Microstructure and Properties of 3D-GH3625 Electron beam welded

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Abstract GH3625 superalloy is a key part material in the field of aviation, aerospace and chemical industry. The vacuum electron beam docking test was for GH3625 laser 3D printing plate with 4mm thickness. The microstructure and mechanical properties of welded joint under optimum welding process are analyzed. The causes of thermal crack in the heat affected zone of electron beam welding joint of nickel base superalloy were studied. The results show that the existence of low melting eutectic and composition segregation is the main cause of the hot crack. The grain size and grain boundary in the weld are measured by EDS spectrometer, and it is found that Nb elements migrate to the grain boundary during the welding process. The brittle refractory compound is formed with the residual carbon element in the substrate, which reduces the adhesion of the grain boundary and leads to cracking. The microhardness of the welded joint is a symmetrical distribution with the center line of the weld as a symmetry axis, and the whole is "M" shape. The average hardness of the welded joint is higher than the base metal.

1. Introduction

The composition and properties of the GH3625 alloy are very similar to that of the American alloy class Inconel625 alloy [1]. Inconel625 alloys are the International Nickel Company in the 1950s. In order to meet the demand of high strength main steam pipeline materials, a solid solution-strengthened Ni-base deformed superalloy. Inconel625 alloy with Mo and Nb as main strengthening elements has excellent high temperature creep property and rupture property below 650 °C. Oxidation resistance and corrosion resistance [2-4], from low temperature to 1095 °C temperature ranges have good Strength and toughness [5-7]. Inconel625 alloys are often used in piping systems for industrial gas turbines, nuclear power plants, chemical plants and seawater special-purpose equipment [8], and are key industrial components materials such as aviation, aerospace, nuclear energy, petroleum and chemical industry [9-10]. In addition, laser 3D printing technology is mainly used for the preparation of GH3625 alloy parts at home and abroad. Due to the limitation of the domestic laser 3D printing equipment, it is difficult to finish the one-time 3D printing of the workpiece and requires multiple groups of 3D parts to be welded and assembled. At present, there are almost no reports about vacuum electron beam welding technology for assembling 3D printed GH3625 alloy parts. Because of its high energy density [11], high weld penetration [12] and low heat input [13-14], it is especially suitable for the welding of nickel-based superalloys [15]. In this paper, the electron beam butting test of laser 3D printing material GH3625
nickel base superalloy sheet was carried out. By analyzing the microstructure transformation and mechanical properties of the electron beam welded joint, the experimental basis and theoretical basis for the welding and assembling of 3D printed GH3625 alloy parts were provided.

2. Experimental procedure

Laser 3D printing material GH3625 nickel base superalloy sheet is used as test material. The chemical composition of the material is shown in table 1. The size is 60mm×40mm×4mm. The surface of the sample and the butt face were polished with sandpaper before welding to remove the oxidation layer and oil stain on the surface and then cleaned and dried with acetone. The equipment used in welding test is KS15-PN150KM electron beam welding machine made in Germany. After welding, metallographic samples are cut at the cross-section of the weld by wire cutting. The metallographic specimens were corroded by 100mlC2H5OH + 20mlHCl + 5gFeCl3 reagent. OM images of welded joints were observed by XJP-2C optical microscope. The hardness of welded joints was measured by 401MVD type digital display microhardness meter. The load was 200Ns and the loading time was 5s. The nanoindentation test system Tribo IndCenter (Hysitron Company) was used to test the nanoindentation of GH3625 superalloy welded joints using Berkovich indenter. The controlled indentation depth was 2200nm and the pressure down speed was 0.02 nm/s. The Nova Nano SEM450 type energy spectrometer was used to analyze the energy spectrum of welded joints.

| Table 1 Chemical composition of GH3625 (mass fraction, %) |
|----------------|--------|--------|--------|--------|--------|--------|
| C              | Cr     | Mo     | Nb     | Fe     | Al     | Ti     |
| 0.048          | 20.24  | 8.58   | 3.44   | 4.93   | 0.18   | 0.299  |
| Bal.            |        |        |        |        |        |        |

3. Results and Discussion

Through a large number of pre-tests, the welding parameters are determined to be electron beam current \(I_b=25mA\), working distance \(D=300mm\), acceleration voltage \(U=60kV\), focusing current \(I_f=498mA\), welding speed \(V=1200mm/min\). Figure 1(a) is the microstructure of GH3625 base metal. It can be seen from the diagram that the base metal is mainly composed of equiaxed austenite structure and a small amount of black granular carbides are precipitated between austenite crystals. Figure 1(b) and 1(d) are microstructures near the fusion line of GH3625. Compared with the equiaxed structure of the base metal, the inside of the weld is dendritic, and the growth direction of the dendrite is basically perpendicular to the fusion line and grows toward the center. The melting zone near the fusion line forms a relatively small cellular crystal structure, which is narrower and distributes in a band. This is because the metal in the melting pool of the superalloy is first nucleated by the base metal after electron beam welding. However, the initial temperature gradient of nucleation is relatively small, and the crystals after nucleation growth around to form cellular crystals. Along with the solidification process, there is a large temperature gradient along the direction perpendicular to the fusion line, and the grain growth is preferred after partial nucleation, and a large columnar structure is formed along this direction. In this process, impurity elements and low melting point elements in the alloy are pushed to the center of the weld. The columnar crystal intersects, the molten pool is divided into a plurality of small melting pools, resulting in the formation of fine eutectic structure. The small amount of equiaxed structure can also be found at the center of the weld, which is mainly due to the growth of columnar crystals at the edge of the weld. The superheated liquid metal in the center of the weld pool reaches the composition undercooling state after different degrees of heat dissipation and finally grows and forms equiaxed crystals attached to the already formed columnar crystals.

The formation of welded joint cannot be separated from the solidification process of liquid metal. According to the solidification theory, the process of solidification and crystallization of liquid metal in the process of nucleation and growth of crystals, in which nucleation can be divided into homogeneous and heterogeneous nucleation. In the welding process, heterogeneous nucleation dominates the nucleation of molten metal in the molten pool. The nucleation work required by heterogeneous nucleation and the nucleation work required by homogeneous nucleation is \((\Delta G_1^* \text{ and } \Delta G_2^*)\):
\[
\begin{align*}
\Delta G_k &= \frac{16\pi\sigma^2T_m^2}{3L_m^2\Delta T^2} \\
\Delta G'_k &= \frac{16\pi\sigma^2T_m^2}{3L_m^2\Delta T^2} \left( \frac{2-3\cos \theta + \cos^3 \theta}{4} \right) \\
\Delta G_k &= \left( \frac{2-3\cos \theta + \cos^3 \theta}{4} \right)
\end{align*}
\]

In the formula: \( \Delta G_k \) and \( \Delta G'_k \) are the nucleation work required for homogeneous nuclei and heterogeneous nuclei, \( T_m \) is the crystallization temperature of liquid metal theory. \( L_m \) is latent heat of fusion, \( T \) is the degree of supercooling, \( \theta \) is the infiltration angle of the nucleus and substrate. By (1) and (2) it can be seen that the homogeneous nucleation and heterogeneous nucleation of nucleation work \( \Delta G_k \) and \( \Delta G'_k \) a difference between multiples, when infiltrating angle \( \theta = 0^\circ \), the crystallization nucleation work needed to minimum, new phase which is formed by the crystallization can achieve complete wetting between basement, namely between two phase similar crystal structure, growth of new grain can be directed along the surface of the original grain growth [16].

It can be seen from figure 1(a) and 1(d) that there are microcracks in the near heat affected zone of the base metal, and the microcracks are located at the grain boundary. Therefore, the microcracks are taken out for local amplification under the scanning electron microscope, and the morphology of the cracks under the SEM is shown in figure 2. It is known that hot cracks usually occur immediately after welding in the temperature range near the solid line after crystallization. The cracks occur in the center of the weld, arc pits, some in the columnar grain boundary of the weld, and some in the heat-affected zone. It is characterized by intergranular cracking along the grain boundary of the original grain, obtuse of the crack tip, and the surface of the crack may be accompanied by oxidation color. The occurrence of cracks is related to the low melting eutectic of the weld and heat affected zone [17]. The main forms of hot crack are solidification crack and liquefaction crack. Liquefaction crack is a kind of cracking phenomenon caused by the separation of the residual liquid film between grains at high temperature in the seam near zone of base metal or the interlayer metal of the weld seam. Figure 3 shows the diagram of liquefaction crack.
In order to further explore the cause of crack formation, the element measurement of grain and grain boundary in the weld seam is carried out by EDS energy spectrometer. From the comparison of figure 4(b) and the grain boundary 4(C), it was found that Nb element appeared in the grain boundary, which indicated that Nb element migrated to grain boundary during the welding process. In the process of GH3625 welding, the residual carbon elements in the substrate and the alloying elements Nb and Mo are easy to form carbides, which may strengthen the grain boundaries, but the compounds themselves are brittle refractory compounds and reduce the adhesion of grain boundaries. In order to prove that the crack originated from the brittle compound in the grain boundary, the crack source was measured by EDS energy spectrum. Figure 5 shows by comparing the distribution of the four main elements in Cr/Fe/Ni/Mo, it is found that the sharp decrease of Mo content in the crack is the largest, which is consistent with the Nb and Mo elements in figure 4 and the residual carbon elements precipitated at the grain boundary. So the content of Mo in the crack decreased sharply because Mo precipitated at the grain boundary and cracked and peeled off. To sum up, the main reason for the crack is that the intergranular carbide is produced under the high temperature melting state of GH3625, which leads to the inconsistent melting rate of the liquid film and the cracking phenomenon. The crack is liquefaction crack.

Fig. 2 The crack morphology under scanning electron microscope

Fig. 3 Liquefaction crack diagram
Fig. 4 Analysis of elements in Crystal and Grain Boundary
Figure 6 is a microhardness distribution of the cross-section of the welded joint along the center line. It can be seen that the microhardness of the GH3625 welded joint is substantially symmetrical with respect to the centerline of the weld. The hardness value of the welded joint is gradually increased at the upper part and the lower part of the welding joint and the hardness value of the welded joint is gradually decreased when the welding joint is close to the heat affected zone (HAZ). The microhardness curve of the welded joint of GH3625 can be obtained, and when the GH3625 is welded by the electron beam, the welding joint is welded. A certain degree of softening occurs in the central region of the head. The reason why the microhardness of the electron beam welded joints is higher than that of the base metal is mainly due to the finer grains in the joints after the electron beam welding. According to the Hall-patch formula [18], that is:

$$HV = H_0 + KHd^{-1/2}$$

In the formula, the microhardness of HV is the grain diameter, in which $H_0$ and $KH$ are the constants related to the hardness measurement. It can be seen that the microhardness of the electron beam welding joint is higher than that of the base metal due to the finer grain size of the electron beam welding joint.
In order to more accurately distinguish the hardness and elastic-plastic changes of welded joints, nanoindentation tests were carried out on the base metal, heat-affected zone and weld zone. The average values were taken for 5 times in each group. Figure 7 shows the load-displacement response curve. It can be seen from the diagram that the average nano-hardness value of the weld zone is higher than that of heat-affected zone, and the average nano-hardness value of the heat-affected zone is higher than that of base metal. The nanoindentation load-displacement curves of welded joints in accordance with the same trend, and each region is not sensitive to the load rate, and the load-displacement curves of different loading rates are consistent. The load-displacement curve obtained by the test is quite smooth and there is no load abrupt change. In the known nano-indentation test, if the unloading curve coincides with the loading curve completely, it is a completely elastic material, and if the unloading curve is perpendicular to the x-axis, it is a completely plastic material. According to the trend and characteristics of the loading and unloading phase curves, it can be concluded that GH3625 belongs to the plastic material before and after electron beam welding, and the elastoplasticity of the material does not change greatly before and after welding.

4. Conclusions

(1) The base metal is mainly composed of equiaxed austenite structure and a small amount of black granular carbides are precipitated between austenite crystals. Compared with the equiaxed structure of the base metal, the inside of the weld is dendritic, the growth direction of the dendrite is perpendicular
to the fusion line and grows toward the center, and the melting zone near the fusion line forms a relatively small cellular structure.

(2) The microhardness of GH3625 joint is a symmetrical distribution with the center line of the weld as a symmetry axis, and the whole is "M" shape. The average Vickers hardness of the welded joint is higher than that of the base metal, and the hardness value increases gradually between the HV0.2210~265 and the center of the weld but decreases gradually when it is close to the heat affected zone.

References
[1] Feng Di. China material Engineering- Iron and Steel Materials Engineering [M]. Beijing: Chemical Industry Press, 2006: 705.
[2] Evans N D, Maziasz P J, Shingledecker J P, et al. Microstructure evolution of alloy 625 foil and sheet during creep at 750 °C[J]. Materials Science and Engineering A, 2008, 498(1 /2): 412-420.
[3] Rodriguez R, Hayes R W, Berbon P B, et al. Tensile and creep behavior of cryomilled Inconel 625[J]. Acta Materialia, 2003, 51(4) : 911-929.
[4] Mathew M D, Rao K B S, Mannan S L. Creep properties of service-exposed alloy 625 after resolution annealing treatment[J]. Materials Science and Engineering A, 2004, 372 (1 /2): 327-333.
[5] Dinda G P, Dasgupta A K, Mazumder J. Laser aided direct metal deposition of Inconel 625 superalloy: microstructural evolution and thermal stability [J]. Materials Science and Engineering A, 2009, 509(1 /2): 98-104.
[6] Ye Jun. American nickel base superalloy [M]. Beijing: science Press, 1978: 228-241.[7] Narutaki N, Yamane Y, Hayashi K, et al. High-speed machining of Inconel 718 with ceramic tools[J]. Manufacturing Technology, 1993, 42: 103-106.
[7] ZHANG Hong-bin. Inconel 625 alloy progress abroad[J]. Special Steel Technology, 2000(3): 69-80.
[8] Guo Jianting. Study of superalloy materials [M]. Beijing: science Press 2008.
[9] Mittra J, Dubey J S, Banerjee S. Acoustic emission technique used for detecting early stages of precipitation during aging of Inconel 625[J]. Scripta Materialia, 2003(49): 1209-1214.
[10] ZHAO P Z, LIU J, CHI Z D. Effect of Si content on laser welding performance of Al-Mn-Mg alloy[J]. Transactions of Nonferrous Metals Society of China, 2014, 24(7): 2208-2213.
[11] Zhan Xiaohong, Chen Jie, Tao Wang, Yang Zhibin, Wei Yanhong, Chen Yanbin, Qu Wenmin. Properties of a new type of aluminum alloy sheet butt laser welded joint [J]. Journal of Welding, 2012, 12: 3740.
[12] Gao Xiangdong, Yang Yongchen, Zhang Yanxi. Analysis of 3D shape Restoration algorithm for Laser Welding molten Pool Image [J]. Journal of Welding, 2013,11: 58.
[13] Katayama S, Gao X D, Wen Q. Analysis of high-power disk laser welding stability based on classification of plume and spatter characteristics[J]. Transactions of Nonferrous Metals Society of China, 2013, 23(12):3748-3757.
[14] Henderon M B, Arrell D, Larsson R, Heobel M, Marchant G. Science and Technology of Welding and Joining[J]. 2004, 9(1): 13-21.
[15] Ao Sansan, Luo Zhen, Shan Pinget, et al. The Chinese Journal of Nonferrous Metals[J]. 2015, 25(8): 2099-2107.
[16] Li Gang. Microstructure and properties of laser filler wire welding joints for nuclear power dissimilar metals and mechanism of hot crack formation [D]. Shanghai Jiaotong University, 2015.
[17] Xing Li, Song Xiao, Ke Liming. The Chinese Journal of Nonferrous Metals[J], 2014, 07-1714-07.