Phase Analysis of a Laserglazed Rene’N4 Superalloy

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It is the first time that the phases in laserglazed single crystal nickel-base superalloy Rene’N4 have been studied systematically. The results show that there are three kinds of carbides and finely dispersed γ’ phase, as well as the γ/γ’ eutectic in the microstructure, and the carbides are f.c.c TiC, TaC and hexagonal WC. These carbides and γ/γ’ eutectic are distributed in the interdendritic regions. The cooling rate strongly affects not only the morphology of TiC but also the morphology and size of the γ’. The morphology of TiC changes from the as-cast blocky form to dendritic and finally to a well-branched chrysanthemum-like form as the cooling rate is increased. At the same time, the size of γ’ becomes smaller and the morphology of γ’ changes from cubic to rounded-cubic. After rapid solidification, the as-cast well-developed mushroom-like γ/γ’ eutectic alters into featherlike. The existence of γ’ and γ/γ’ eutectic indicates that their precipitation rates are too large to be able to suppress their precipitations under a rapid solidification condition.

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I. Introduction

In order to satisfy the design of gas turbine engine, the cast superalloys have been used extensively. Especially, when the service temperature becomes higher the single-crystal nickel-base superalloy becomes a most promising candidate for the blade materials. Therefore, it can be proved from the international and national symposia that the research on single crystal superalloy is one of the current important projects(1)–(3).

Laserglazing is one of the effective methods in studying rapid solidification(4)–(8), and it is also an important way in improving the surface properties(9). At present, the laserglazing of superalloy is still a prime field. Especially, because the difference of corrosion resistance between the original single-crystal and laserglazed area makes it difficult to prepare the TEM foils, the systematical research on the microstructure of the laserglazed superalloys is limited, to some extent, to a level of macroscopic scale and replica observation(9). Because the distribution, size and morphology of the phases in an alloy strongly affect its mechanical properties, the main purpose of this paper is thus to investigate them in a laserglazed nickel-base superalloy in order to understand the effect of rapid solidification on the microstructure and properties of rapidly solidified superalloys.

II. Experimental

Laser rapid solidification processing had been performed on a 5 kW CO₂ continuous wave laser. The samples used in this study were Rene’N4 single crystal nickel-base superalloy bars having the <001> growth direction, prepared by a spiral selection method in a mould withdrawal and unidirectional solidification furnace. To speed up the selection process, a starter section could be placed below the actual bar section. The growing dendritic interface in the starter was then required to make three or four direction changes before it encounters a useful mold section, so that only one grain having the <001> crystallographic direction survives and dominates, as shown in Fig. 1. The following was the chemical composition (mass%):

<table>
<thead>
<tr>
<th>Al</th>
<th>Ti</th>
<th>Nb</th>
<th>Ta</th>
<th>W</th>
<th>Mo</th>
<th>Cr</th>
<th>Co</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.72</td>
<td>4.15</td>
<td>0.62</td>
<td>3.94</td>
<td>5.79</td>
<td>1.58</td>
<td>8.90</td>
<td>7.50</td>
<td>balance</td>
</tr>
</tbody>
</table>

Fig. 1 Schematic view of the spiral selection method for preparing single crystals.
The laser-irradiated regions were formed by moving the substrate beneath a laser beam normal to the (001) surface of the specimens. The cooling rates were calculated according to the reference(10). The scanning speed of the laser beam was in the range of 1 to 40 mm/s and the corresponding range of cooling rate was $1.2 \times 10^3$ to $1.1 \times 10^3$ K/s. The power of the laser beam was 2700 W.

The TEM foils were cut from the irradiated surface and produced with the twin-jet electropolishing method using a hydrochloric acid-alcohol electrolyte. The single stage extraction replica was produced from the surface of the laser glazed region. The microstructure was observed by using PHILIPS EM-420 transmission electron microscopy (TEM) and the chemical compositions of the phases were analyzed by TEM-EDAX. At the same time, the CAMERAX-MICRO type electron probe was also used to analyze the composition.

### III. Results and Discussion

There were a lot of $\gamma'/\gamma$ eutectics and a little blocky TiC in the interdendritic regions as well as a large quantity of dispersed $\gamma'$ particles in the as-cast Rene’N4 single crystal nickel-base superalloy. After laser glazing treatment, the kinds and morphologies of the phases were changed greatly.

Figure 2 showed the morphology and the diffraction patterns of TiC before and after laser glazing treatment. It could be seen that the amount of the blocky TiC was not much in the interdendritic regions (denoted by the arrow in Fig. 2(a)) and its size was about 3 $\mu$m. However, in the rapidly solidified microstructure, although TiC was still distributed in the interdendritic regions, its morphology and size had been varied greatly. The size of TiC was about 2 $\mu$m. The lattice constant of TiC which was obtained from the selected area diffraction patterns was 0.4591 nm, that was slightly larger than that of normal composition TiC (0.4360 nm). This was because some Ti atoms in the TiC lattice had been replaced by other atoms such as Ta, Cr and W, which had larger atomic radius than that of Ti atom and hence to enlarge the lattice constant. Figure 3 is the main composition of TiC obtained by TEM-EDAX. It could also be seen from Fig. 2(b) that the advancing front had the faceted character obviously. It was evident that the growth mechanism of TiC even under rapid solidification condition was still the faceted growth mechanism. Further more, the growth morphologies continued to vary considerably as a function of cooling rate during the rapid solidification, altering the shape from dendritic (cooling rate $dT/dt=1.2 \times 10^3$ K/s) to radially branched chrysanthemum-like ($dT/dt=1.1 \times 10^3$ K/s), as shown in Fig. 4. Despite a variety of the growth morphologies, they had basically a radial centre being the carbide nucleus. However, the morphologies of TiC formed at different cooling rates also had different characters. When the cooling rate was $1.2 \times 10^3$ K/s (as shown in Fig. 4(a)), the primary crystal axes of TiC grown in the faceted growth mechanism were symmetrical, it is because the diffuseness of the solute atoms were more fully in this case, hence the growth of TiC was complete compared and was of the anisotropy. However, as the cooling rate increased, the morphologies changed greatly (as shown in Fig. 4(b) and (c)). Firstly, because of the crystal anisotropy, some main axes of TiC were formed. Subsequently, since the growth of TiC is a diffusion-controlled process and may bifurcate repeatedly from these main axes to accommodate the diffusion of solute atoms, as a result, the degree of branching of TiC increased with the increase of cooling rate, hence the

Fig. 2  Morphologies and the diffraction patterns of TiC. (a) The morphology of TiC (denoted by the arrow) in the original sample, (b) The morphology of TiC in rapid solidified sample.

Fig. 3  The main composition of TiC obtained by TEM-EDAX at cooling rate: $4.8 \times 10^3$ K/s.
growth of TiC tended to crystal isotropy. Therefore, the chrysomum-like TiC had both the crystal anisotropy and the crystal isotropy.

After laserglazing, the cubic $\gamma'$ particles dispersed in the as-cast microstructure was transformed into rounded-cubic $\gamma'$ particles in the laser-irradiated region (as illustrated in Fig. 5). The formation of rounded-cubic $\gamma'$ particles was also the result of the limitation of time and space. The cooling rate strongly affected not only the morphology of $\gamma'$ phase but also its size. Figure 6 was the relationship of scanning speed and the average $\gamma'$ size. The average $\gamma'$ size was only about 9 nm when the scanning was 40 nm/s.

Figure 7 showed the morphologies of $\gamma/\gamma'$ eutectics before and after laser remelting. Figure 7(b) was the dark-field image of featherlike $\gamma/\gamma'$ eutectic which was formed by surrounding the superlattice denoted by the pinpoint. It was interesting to note that there was a great difference between the morphologies of the as-cast and laserglazed $\gamma/\gamma'$ eutectic. The as-cast microstructure of the $\gamma/\gamma'$ eutectic was a well-developed mushroom-like shape (as shown in Fig. 7(a)). Generally, the $\gamma/\gamma'$ eutectic, having a larger size (about 20 $\mu$m) could be divided into three parts: a—the eutectic nucleus, b—the eutectic core, c—the eutectic cap. After the rapid solidification, the reprecipitated $\gamma'/\gamma'$ eutectic still existed and was distributed in the interdendritic region, but turned the morphology from the mushroom-like to the featherlike shape. The featherlike $\gamma/\gamma'$ eutectic only had two parts compared with the as-cast $\gamma/\gamma'$ eutectic: the eutectic nucleus (a) and the eutectic cap (c) and its size was only about 0.8 $\mu$m. The morphology of $\gamma'$ in featherlike $\gamma/\gamma'$ eutectic was of elliptical shape. The existence of the $\gamma/\gamma'$ eutectic denoted that the precipitation rate of the $\gamma/\gamma'$ eutectic was very high and the cooling rate ($1.1 \times 10^3$ K/s) could not suppress the eutectic growth.

In addition to the phases of TiC, $\gamma'$ and $\gamma/\gamma'$ eutectics which existed in the as-cast condition, there were also other two kinds of carbides in the microstructure of rapid solidification. Figure 8 showed the plate-like carbide denoted by the arrow. The analytical results of the selected area electron diffraction proved it was the hexagonal WC.

In the alloy, a higher Ta content and a segregation of
Ta solutes were observed in the interdendritic region, as shown in Fig. 9, it was possible that the Ta-rich phase, i.e. MC type TaC, might exist because of the enrichment of Ta solutes in a local region. Figure 10 showed the morphology of another carbide and its diffraction patterns. Figure 10(a) and (b) were the bright-field and the dark-field image, respectively. According to the indexed SAD patterns (as shown in Fig. 10(c)), it was proved that this carbide was MC type TaC. It was obvious that the morphology of TaC was similar to that of TiC, but there was no faceted character on the TaC crystal planes under the condition of rapid solidification.

In a word, there were five kinds of phases and structures in the rapidly solidified microstructure of the laser glazed Rene'N4 superalloy, including three kinds of carbides, i.e. TiC, TaC, WC and dispersed γ', as well as the γ/γ' eutectic. Table 1 summarizes the phases present in a Rene'N4 single crystal nickel-base superalloy before and after laser glazing.
IV. Conclusions

From phase analysis, the following five phases had been observed in a laser glazed Rene’N4 single crystal nickel-base superalloy.

1. Three kinds of carbides: MC type TiC, TaC and hexagonal WC. The morphology of TaC was similar to that of TiC which had a dendritic structure. WC existed in a thin plate-like shape. With increasing the cooling rate, the morphology of TiC changed from dendritic at 1.2 × 10³ K/s to well-branched chrysanthemum-like form at 1.1 × 10³ K/s.

2. Fine dispersed γ’ particles: With the increase of scanning speed, the γ’ morphology transforms from cubic to rounded-cubic shape, at the same time, the size of γ’ decreases from 500 to 9 nm.

3. Featherlike γ/γ’ eutectic. It is the first time that a featherlike γ/γ’ eutectic was observed in the laser glazed rapidly solidified microstructure of the Rene’N4 single crystal nickel-base superalloy. The existence of the γ/γ’ eutectic indicated that the growth rate of γ/γ’ eutectic is very high and the cooling rate (1.1 × 10³ K/s) is not high enough to suppress the eutectic growth.

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REFERENCES


