The influence of microstructure on CTOD fracture toughness of DZ125 base metal and brazed joint

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Abstract: The microstructure of the base metal zone and the weld zone of the DZ125 directionally solidified nickel-based superalloy brazed joint is studied by means of scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) and optical microscope. The CTOD fracture toughness of the base metal zone and the weld zone at 950 °C was tested respectively. The results show that the microstructure of the DZ125 alloy base metal grows with dendrites, the arrangement of the dendrites is close, the divergence angle is small, the dendrites are distributed with a uniform and fine γ' phase, script-shaped MC carbides and petal-shaped γ'/γ eutectic phases are distributed in the inter-dendrite zone. The uniform γ' phase is distributed in the weld zone. At 950 °C, the CTOD fracture toughness of base metal and brazed joint is 0.080 mm and 0.048 mm, respectively. The fracture toughness of the base metal is higher than that of the brazed joint. The fracture morphology shows that the base metal has good toughness at 950 °C, while the weld zone has poor toughness at this temperature. It can be inferred that the long needle-shaped M₆C carbides will weaken the structure performance.

1. Introduction

The microstructure of directional solidification (DS) nickel-based superalloys mainly consists of γ matrix and γ' phases [1]. DZ125 superalloy is one of the DS casting alloys [2] possessing excellent mechanical properties at high temperature and has been widely applied as structural materials for making turbine blades for aero-engines and industry gas turbine [3, 4].

Due to the complexity of the structure of the advanced air engine hollow air-cooled blades, in the manufacturing process, the required structural components are often not obtained by the casting process alone, and vacuum brazing is usually one of its indispensable important processing techniques. The working temperature of the blade is relatively high, about 900 °C-1000 °C, so the high temperature toughness is one of the important indicators to assess the vacuum brazed joint of the blade. The crack tip opening displacement (CTOD) fracture toughness which was first measured by Wells in 1965 [5] refers to the relative distance between the two surfaces opened after the crack tip is subjected to an open load, which reflects the crack tip material's ability to resist cracking. CTOD fracture toughness is an
important parameter for evaluating the brittle fracture resistance of materials and welded joints, and it can also reflect the plasticity of the material. The CTOD test can not only select the toughness of the material, but also provide a test basis for assessing the safety and reliability of the structure.

In recent years, with the rapid development and a large number of applications of DZ125 directional solidification alloys, the researchers mainly focus on the heat treatment process and thermal fatigue properties of DZ125 [6-9]. There are a few studies on the performance and processing technology of the brazed joint of DZ125 alloy, especially the research on its fracture toughness. Brazed joints need to have high strength and toughness [10-12]. Based on the successful development of the DZ125 directional solidification alloy brazed joint in the early stage, the microstructure and CTOD fracture toughness of the base metal and the weld zone were studied respectively. Since further exploring the influence of microstructure on CTOD performance, this study is expected to provide a reliable basis for further research and practical application of the alloy, and also provide technical support for the safe use of materials.

2. Materials and Methods

The vacuum induction furnace (IS65V8 type and ZG-0.05 type) was used to smelt the master alloy. The ISP2 / III-DS type directional solidification furnace was used to prepare the specimen blank by the mobile casting method. The casting molds are all 811A shell type of alumina series. The chemical composition of the DZ125 alloy is shown in Table 1.

| C  | Cr | Co  | W  | Mo | Al | Ti | Ta | Hf | B  | Ni  |
|----|----|-----|----|----|----|----|----|----|----|-----|
| 0.09 | 8.9 | 10.0 | 7.5 | 1.6 | 5.3 | 0.8 | 3.8 | 1.5 | 0.015 | Balance |

The brazing material uses the alloy with the same composition as the base metal. The brazing is performed in a double-chamber vacuum brazing furnace. The vacuum degree during welding heating is better than $2 \times 10^{-2}$ Pa. The metallographic specimens and performance specimens of the joints are all in the form of butt joints. The gap between the brazed joints is 0.1 mm, and the welding direction is perpendicular to the direction of the DZ125 alloy dendrites.

The brazed joint was treated with heat treatment of three steps. First, at the heating temperature of 1230 °C for 3 h, whereas consequently at 1100 °C for 4 h, and finally at 870 °C for 20 h. Every step was followed by air cooling.

Optical microscope (OM, Leica DM4000, Leica Microsystems, Germany) was used to observe the microstructure of different specimens. The morphology of the fracture surfaces was observed by a scanning electron microscope (SEM, JSM-5800, Japan Electron Optics Laboratory Co. Ltd.). The chemical compositions of different specimens were analyzed by an energy dispersive spectrometer (EDS) equipped within the SEM.

In the CTOD test, the base metal and the weld zone are selected as the notched test site respectively. The notch direction of the base metal specimen is perpendicular to the direction of the alloy dendrite. The weld specimen is notched at the core of the weld. The specimen is tested by the compact tensile test specimen, as shown in Figure 1, the specimen thickness B is 4 mm. The initial notch is cut with molybdenum wire with a radius not exceeding 0.15 mm, and the root radius of the cut not exceeding 0.2 mm. The test is carried out in accordance with the BS7448 Fracture Toughness Test Method [13]. The base metal specimen is indicated by BM and the weld specimen is indicated by WS. Three parallel specimens will be used to ensure the accuracy of the test data.
Before the CTOD test, fatigue cracks need to be prefabricated on the notch in order to get a satisfactory result [14, 15]. This test presets the fatigue crack to \( a/W = 0.5 \) at room temperature (\( a \) is the length of prefabricated crack and \( W \) is the effective width of the specimen), and the test is completed on Instron 8801. Firstly, the initial crack is induced by the constant force loading method. At this stage, as the crack expands, the stress intensity factor value increases and the crack propagation rate increases. The maximum force of the precast crack is 3000 N. When the crack propagates to 1.5 mm from the center of the specimen, the constant K force reduction method is used to propagate the crack to the center of the specimen, \( \Delta K = 20 \text{ MPa} \cdot \text{m}^{1/2} \).

The CTOD test was conducted on a Zwick Z100 electronic tensile testing machine. The specimen is clamped in the heating furnace of the testing machine and heated to 950 °C for 30 minutes and then tested. The loading rate is 1 mm / min, and the load (P)-extensometer displacement (V) curve of the specimen is recorded at the same time. When the applied stress passes the highest point and the load-displacement curve drops significantly, the test is stopped. The displacement \( V \) of the extensometer is divided into the elastic part \( V_e \) and the plastic part \( V_p \). The \( V_p \) value is required to calculate the CTOD fracture toughness. According to the standard BS7448, a straight line parallel to the elastic section of the force-displacement curve is made through the maximum force point \( F_m \), and its intersection with the horizontal axis is \( V_p \).

The CTOD is composed of two parts, the first part \( \delta_e \) is the elastic component, and the second part \( \delta_p \) is the plastic component [16]. For compact tensile specimens, the CTOD calculation method is as shown in formula (1) and (2),

\[
\delta_t = \delta_e + \delta_p = \left[ \frac{F_m}{BW^{3/2}} \times f(a/W) \right]^{1/2} \frac{1 - \nu^2}{2\sigma_y E} + \frac{0.46(W-a_0)V_p}{0.46W + 0.54a_0 + z} \tag{1}
\]

\[
f'(a/W) = \frac{(2 + a_0)(0.866 + 4.64a_0/W - 13.32(a_0/W)^2 + 14.72(a_0/W)^3 - 5.6(a_0/W)^4)}{1 - (a_0/W)^{1.5}} \tag{2}
\]

Among them, \( \delta_t \) is CTOD fracture toughness; \( \delta_e \) is a component related to elastic deformation; \( \delta_p \) is a component related to plastic deformation. \( F_m \) is maximum force; \( \nu \) is Poisson's ratio; \( \sigma_y \) is yield strength; \( E \) is elastic modulus; \( V_p \) is plastic opening displacement; \( a_0 \) is initial crack length; \( z \) is blade thickness (blade thickness is 0 in this test). The initial crack length measurement is strictly done according to BS7448.

3. Results and Discussion
3.1 Microstructure of DZ125 alloy

The structure of the DZ125 alloy after heat treatment is mainly composed of dendrites, \( \gamma/\gamma' \) phases, and primary MC carbides. Figure 2 is the metallographic and scanning electron micrograph of the
microstructure of DZ125 alloy. Figure 2(a) shows the low-magnification metallographic structure of DZ125 alloy. From the figure, we can see the obvious dendrite stem and dendrite orientation. The dendrites are closely arranged and the divergence angle is small. Figure 2(b) is a magnified image of the metallographic structure. It can be seen from the Figure 2(b) that the obvious script MC carbides [17] are mainly distributed between the dendrites. Figure 2(c) is the magnified structure of the carbide in the alloy. It can be seen that the carbide is massive.

It could be known from Figure 3 that the carbide phase is rich in C, Ti, Hf, Ta, and the atom percentage content of C and metal is similar, which can be determined as MC carbides. The small γ' phases are distributed at the stem of the dendrites, which are square in shape and relatively uniform in size, with a side length of about 1 µm.

Figure 2(d) shows the morphology of γ/γ' eutectic of DZ125 alloy, which is petal-shaped, and this eutectic phase is distributed between dendrites. The γ' phase in the eutectic is closely arranged in a rectangular shape, and the γ phase in the dendrite is square in shape. MC carbide phases are distributed around the γ/γ' eutectic.

3.2 Microstructure of DZ125 alloy brazed joint

Figure 4 is a SEM image of the DZ125 alloy brazed joint. Figure 4(a) is a low-power photograph of the weld zone. The white phase in the Figure 4 is the carbide phase. It can be clearly seen that there is a band-shaped area at the core of the weld seam, in which almost no carbide phase exists. Figure 4(b) is the microstructure of the core of the weld zone. It can be seen from Figure 4(b) that the weld zone is distributed with a uniform γ' phase, and there is no carbide in the core of the weld zone. There is a heat-affected zone between the core of the weld and the base metal. Figure 4(c) and (d) shows the structure of the heat-affected zone of the brazed joint, in which the carbides are all long needles and are surrounded by the γ' phase. The EDS result of long needle-like carbides is shown in Figure 5.

Figure 2 Microstructure of DZ125 alloy, (a) image of base metal; (b) inter-dendrite MC carbides; (c) MC carbides and (d) inter-dendrite γ/γ' eutectic phase
Figure 3 Chemical compositions of point A

Figure 4 Microstructure of brazed joints of DZ125 alloy, (a) microstructure of brazed joints; (b) weld zone; (c) heat affected zone and (d) M₆C carbide
Figure 5 Chemical compositions of point B

It can be seen from Figure 5 that the contents of Ti, Hf, Ta, W, and Mo are relatively high. It is known that Ti, Hf, and Ta easily form MC carbide, while W and Mo easily form M6C carbide, and M6C carbide is easy to form long needle shape [18], which can be determined that the white phase is M6C carbide. This is because during the welding process, due to the influence of temperature, MC carbides will decompose and generate M6C carbides and γ' phase and the long needle-like M6C phase is surrounded by γ' phase. The reaction equation is as follows[19, 20],

\[ MC + \gamma \rightarrow M_6C + \gamma' \]  

(3)

3.3 CTOD fracture toughness test results

The mechanical properties of DZ125 alloy brazed joint were tested at 950 °C. The results are as follows: yield strength \( \sigma_y = 520 \) Mpa, and elastic modulus \( E = 90 \) Gpa. According to the BS 7448 fracture toughness test method, the CTOD fracture toughness of base metal and the brazed joint is measured at 950 °C. Figure 6 shows the P-V curves of the two specimens of BM-1 and WS-1. As can be seen from Figure 6, the test maximum load \( F_m \) of the base metal and the extensometer opening displacement \( V_p \) are significantly higher than the brazed joint. The calculated average fracture toughness of the CTOD of the base metal is 0.080 mm, and the brazed joint is 0.048 mm, indicating that the fracture toughness of the base metal is better than that of the brazed joint.

Figure 6 The load-displacement curve
3.4 Fracture surface analysis

Figure 7(a) is the fracture morphology of the high temperature fracture toughness test of the base metal. The cracks propagate from left to right, and the specimen surface shrinks. Figure 7(b) is a magnified view of the region I in Figure 7(a). It can be seen from Figure 7(b) that there is a short secondary crack in the dendrite gap at the core of the fracture. Figure 7(c) is a magnified view of region II in Figure 7(b). In Fig. 7(c), obvious dendrite arm characteristics can be seen. Comprehensive analysis can show that the base metal has good toughness at 950 °C.

Figure 8 is the fracture morphology of the high-temperature fracture toughness test of the weld zone. It can be seen from the Figure 8(a) that there is no necking on the surfaces of the specimen, and there are deep and continuous secondary cracks on the fracture surface, indicating that the toughness of the weld zone is poor. Figure 8(b) is a magnified view of the region I in Figure 8(a). It can be seen from Figure 8(b) that there are lots of short secondary crack in the fracture surface. Figure 8(c) is a magnified view of region II in Figure 8(b). It can be seen from Figure 8(c) that the fracture surface has been seriously oxidized and the surface is covered with a layer of oxidation products.

The microstructure of the base metal zone is dendrite structure and the dendrites are closely arranged. The divergence angle is small. The uniform and fine γ' phases are distributed in the dendrite stems and script-shaped MC carbides and petal-shaped γ'/γ eutectic phases are distributed in the inter-dendrite. The uniform γ' phase is distributed in the weld zone, and there is no carbide. The long needle-shaped M₆C carbide is distributed in the heat affected zone where the γ' phase is surrounded. The appearance of M₆C carbides is due to the following reaction: MC + γ → M₆C + γ'.

Figure 7 The fracture surface of base metal, (a) Low magnification image;(b) magnified image of region I and (c)magnified image of region II

Figure 8 The fracture surface of weld zone, (a) Low magnification image;(b) magnified image of region I and (c)magnified image of region II
The measured values of CTOD fracture toughness of the base metal zone and the weld zone at 950 °C are 0.080 mm and 0.048 mm respectively, indicating that the fracture toughness of the base metal is better than the brazed joint. Comparing the fracture morphology of the high temperature fracture toughness of the base metal and the weld zone at 950 °C, it can be seen that the surface of the base metal is necked and there is a short secondary crack in the dendrite gap. There is no necking on the surface of the weld zone, and there are deeper and continuous secondary cracks on the fracture surface. This phenomenon indicates that the base metal has good toughness at 950 °C, and the weld zone has poor toughness at this temperature.

In summary, it can be inferred that the difference in the microstructure of the base metal and the brazed joint plays a key role in the mechanical properties and fracture morphology. The microstructure of the base metal is dendrites, which are closely arranged and have a small divergence angle. The uniform and fine γ' phase are distributed in the dendrites. Script-shaped MC carbide and petal-shaped γ/γ' eutectic phase are distributed between the dendrites, which determines that the base metal has a higher high-temperature fracture toughness test result than the brazed joint, and the fracture has the characteristics of ductile fracture.

The homogeneous γ' phase is distributed in the weld zone, and there is no carbide. The long needle-shaped M6C carbides are distributed in the heat-affected zone and surrounded by γ' phase. It can be inferred that the long needle-shaped M6C carbides will adversely affect the structure, resulting in the decrease of high-temperature fracture toughness of the brazed joint and fracture morphology shows brittle fracture characteristics.

4. Conclusions

This paper studies the difference between the fracture toughness of the base metal and the brazed joint. Based on the above analysis, conclusions can be drawn as follows,

(1) The fracture toughness of the base metal is significantly higher than that of the brazed joint, and the high-temperature fracture resistance of the brazed joint still needs to be improved;
(2) The base metal has good toughness, while the toughness of the weld zone is poor;
(3) The uniform and fine γ' phase, script-shaped MC carbides and petal-shaped γ/γ' eutectic phase will not adversely affect the performance of the DZ125. Long needle-shaped M6C carbide will weaken the structure, leading to a decrease in the toughness of brazed joint.

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