Effect of Heat Treatment on Microstructure and Mechanical Properties of Inconel 625 Alloy Fabricated by Pulsed Plasma Arc Deposition

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Abstract

Pulsed plasma arc deposition (PPAD) was successfully used to fabricate the Ni-based superalloy Inconel 625 samples. The effects of three heat treatment technologies on microstructure and mechanical properties of the as-deposited material were investigated. It was found that the as-deposited structure exhibited homogenous cellular dendrite structure, which grew epitaxially along the deposition direction. Moreover, some intermetallic phases including Laves phase and MC carbides were precipitated in the interdendritic region as a result of Nb segregation. Compared with the as-deposited microstructure, the direct aged (DA) microstructure changed little except the precipitation of hardening phases $\gamma'$ and $\gamma''$ (Ni3Nb), which enhanced the hardness and tensile strength. But the plastic property was inferior due to the existence of brittle Laves phase. After solution and aging heat treatment (STA), a large amount of Laves particles in the interdendritic regions were dissolved, resulting in the reduction of Nb segregation and the precipitation of needle-like $\gamma''$ (Ni3Nb) in the interdendritic regions and grain boundaries. The hardness and tensile strength were improved without sacrificing the ductility. By homogenization and STA heat treatment (HSTA), Laves particles were dissolved into the matrix completely and resulted in recrystallized large grains with bands of annealing twins. The primary MC particles and remaining phase still appeared in the matrix and grain boundaries. Compared with the as-deposited sample, the mechanical properties decreased severely as a result of the grain growth coarsening. The failure modes of all the tensile specimens were analyzed with fractography.

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1. Introduction

Pulsed plasma arc deposition (PPAD) which combines pulsed plasma cladding with rapid prototyping, is a promising technology for manufacturing near net shape components due to its superiority in cost and convenience of processing. Compared with free arc (GTAW arc), plasma arc has some excellent advantages as a heat source for rapid prototyping, such as high energy density, good stiffness, high stability, small heat-affected area and precise controlling of power source parameters (Zhang H, Xu J, Wang G, 2003; Correa E, Costa S, Santos J, 2008; Zhao W, Liu L, 2006). This enables itself as a good choice for fabricating near net shape components.

Ni-based superalloy Inconel 625 is widely applied in aeronautical, aerospace, chemical, petrochemical and marine industries. This material has a good combination of yield strength, tensile strength, creep strength, excellent process ability, weldability and good resistance to high temperature corrosion on prolonged exposure to aggressive environments (Shankar V, Rao K, Mannan S, 2001; Liu F, Lin X, Yang G, Song M, Chen J, Huang W, 2011). Inconel 625 exhibits these excellent mechanical properties mainly depending upon the solid-solution hardening effect of the refractory metals, niobium and molybdenum in a nickel-chromium matrix (Paul C, Ganesh P, Mishra S, Bhargava P, Negi J, Nath A, 2007; Zhang H, Zhang S, Cheng M, Li Z, 2010; Rai S, Kumar A, Shankar V, Jayakumar T, Rao K, Raj B, 2004). However, the solidification process of cast or welded Inconel 625 is often associated with segregation of high concentration refractory elements, such as Nb and Mo (Thivillon L, Bertrand P, Laget B, Smurov I, 2009). As a result, a brittle intermetallic compound called Laves phase, represented as (Ni, Cr, Fe)2(Nb, Mo, Ti), often forms at the interdendritic regions. Laves phase was reported to be detrimental to the material tensile ductility, fatigue, and creep rupture properties (Song K, Nakata K, 2010; Janaki Ram D, Venugopal R, Prasad R, Madhusudhan R, 2005). So many researches have been done to control the Nb segregation and Laves phase formation in the casts and weld joint. The results presented that the morphology and composition of Laves phase depended strongly on the heat input and the cooling rate of a welding process (Qi H, Azer M, Ritter A, 2009).

However, little work has been carried out using PPAD technology for prototyping Inconel 625 alloy. PPAD process has more complex thermal cycles than the welding. Thus it is difficult to control the Nb segregation and Laves formation. This paper aims to study the microstructure characteristic of the PPAD Inconel 625 alloy. In order to reduce the element segregation, the influences of three standard heat treatment methods (including DA, STA and HSTA) on the microstructures of PPAD Inconel 625 alloy were investigated. The mechanical properties of the alloy in various conditions are compared and correlated with their corresponding microstructure features.

2. Experimental procedure

The forming of Inconel 625 components were carried out by PPAD system, which consists of plasma arc welding source, welding torch, wire feeding device, integrated with welding robot, water-cooled system and computer control system. Commercially available, Inconel 625 welding wire (ERNiCrMo-3) with 1.2mm diameter was used as filler metal. The average chemical composition of the welding wire was 64.24 Ni, 22.65 Cr, 8.73 Mo, 3.53 Nb, 0.01 C, 0.16 Al and 0.2 Ti (in weight %). The substrate was a Q235A plate with dimensions of 200×140×12mm³ (length × width × height). Fig.1a presents the principle of PPAD technology. In the forming process, Inconel 625 welding wire was filled into a molten pool created by pulsed plasma arc, and rapidly solidified onto the previous layer. By adding successive layers, three dimensional components free of cracks and porosity were formed. Fig.1b shows the multilayer sample deposited by PPAD.

Heat treatment is necessary for the precipitation of strengthening phases and improvement of the mechanical properties. The detailed conditions for each heat treatment method are listed in Table 1. The microstructures of the as-deposited and heat treated samples were examined by using a Philips Quant 200 SEM and OLYMPUS optical microscope. The precipitation phases and their crystal structures were determined by means of transmission electron microscopy. The micro-hardness of the deposited samples was measured by a Vickers micro-hardness tester (HXZ-1000) using a 200g load for a dwell time of 15 s. Tensile tests of plate-shaped standard tensile samples were carried out at room temperature on a SANS-5504 tensile testing machine.
Table 1. Three heat treatment processes for Inconel 625 alloy deposited by PPAD

<table>
<thead>
<tr>
<th>DA (Step1)</th>
<th>STA (Step 2→Step 1)</th>
<th>Homogenization +STA (Step3→Step2→Step1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 1 aging</td>
<td>Heat to 720°C, hold for 8h, furnace cool to 620°C, hold for 8 h, air cool</td>
<td></td>
</tr>
<tr>
<td>Step 2 solution</td>
<td>Heat to 980°C, hold for 1h, air cool</td>
<td></td>
</tr>
<tr>
<td>Step 3 homogenization</td>
<td>Heat to 1080 °C, hold for 1h, air cool</td>
<td></td>
</tr>
</tbody>
</table>

3. Results and discussion

1.1. Microstructure characterization

Fig.2 presents the as-deposited microstructure in a cross section vertical to the plasma arc scanning direction. The staggered individual deposited tracks can be observed from the low magnification microscopy, as shown in Fig.2a. The as-deposited microstructure exhibits homogenous cellular dendrite structure, which grew epitaxially along the deposition direction. The transition interface exists between the two adjacent beads or layers. Some columnar dendrites are so large as to cover several deposited layers. The microstructure of the inner region of single pass reveals that a large number of irregular shape phases and some small blocky particles were precipitated in the interdendritic regions, as shown in Fig.2b. The two kinds of phases were identified as Laves phase and NbC particles. Fig.2c highlights the microstructure of the interface region between two adjacent tracks. The heat input and dissipation mode keep consistent when every track was deposited, leading to good continuity for directional solidification characteristics. However, the size and the distribution of Laves phase in the interface exhibit different characteristics from that of Laves phase in the inner region of single pass. As can be seen, the coarse and interconnected Laves particles were precipitated in the inner region of welding track. In contrast, finer and discrete Laves particles were precipitated in the interface region between two adjacent layers, as shown in Fig.2d. During the PPAD process, material is deposited layer by layer. When depositing a layer of material, the dendrites of the top portion always grow preferably inclining towards the scanning direction because of the temperature gradient direction changing from the deposition direction at the bottom of molten pool to the scanning direction at the top of the molten pool. However, the orientation transition of dendrites growth was not observed, resulting from top portion remelting during the subsequent layer deposition. Therefore, dendrite growth directions within two adjacent layers were the same. For the solidification process of the layer interface, the temperature gradient is very high but the cooling rate is very slow. As a result, layer interface consisting of fine cellular structure and precipitates can be observed. Meanwhile, the differences observed in the size and distribution of precipitated phases can be explained on the basis of the differences observed in their dendrite morphologies. The fine dendrite structure presents more but smaller and well-separated interdendritic regions with lower Nb concentration leading to the formation of finer and discrete Laves phase.
Compared with the as-deposited microstructure, direct aging treatment did not result in any perceivable change in the size of the dendrites and the morphology of interdendritic Laves phase. The aging temperatures are designed for the precipitation of $\gamma'$ and $\gamma''$ strengthening phases. However, the temperatures are not high enough to dissolve the Laves phase. Fig.3a and Fig.3b present the bright field image of $\gamma''$ precipitation and its corresponding selected area diffraction respectively. Fig.3c and Fig.3d show the microstructure after solution and aging heat treatment. A large amount of Laves particles in the interdendritic regions were dissolved, resulting in the precipitation of needle-like $\delta$ (Ni$_3$Nb) and MC carbides at the interdendritic regions and grain boundaries after STA treatment. $\delta$ (Ni$_3$Nb) at the grain boundaries is beneficial for improving the ductility and strength. Because the $\delta$-Ni$_3$Nb phase requires less niobium content (about 6-8%) than Laves phase, the transformation indicated that the high concentration of niobium has partially been dissolved into the matrix. $\delta$ (Ni$_3$Nb) phase can not form during PPAD process, because the time for its precipitation is very brief. Although much of the Laves phase was dissolved, solution treatment at 980°C did not make Laves phase be completely dissolved. There are still occasionally some Laves phase particles connecting with the $\delta$ (Ni$_3$Nb) phase remaining in the interdendritic regions. Fig.3e and Fig.3f illustrate the microstructure that went through the homogenization and STA heat treatment. As can be seen, homogenization treatment at 1080°C has completely broken-up the original dendrite structure and resulted in recrystallized grains with bands of annealing twins (see Fig.3e). The almost entire amount of Laves phase has been dissolved into the matrix. It can be seen from Fig.3f that the primary MC particles still remain in the matrix and little $\delta$ (Ni$_3$Nb) phase precipitates in the grain boundaries after the subsequent solution treatment. Although homogenization and STA heat treatment eliminates the segregation of Nb, Mo and Ti elements, it causes noticeable grain coarsening. As a detrimental phase in alloy 625, Laves phase can be dissolved in the matrix when it is exposed to high temperature (Madhusudhan Reddy G, Srinivasa Murthy CV, Srinivasa Rao K, Prasad Rao K, 2009). However, dissolution of Laves phase is rather difficult to achieve as a result of the poor diffusivity of Nb atoms (Janaki Ram DJ, Venugopal Reddy A, Prasad Rao K, Madhusudhan Reddy G, 2005). Thus compared with finer Laves particles, coarser Laves particles with higher Nb concentration require higher temperature and longer time for dissolution. But excessive heat treatment temperature can causes noticeable grain growth which is detrimental to the properties of the PPAD material. Among the three heat treatments, 980°C STA treatment is recommended for the full treatment of the PPAD Inconel 625 alloy.
1.2. Mechanical properties

Micro-hardness measurements show the effects of different heat treatments on the as-deposited samples. The series of points measured are in a straight line from bottom to upper part of each sample. The reported values correspond to the average of all measurements, as shown in Fig.4. Compared with the as-deposited sample, the sample in the DA conditions exhibits higher micro-hardness (324HV). The 980°C STA sample has the highest hardness (342HV) in all cases. Therefore, DA and STA heat treatments have resulted in a significant increase in the hardness levels. However, the hardness of sample in 1080°C HSTA condition is considerably lower than that in as-deposited condition.

The results of room temperature tensile tests conducted on the as-deposited and post heat treated Inconel 625 alloy are listed in Table 2. The as-deposited material produces low tensile strength (771MPa) and yield strength (480MPa) but relatively high plastic elongation (50%). After DA heat treatment, the tensile strength increased by 8% to 833MPa, while the yield strength increased by 3% to 495MPa. However, the plastic elongation of the DA sample decreased significantly from 50% to 38%. The 980°C STA treatment results in slightly lower tensile strength and plastic elongation than wrought material, but it produced much better yield strength. The mechanical properties of the material dropped heavily after 1080°C HSTA treatment.
Table 2. Tensile test results for as-deposited and post heat treated materials

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Tensile strength (MPa)</th>
<th>Elongation (%)</th>
<th>Yield strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-deposited</td>
<td>771</td>
<td>50</td>
<td>480</td>
</tr>
<tr>
<td>DA</td>
<td>833</td>
<td>38</td>
<td>495</td>
</tr>
<tr>
<td>980°C STA</td>
<td>851</td>
<td>44</td>
<td>535</td>
</tr>
<tr>
<td>1080°C HSTA</td>
<td>732</td>
<td>40</td>
<td>449</td>
</tr>
<tr>
<td>Wrought (Asm International Handbook Committee, 1999)</td>
<td>855</td>
<td>50</td>
<td>490</td>
</tr>
</tbody>
</table>

Fig. 5 presents the fracture surfaces of the tested specimens in various conditions. It can been seen from Fig. 5a that the as-deposited fracture surface exhibits a fine dimpled surface, which is the typical characteristic of dimpled ductile mode of failure associated with good plastic elongation. The DA fracture surface exhibits distinct morphology on the fracture surface with coarse Laves particles and MC carbides inside the larger dimples, confirming a transgranular ductile mode of failure (see Fig. 5b). Fig. 5c shows the fracture surface of STA specimen, which exhibits a considerable amount of fine dimples surface, indicating the typical characteristic of ductile fracture associated with good ductility. In addition, the needle-like delta phase and carbide particles are present inside the dimples of the fracture surface of the two STA samples, which illustrates that these particles are the microvoid initiation sites and lead to the fracture. The fracture surface of HSTA specimen consisted of fine shallow dimples and a few of flat surface, as shown in Fig. 5d. This suggests that the failure initiated from the grain boundaries and propagated through coarsened grains.

According to the above observation, it is found that mechanical properties are affected by many factors, such as precipitates, grain size and grain boundary conditions, etc (Liu FC, Lin X, Yang GL, Song MH, Chen J, Huang WD, 2011). Compared with the as-deposited microstructure, DA treatment extensively precipitates fine γ′ and γ″ strength phases in the matrix. Therefore, the micro-hardness and tensile strength are improved. But the Laves phase became the relatively weaker sites, and caused the reduced ductility of the material. The standard STA heat treatment dissolved a large amount of Laves phases and resulted in the precipitation of needle-like delta phase. Meanwhile, with the concentration of Nb element reduced, more Nb elements are used for the precipitation of γ′ and γ″ strength phases. In addition, fine acicular delta phase precipitates at the grain boundaries, which restricts grain boundaries sliding and improves the fracture ductility (Qi H, Azer M, Ritter A, 2009). Thus the mechanical properties were enhanced without sacrificing ductility. After the HSTA treatment, the mechanical properties were severely decreased because of the noticeable grain growth. In conclusion, 980°C STA treatment is recommended for the post heat treatment of the PPAD Inconel 625 alloy.
4. Conclusions

(1) Compared with the as-deposited microstructure, the direct aged (DA) microstructure changed the little except the precipitation of hardening phases $\gamma^\prime$ and $\gamma^{\prime\prime}$ (Ni$_3$Nb), which enhanced the hardness and tensile strength. But the plastic property was inferior due to the existence of brittle Laves phase.

(2) The 980°C STA treatment dissolved a large amount of Laves phases, leading to the precipitation of needle-like delta phase. The mechanical properties of the sample were enhanced without sacrificing ductility.

(3) The 1080°C HSTA treatment resulted in the complete dissolution of Laves phase. But mechanical properties were severely decreased because of the noticeable grain growth

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