RECENT PROGRESS OF MANUFACTURING TECHNOLOGIES ON C&W SUPERALLOYS IN CHINA

Jinhui Du¹, Qun Deng², Jianxin Dong², Xishan Xie², Zhigang Wang³, Changhong Zhao³, Guosheng Chen⁴, Wei Xie⁴, Tongwei Luo⁵, Xincai Wang⁵, Yong Zhang⁶

1. Central Iron and Steel Research Institute, Beijing, 100081, China;
2. University of Science and Technology Beijing, Beijing, 100083, China;
3. Fushun Special Steel Co., Ltd., Fushun, Liaoning, 113001, China;
4. Baoshan Special Steel Co., Ltd., Shanghai, 200940, China;
5. Changcheng Special Steel Co., Ltd., Jiangyou, Sichuan, 621701, China;
6. Beijing Institute of Aeronautical Materials, Beijing, 100095, China

Keywords: Recent Progress, Manufacture Technology, C&W Superalloys

Abstract
In order to meet the growing demand on markets both domestic and international, the manufacturing technologies of superalloys keep improving in China. This paper reviews the progresses and trends on melting, cogging, and forging technologies for China’s C&W superalloys in the recent 20 years.

Introduction
Cast and Wrought (C&W) superalloys have been developed since 1950s in China [1]. The past 60 years has witnessed the progress of China’s C&W superalloy, starting from imitation to innovation. The manufacturing technologies and product quality keep improving.

Especially since the 1990’s, the demand for superalloy products grows rapidly with the fast development of China's energy, power and other areas of national economy. Under the driving force of strong market demands both domestic and international, new processes and new technologies keep being developed and the quality of products gets improved continuously.

The international demands also drive Chinese manufacturers to upgrade and extend capacity. In recent years, many advanced new devices have been installed in China, such as 6/12 ton Vacuum Induction Melting (VIM) Furnace, 5/20 ton Protective atmosphere Electro Slag Remelting (PREP) furnace, 10 ton Helium-cooled Vacuum Arc Remelting (VAR) furnace; 4500/6000 ton billetizing Press, 1850 ton radial forging Press, 80000 ton hydraulic die-press, 35500 ton screw press, 50000 ton and 36000 ton extruder. These advanced equipments also facilitate the implementation of improved processes and technologies for C&W superalloys production.
The present paper reviews the progresses and trends on melting, cogging, and die forging technologies for C&W superalloys in China over the past 20 years. The base elemental composition of superalloys appeared in the present paper is shown in Table1.

<table>
<thead>
<tr>
<th>Tread mark in China</th>
<th>Typical composition (w.t.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
</tr>
<tr>
<td>GH4169</td>
<td>0.04</td>
</tr>
<tr>
<td>GH4738/4864</td>
<td>0.04</td>
</tr>
<tr>
<td>GH4706</td>
<td>0.04</td>
</tr>
<tr>
<td>GH4033</td>
<td>0.05</td>
</tr>
<tr>
<td>GH4698</td>
<td>0.05</td>
</tr>
<tr>
<td>GH2132</td>
<td>0.05</td>
</tr>
<tr>
<td>GH2674</td>
<td>0.05</td>
</tr>
<tr>
<td>GH4720Li</td>
<td>0.01</td>
</tr>
<tr>
<td>GH4742</td>
<td>0.06</td>
</tr>
</tbody>
</table>

**Melting**

**Purifying**

Not only the mechanical properties such as plasticity, creep-rupture property, LCF (low cycle fatigue) property, but also the life and safe reliability of components are significantly affected by the purity of superalloys [1, 2]. Therefore, reducing S, Si, Mn, gas elements N, O, H, and trace elements Pb, Ag, Sn, Sb, Se, Te, Tl and Bi contents has always been the goal of melting technology progress for superalloys. The sulfur level can be effectively reduced by introducing of lime (CaO), Ni-Ca, and Ni-Mg and providing slag during the VIM process. Further more, the pouring systems with multi slag stopping implements and ceramic filter can improve the purity of molten alloys. It was reported that the S content could be reduced to 20ppm by slag processing during VIM [3].

Triple-Melt (VIM+PESR+VAR) have been used since the 2000’s in China, and gradually being applied in producing GH4169, GH4738/4864, GH4706 and other C&W superalloys. Compared with the traditional double vacuum melt (VIM+VAR), Triple-Melt can further reduce the level of S and O. ESR under protective gases (PREP) can also effectively reduce the content of N and O. Thanks to the above methods, the purity of C&W superalloys in China has been improved. As the example shown in Figure 1, using VIM+VAR, the sulfur level in GH4169 can be controlled less than 20ppm compared to the former 40ppm. Further more, the S content can be lower than 10ppm using Triple-Melt. The ideal can even be less than 5ppm.
Minor Elements Control
Besides controlling harmful elements, a series of studies on optimizing minor elements such as P, B, Zr, and Mg have also been carried out in China to improve the performances of superalloys [4-10]. One of the significant efforts involved is optimization of phosphorus in GH4169, whose content had always been controlled under 50ppm, and once been pursued to the “ultra-low” level as it was initially considered harmful to the alloy. However, later studies revealed that the creep resistance can be strongly improved when the P content is increased up to 260ppm, as shown in Figure 2. It is also found that excessive P and B has adverse effects on segregation behaviors, hot working ductility and weldability[11]. So P content is now controlled so it does not exceed the upper limit of 150ppm in China by now.

Uniformity Improvement
After the 1990’s, most of the C&W superalloys in China are produced by VIM+VAR or VIM+ESR. One of the most common problems for traditional ESR is the uneven melting loss rate for the elements with high oxidizability such as Al, Ti and C. However, argon protected ESR can significantly change the uneven level of C, Al, and Ti, which is shown in Table 2. Accordingly, PESR is also widely used in Triple-Melt, especially for the alloys with high Al and Ti contents. Table 3 shows an actual uniformity level of chemical composition for China's superalloys.
Figure 2 The effect of P content on creep rupture properties for GH4169 alloy

Table 2 The melting loss difference between ESR and PESR (w.t.%)

<table>
<thead>
<tr>
<th>Melting route</th>
<th>Sampling position</th>
<th>GH4033 C</th>
<th>Al</th>
<th>Ti</th>
<th>GH2132 C</th>
<th>Al</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>VIM+ESR</td>
<td>head</td>
<td>0.037</td>
<td>0.92</td>
<td>2.45</td>
<td>0.049</td>
<td>0.24</td>
<td>1.98</td>
</tr>
<tr>
<td></td>
<td>bottom</td>
<td>0.033</td>
<td>0.82</td>
<td>2.71</td>
<td>0.055</td>
<td>0.15</td>
<td>2.20</td>
</tr>
<tr>
<td>VIM+ESR+VAR</td>
<td>head</td>
<td>0.037</td>
<td>0.87</td>
<td>2.64</td>
<td>0.051</td>
<td>0.22</td>
<td>2.15</td>
</tr>
<tr>
<td></td>
<td>bottom</td>
<td>0.035</td>
<td>0.82</td>
<td>2.72</td>
<td>0.053</td>
<td>0.18</td>
<td>2.25</td>
</tr>
</tbody>
</table>

Table 3 Uniformity level of chemical composition for Φ220mm bars (w.t.%)

<table>
<thead>
<tr>
<th>Sampling position</th>
<th>GH4169 C</th>
<th>Al</th>
<th>Ti</th>
<th>Nb</th>
<th>Mo</th>
<th>B</th>
<th>GH2132 C</th>
<th>Al</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>head</td>
<td>0.028</td>
<td>0.45</td>
<td>0.97</td>
<td>5.04</td>
<td>3.12</td>
<td>0.0045</td>
<td>0.041</td>
<td>0.18</td>
<td>2.18</td>
</tr>
<tr>
<td>middle</td>
<td>0.029</td>
<td>0.45</td>
<td>0.98</td>
<td>5.01</td>
<td>3.14</td>
<td>0.0044</td>
<td>0.043</td>
<td>0.19</td>
<td>2.18</td>
</tr>
<tr>
<td>bottom</td>
<td>0.028</td>
<td>0.44</td>
<td>0.99</td>
<td>5.03</td>
<td>3.14</td>
<td>0.0045</td>
<td>0.043</td>
<td>0.20</td>
<td>2.17</td>
</tr>
<tr>
<td>central</td>
<td>0.028</td>
<td>0.44</td>
<td>1.00</td>
<td>5.03</td>
<td>3.12</td>
<td>0.0049</td>
<td>0.042</td>
<td>0.18</td>
<td>2.15</td>
</tr>
<tr>
<td>R/2</td>
<td>0.029</td>
<td>0.45</td>
<td>0.98</td>
<td>5.01</td>
<td>3.14</td>
<td>0.0044</td>
<td>0.043</td>
<td>0.19</td>
<td>2.18</td>
</tr>
<tr>
<td>Rim</td>
<td>0.029</td>
<td>0.45</td>
<td>1.00</td>
<td>5.01</td>
<td>3.16</td>
<td>0.0045</td>
<td>0.042</td>
<td>0.19</td>
<td>2.18</td>
</tr>
</tbody>
</table>

Segregation Control
Segregation control is very important for C&W superalloys, especially for the compositions with high alloying level and large ingot size. Macro-segregation in the form of freckles and white spots can occur. Macro-segregation may pass on to billet or forged products, which directly affects the performance or reduces the stability during long term operation [1, 2]. Accordingly, current or melting rate are controlled carefully during VAR process. Helium-cooling is also widely used in China to reduce the segregation level. It is found that φ508mm VAR ingots cooled by He + water have the same segregation degree with the single water cooled φ406mm VAR ingots.
Several producers of critical components for power or energy industry in China have adopted the use of a hybrid re-melting process: VIM to ensure an initial electrode with low oxygen and precise chemistry, followed by ESR. The ESR electrode will be cleaner and sound but may contain freckles. The final segregation-free structure is obtained through the application of a third melting process (VAR) to the ESR ingot. The improved cleanliness and soundness of the electrode by ESR facilitates VAR control as shown in Figure 3 [2]. Therefore, the products, referred to as Triple Melt, have a much reduced frequency of freckles or dirty white spot occurrence compared to Double-Melt (VIM +VAR) product. Also, at larger diameters, the sound electrode facilitates control to avoid formation of positive segregation.

![Figure 3 Curves of VAR parameters for φ508mm GH4169 ingots](image)

**Figure 3 Curves of VAR parameters for φ508mm GH4169 ingots**

**Ingot Diameter Increases**

A remarkable sign of melting technology progress for superalloys in China is the increasing of ingot diameter. In order to meet the demand of large and powerful engine or power equipment manufacturing, larger forgings and billets are needed. Therefore ingots with larger diameter are made in China to enable a sufficient forging ratio. Over the past 20 years, Chinese metallurgical plants have brought in numbers of advanced melting equipment such as 12 ton VIM furnace, 5/20 ton PESR furnace and 10 ton Helium-cooled VAR furnace from companies like ALD and CONSARC, which provide good hardware conditions for producing large size superalloys ingot with high quality. On the other hand, a series of technology progresses like Triple-Melt and helium-cooling for VAR have been used to solve the segregation problems for large ingots. So far, the VAR ingot for superalloys with high Nb content like GH4169 have expanded from φ406mm to φ660mm in China; the VAR ingots for Nb-containing alloys like GH4698, and Nb-free alloys such as GH4738/4864 can be made up to φ810mm; the largest VIM+ESR+VAR ingot is for GH4706 with 920mm in diameter as shown in Figure 4. Its ESR electrode is as large as φ1100mm (forged before VAR).
New Melting Techniques
Besides the “mainstream” melting process like Triple-Melt, China has also developed a number of special melting techniques. For example, high purity, low segregation ingot with better plasticity can be produced by ESR-CDS (Continuous Directional Solidification), which is very suitable for the alloys with high Al and Ti content. Figure 5 shows the work principle of ESR-CDS and an ingot (Al+Ti+Nb>8%) produced by this technique [12]. The ingot is continuous drew out during solidification, and its direction is controlled by the temperature gradient of slag pool. With controlled current and proper drew out speed, an optimized microstructure could be gotten as shown in Figure 5b. The direction of columnar crystals is approximately parallel to the ingot axis and the molten pool is quite flat.

Parallel <001> columnar crystal exhibits better thermoplasticity under axial deformation due to the start of {111} slip. In addition, the depth of molten pool for ESR-CDS ingots is only about 10% of the diameter, which can avoid the macro defects like freckle. Furthermore, flat and stable liquid-solid interface is benefit for floating and removing the inclusions.

Vacuum horizontal continuous casting technology can also be used to produce electrode or master alloy. The carbide content is lower than electrode bars made by traditional process due to its characteristic of drawing from the bottom of ingot. Also, the degree of segregation is lower as a result of its fast cooling rate.

Figure 4 φ1100mm ESR electrode and φ920mm VAR ingot for GH4706
Low Cost Melting Process
Ni based or Ni-Fe based Corrosion Resistant Alloys (CRAs) have the same or even greater demand than high temperature superalloys in China’s Oil, gas, chemical and power industries. So that non-vacuumed melting route as Electric Arc Furnace (EAF) + Argon Oxygen Decarburization (AOD)+ESR have also developed in China. It is a particular advantage of the EAF/AOD process that the raw materials used in the process are the least costly of that used in any superalloy melting processes. Scrap may be used without requiring premelt preparation. To a limited degree, oxides of expensive raw materials (such as niobium and molybdenum) may be used to replace more costly pure metal material [2].

Breakdown/ Cogging
Primary hot working operations for superalloys like cogging are directed toward converting cast ingots into mill products and breaking down the as cast microstructure. Superalloys are process-history sensitive. These thermal-mechanical processing cycles can have an important effect on the final properties of the component. Accordingly, for the latest 20 years, finer, more uniform microstructure is always the goal progressing in China for cogging technology. Close to or even the same grain size and properties as final forgings are required for the billets of some essential components.

By now, upset and drew method has been successfully applied to cogging process for GH4169, GH4738/864 and some other alloys. It increases the deformation accumulating with multi-direction, which can effectively promote recrystallization and increase uniformity of the
material. This is especially crucial for bars larger than φ250mm which may lack the forging ratio by ordinary cogging. Furthermore, the upset and draw method also improves the carbide distribution. As shown in Figure 6, carbide cluster become rarer after upset and drew process. It is also found that the ultrasonic testing result is improved using upset and drew. For example, the ultrasonic testing level of φ250mm GH4169 bars can improve from Class A to AA. Generally, either the improvement of microstructure uniformity or the carbide distribution state is beneficial to fatigue properties of the material.

Advanced 1250 ~ 1850 ton GFM radial forging presses were introduced and put into application in almost all the Chinese metallurgical plants in recent years. Surface quality and microstructure at rims both is improved obviously using radial forging, which can result in a significant increased yield for bars. The effect on microstructure uniformity improvement is shown in Figure 7. Radial forging also helps to solve the problem of residual large grain on the surface of final forging products.

![Figure 6 Carbide distribution of φ250mm GH4169 bars produced by cogging without (a) and with upset & drew (b)](image)

In the 1980’s, the most widely used cogging equipment was 2000 ton billetizing press in China. As advanced 3150/4500/6000 ton billetizing presses with high automation level have been put into use, the cogging efficiency increases significantly. Taking φ200mm-φ250mm GH4169 bar products as an example, reheating cycles have been decreased from 7~8 in 1990s to 5~6 in 2000s, then to 3~4 times at present. Meanwhile, thanks to the lower heating temperature, mean grain size for the bars is controlled to “finer than ASTM 6” from the original “finer than ASTM 4”, and some smaller size bars can reach ASTM8~10, which simplifies the subsequent forging process.
Figure 7 Transverse sections of φ250mm GH4169 bars produced by cogging without (a) and with radial forging (b)

Soft Lagging Casing

Compared to conventional Ni-based superalloys with low γ' fraction, the hot working temperature window for “hard-to-deform superalloys” with high Al+Ti content is quite limited. Thereby, effective heat insulation measures are important for cogging operation to prevent surface cracking and large grains at rims. Accordingly, a soft lagging casing method using fiber heat-insulator and glass binder was proposed in China, as shown in Figure 8.

Figure 8 Cogging with soft lagging casing

This kind of “soft” cladding technique is much more convenient than the “hard” canning method using steel sheet or piping. The key points are improvement of adhesion between lagging and ingot, shatterproof properties and how to reduce the harm to workers. So far, “soft” cladding technique has been successfully applied to GH4720Li, and GH4742. With the help of this method, the yield of cogging for GH4720Li can be controlled around 60%.

Die Forging
**Composite Casing**

Just like the cogging process, soft lagging casing is also widely used during die forging in China. In order to prevent it being damaged during transfer and forging, a jacket made by stainless steel plates is used. As demonstrated in Figure 9, the surface, 2R/3 and central temperature of a 340mm diameter and 460mm long billet drops much slower when using the composite casing method (measured by thermocouple). The surface temperature drop is less than 20°C after ordinary transfer and forging process. In addition, this composite casing is also beneficial for lubrication during die forging.

![Figure 9](image)

*Figure 9 Effect of composite casing using in die forging process*

Upsetting and digging center hole have also been adopted before final die forging. This technique helps to improve metal flow behavior, and reduce equipment load and die wearing rate.

**Hot Die Forging and Isothermal Forging**

Besides the composite casing, the hot die forging technique was also applied to reduce the negative effect caused by “cold” die. As shown in Figure 10, structure nearest the cold die exhibited non-recrystallization grains and much delta phase which was reduced significantly for a hot die forged GH4169 disk (die temperature = 900°C).

**Large Presses and Large Forgings**

Since 2010, China has built many large forging presses, such as 80000t hydraulic press in Sichuan, 40000t hydraulic press in Shaanxi, 30000t hydraulic press in Jiangsu, and 35500T high energy screw press in Jiangsu (Figure 11). These advanced presses are all equipped with more precise and reliable control system. For example, the horizontal control error is 0.2mm/m for 80000t hydraulic press. As shown in Figure 12, larger disks have been forged by these powerful presses, like φ1450mm GH4738/4864 disk, φ1200mm GH4169 and φ1200mm GH4698 disk. A φ2000mm GH4706 disk is currently being also developed.
In addition, isothermal forging with induction heating has also been successfully applied to some “hard-to-deformed superalloys” with high Al+Ti content such as GH4720Li. In addition, large-scale disk could also be forged by small presses using accumulated deformations. As shown in figure 11, GH2674 disks with the diameter over 2000mm have been successfully produced by zoning forging using 4000ton press. The analysis results show that, qualified microstructure and properties could be obtained under optimized process control [13]. The mechanical properties with different sampling positions of this large GH2674 disk are shown in Table 4.

Figure 10 Microstructure of a hot-die forged GH4169 disk

Figure 11 Large forging presses built in China
(a) 80000t hydraulic press in Sichuan; (b) 40000t hydraulic press in Shaanxi
Figure 12 Large-scale disk forgings produced in China
(a) φ1450mm GH4738/4864 disk; (b) φ2118mm GH2674 disk

Table 4 Mechanical properties of large GH2674 disk produced by zoning forging

<table>
<thead>
<tr>
<th>Sampling position</th>
<th>Tensile properties at room temperature</th>
<th>670°C, 550MPa stress rupture life τ/h</th>
<th>Hardness /HBW</th>
<th>Impact toughness (A_k/J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rims-1</td>
<td>1050/785/24.0/36.5</td>
<td>1075.9</td>
<td>313</td>
<td>96</td>
</tr>
<tr>
<td>Rims-2</td>
<td>1060/760/25.0/41.5</td>
<td>834.8</td>
<td>298</td>
<td>80</td>
</tr>
<tr>
<td>Rims-3</td>
<td>1080/740/25.0/42.5</td>
<td>608.3</td>
<td>285</td>
<td>86</td>
</tr>
<tr>
<td>Rims-4</td>
<td>1030/750/24.0/36.0</td>
<td>&gt;216.9</td>
<td>298</td>
<td>94</td>
</tr>
<tr>
<td>Radials</td>
<td>1040/745/24.5/39.5</td>
<td>800.3</td>
<td>298</td>
<td>86</td>
</tr>
<tr>
<td>Hubs</td>
<td>1030/765/25.5/36.5</td>
<td>239</td>
<td>298</td>
<td>130</td>
</tr>
<tr>
<td>Cores</td>
<td>1040/760/23.0/36.0</td>
<td>808</td>
<td>298</td>
<td>80</td>
</tr>
<tr>
<td>Standard</td>
<td>≥930/≥610/≥16/≥20</td>
<td>≥100</td>
<td>-</td>
<td>≥39</td>
</tr>
</tbody>
</table>

**Modeling and Simulation**

In addition to the progress of manufacturing technologies, there is another technology that must be mentioned—modeling and simulation technique. In the last 20 years, modeling and simulation methods have developed very fast in China. It has been used for almost all the processes of C&W superalloys productions like alloy design, melting, cogging, die forging, and heat treatment. Modeling methods developed from simple simulations of temperature, strain field or load to microstructure distribution. Therefore, the function of this technique has improved from the problem-analyzing to process-guiding, so as to shorten the product development cycle, lower the costs and improve the product quality. Figure 13 shows cases using FEM simulation for radial forging and die forging.
Summary

In conclusion, the manufacturing technologies of China’s C&W superalloys have been successfully improved greatly in the past 20 years, leading to an improved product quality, which lays foundations for development of the power, energy and other industries, and China’s superalloy products getting into the international market. Of course, it should be recognized that there is still a gap between China and some developed countries in manufacturing technique details and batch production quality and stability for superalloys, so more efforts and works are still underway.

References

173-179.