. Morphology of superalloy ingots with smelting power of 15 kW (a) smelting 1 time; (b) smelting 2 times; (c) smelting 3 times.](image)

| Smelting times | 1 | 2 | 3 |
|----------------|---|---|---|
| Surface area of the last solidified region/cm² | 4.56 | 1.09 | 0.262 |

### 3.2. Microstructure

Figure 7 shows the microstructure of the cross-section of the last solidified region in the ingots with smelting power of 12 kW and different smelting time duration. The yellow line in figure 7 shows the boundary of the last solidified region in the cross-section of the ingot. The depth of the last solidified region in the ingots with smelting time duration of 0 min, 10 min and 30 min are 1.8 mm, 1.1 mm and 0.87 mm for smelting power of 12 kW, respectively. The depth of the last solidified is reduced with the increase of smelting time under the same smelting power.

![Figure 7. The microstructure of the cross section of last solidified region in the ingots with smelting power of 12 kW (a) smelting 0 min; (b) smelting 10 min; (c) smelting 30 min.](image)
Figure 8 shows the microstructure of the cross-section of last solidified region in the ingots for different number of smelting cycles with the same smelting power and smelting time duration. The depth of the last solidified region in the ingots with smelting 1 time, 2 times and 3 times are 3.3 mm, 0.55 mm and 0.31 mm for smelting power of 15 kW and smelting time duration of 12.5 min, respectively. The depth of the last solidified region is reduced with the increase of the times the smelting is done under the same smelting power and smelting time duration.

![Image](image_url)

**Figure 8.** The microstructure of the cross section of last solidified region in the ingots with smelting power of 15 kW and smelting time of 12.5 min (a) smelting 1 time; (b) smelting 2 times; (c) smelting 3 times.

### 3.3. Inclusion removal

The inclusions are mainly enrichment in the last solidified region. Since the inclusions enrich at the last solidified region, the cleanness of the ingot can be expressed by the rate of the inclusions in the last solidified region in the ingot. Namely, the removal efficiency is proportional to the rate of the last solidified region in the ingot. If the last solidified region is assumed as a cylinder and the density of the last solidified region is the same as that of the superalloy, the volume of the last solidified region can be calculated by the value of the surface area and the depth of the last solidified region. Then, the mass of the last solidified region can be obtained.

\[ v = S \times h \]  
\[ m = v \times \rho \]  

The rate of the last solidified region in the ingot is:

\[ \beta = \frac{m}{M} \times 100 \]  

where \( v \) is the volume of the last solidified region, \( S \) is the area of the last solidified region on the surface of the ingot, \( h \) is the depth of the last solidified region, \( m \) is the mass of the last solidified region, \( \rho \) is the density of the superalloy, \( \beta \) is the rate of the last solidified region in the ingot, \( M \) is the mass of the ingot.

Figure 9 shows the mass loss rate of superalloy and the rate of the last solidified region in the ingot with different smelting time duration for the smelting power of 12 kW. The mass loss rate of superalloy is increased with the increasing of the smelting time. The rate of the last solidified region in the ingot is reduced with the increasing of the smelting time more and more slowly.

Figure 10 shows the mass loss rate of superalloy and the rate of the last solidified region in the ingot with different number of smelting cycles for the same smelting power of 15 kW and smelting time duration of 12.5 min. The mass loss rate of the superalloy changes a little under these smelting power and smelting time. The rate of the last solidified region in the ingot is reduced with the increasing of the smelting times and the reduction between the second and third smelting is smaller.
Figure 9. The mass loss rate of superalloy and the rate of the last solidified region in the ingot with different smelting time duration for the smelting power of 12 kW.

Figure 10. The mass loss rate of superalloy and the rate of last solidified region in the ingot with different number of smelting cycles for the smelting power of 15 kW and smelting time duration of 12.5 min.

4. Conclusions
1. After solidification, two different solidification methods were used to solidify. After electron beam induced solidification, the inclusions were concentrated in the final solidified area.
2. The surface area and depth of the last solidified region of the ingot was reduced with the increase of the smelting time duration at smelting power of 12 kW.
3. The mass rate of the last solidified region of the ingot was reduced with the increase of the number of smelting cycles at the same smelting conditions - smelting power of 15 kW and smelting time of 12.5 min. The mass rate of the last solidified region at smelting 1, 2 and 3 times were about 0.82%, 0.033% and 0.0044% respectively.
4. The optimal inclusion removal was observed after smelting 3 times, while the evaporation losses of the superalloy were smaller, if the smelting had been performed 1 time.

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