The effect of solid solution temperature on the precipitation phase and properties of plasma arc welded joints of Inconel625 high temperature alloy

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Abstract
The effect of solid solution temperature on microstructure and mechanical properties of plasma welded nickel-based high temperature alloy Inconel 625 was investigated by solid solution treatment (The solid solution temperature range is 950 °C to 1150 °C and the intergroup interval is 50 °C). The results show that after solid solution treatment at 950 °C, chain carbides are precipitated at the austenite grain boundary of the base material. With solid solution temperature increasing, carbon chains dissolve gradually at the grain boundary. When solution temperature is higher than 1050 °C, carbides at the grain boundary completely melt back into the matrix. At the weld, dendritic crystal and Laves phase dissolve with the increase of solid solution temperature. When the solid solution temperature is 1100 °C, the dendritic crystal and Laves phase at the weld completely melt into the matrix and transform into cellular crystals. When the solid solution temperature is increased to 1150 °C, the cellular crystal grows further at the weld. When the solid solution temperature is 1100 °C, the comprehensive mechanical properties of the joint are the best.

1. Introduction

Nickel-based high temperature alloy Inconel625 is a kind of Ni-Cr-Mo-Nb system solid solution strengthening alloy with Mo and Nb as the main strengthening elements [1]. This alloy has excellent high-temperature mechanical properties and oxidation resistance, and can adapt to complex stress and atmosphere environment. It is widely used in chemical and industry, nuclear power, aerospace and other fields [2]. Because nickel-based high temperature alloy Inconel625 is rich in Cr, Mo, Nb and other elements, element segregation will occur during hot processing such as welding, forging or rolling [3]. Precipitate harmful second phase and seriously affect the properties of the alloy. Therefore, heat treatment of Inconel25 alloy to reduce the content of harmful second phase has become one of the hot topics in the study of the alloy [3–7].

At present, many scholars at home and abroad have done the corresponding research on the welding and heat treatment of nickel-based high temperature alloy. Bu Xianzeng et al [8] studied the effect of solution treatment on TIG welding Inconel601 joint, It was found that the original organization of the weld was austenitic grains with a large amount of carbide and γ’ phase on the grain boundaries. Gu et al [9] used TIG welding to weld GH3625. Due to the presence of δ phase at the weld, the hardness at the weld of the joint is significantly higher than that of the base metal, and the tensile strength and elongation are significantly decreased. Ramkumar K et al [10] welded Inconel625/Inconel718, studied the microstructure and properties of the dissimilar joint, and found that Laves phase precipitation was observed in the interdendritic region of the two fusion regions. Sukumaran et al [11] found that increasing the solid solution temperature of Inconel625 alloy would decrease the strength and hardness of the alloy and increase the plasticity, which was caused by the changes of the...
microstructure such as carbide dissolution, grain recrystallization and growth. Sridhar et al. [12] studied the effect of different cooling methods on the microstructure of cast Inconel 625 alloy after solid solution treatment at 1090 °C. It was found that Nb-rich carbide precipitates existed on its grain boundaries after water cooling, and chromium carbide precipitation occurred in air cooling or furnace cooling. Wang et al. [13] studied the effect of solid solution temperature on the microstructure of Inconel690 alloy and found that the content of chromium-rich carbide in the alloy decreased with the increase of solid solution temperature. By calculation, the chromium-rich carbide in the alloy was completely dissolved at 1136 °C. Kim et al. [14] conducted heat treatment on surfacing Inconel625 alloy and found that the Inconel625 alloy had austenitic structure after heat treatment. A. Kumar et al. [15] used ERNiCrCoMo-1 as the filler material for tungsten arc welding of P92 and Inconel617 high temperature alloy and found that there was an unmixed zone formed at the interface between the weld area and P92 steel, but no unmixed zone appeared on the Inconel617 side. Vishwa Bhanu et al. [16] used ERNiCrCoMo-1 as filler material for TIG welding of P91 and Inconel800HT high-temperature alloy, and found that due to the high heat input and multiple thermal cycles of TIG welding led to compressive residual stresses in the joint, angular deformation and elemental segregation, the joint fractured in the tensile test on the Inconel800HT side and the joint was ready for practical application.

Previous investigations indicated that the element segregation and harmful second phases would generate at the weld after the Inconel 625 alloy was welded. Thus, the solid solution treatment for the welded joint is necessary. However, most of the investigations about the solid solution treatments for Inconel 625 alloy were only focus on the alloy themselves. And the investigation of solid solution treatments on the welded joint was rarely reported. Thus, in this study, we mainly focused on the effect of the solution temperature for the properties of the joint. Further, the relationship between the properties of the joint and the microstructure also have been discussed. And the results can provide some guidance for the welding processing of the Inconel 625.

2. Experimental methods

Experiment using PM-500 plasma welding machine welding on 4 mm thick Inconel625 alloy plate for self-melting welding. The size of the test plate was 4 × 100 × 200 mm thin plate. The chemical composition of the Inconel625 alloy plate used in the experiment is shown in tables 1, 2 shows the mechanical properties of the receiving material.

| Material     | Tensile strength (MPa) | Yield strength (MPa) | Elongation (%) | Hardness (HV) |
|--------------|------------------------|----------------------|----------------|---------------|
| Inconel625   | 785.4                  | 450.0                | 42.8           | 220           |

Figure 1 shows the Inconel 625 high temperature alloy welding equipment, macroscopic photographs and SEM images. Main welding parameters such as welding current and welding speed are shown in table 3. After the welding is completed, the welded joints are treated with solid solution at 950 °C ~ 1150 °C (temperature interval between groups is 50 °C). After holding for 1 h, air cooling is performed. Then, different samples are taken for observation of microstructure and second phase and mechanical properties experiment. After grinding and polishing, the metallographic sample was wiped and etched to the snowflake corrosion layer on the material surface with a corrosive agent (15 g FeCl₃ + 100 ml HCl), and then the microstructure was observed by Axio Scope A1 optical microscope. WILSONVH1102 automatic Micro-Vickers hardness tester was used to test the hardness of the joint. The spacing of each point was 0.5 mm, the load was HV0.2, and the loading time was 15 s. According to GB/T228-2010, the welding parts were cut and sampled. The tensile test was carried out at the tensile rate of 1 mm min⁻¹ using ASG-X300KN electronic universal testing machine, and the impact test was carried out using JB-750W impact testing machine (impact sample is 10 × 55 × 2.5 mm impact sample). The tensile fracture morphology was observed by SEM after tensile and impact tests.
3. Results and discussion

3.1. Influence of solid solution temperature on joint structure

Figure 2 shows the regional organization of Inconel625 plasma joint. From left to right, the base metal, heat affected zone and weld center are respectively. From figure 2, it can be seen that the original joint base material is austenitic equiaxed crystal. Mainly penetrating twins, a small number of incomplete twins cut off inside the grain. The fusion line area cellular crystal growing perpendicular to the direction of the fusion line, and the weld center is dendrite. The austenite of the base metal does not change significantly when the solid solution temperature is lower than 1050 °C, and the grain in the heat-affected zone does not grow significantly with that of the base metal. Both Gu et al [9] showed similar phenomena when welding GH3625. Austenite grows when solid solution temperature rises to 1100 °C. When the temperature is further increased to 1150 °C, austenite grows abnormally, indicating that solid solution temperature has a significant impact on austenite grain size [12]. Twins still exist in austenite after solid solution treatment, and their morphology is similar to that before solid solution treatment. The dendritic crystal at the weld gradually dissolve with the increase of solid solution temperature. When the solid solution temperature is 1100 °C, the dendritic crystal melt into large cellular crystals. After the temperature was further increased to 1150 °C, the cytosolic crystals grew significantly, and the dendritic crystals at the fusion line also showed similar changes.

3.2. Effect of solid solution treatment temperature on the precipitation of the second phase in each area of the joint

Figures 3 and 4 respectively show the SEM diagram of the base material and EDS spectrum of the precipitated phase of the Inconel625 joint after solid solution treatment. As shown in figure 2, after solid solution treatment

Table 3. Welding process parameters.

| Weiding current (A) | Welding speed (mm s⁻¹) | Welding voltage (V) | Plasma gas flow (L min⁻¹) | Protective gas flow (L min⁻¹) |
|--------------------|------------------------|---------------------|---------------------------|-------------------------------|
| 95                 | 2                      | 30.6                | 3                         | 8                             |

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at 950 °C, chained and massive carbides precipitate inside austenite grain boundaries and crystals [1]. With the increase of solid solution temperature, the second phase carbide gradually melts back into the matrix. When the solid solution temperature rises to 1050 °C, carbon chains at grain boundaries are disconnected, and spheroidization of isolated carbides occurs after carbon chains are disconnected, as shown in figure 4(d). When the solid solution temperature is further increased to 1150 °C, the chain carbides dissolve completely at the grain boundary, and only a small amount of refractory block carbides exist in the grain.

Figures 5 and 6 show SEM images of the weld center and EDS spectra of each precipitated phase at different solid solution temperatures. After solid solution treatment at 950 °C, Laves phase with irregular shape rich in Nb and Mo, large MC type carbides and small M23C6 type carbides exist in the dendrite of weld metal. When the solid solution temperature increases to 1050 °C, the volume of Laves phase in the center of the weld remelts, and MC type carbide and M23C6 type carbide also melt. When the solid solution temperature was further increased to 1150 °C, the Laves phase and small M23C6 type carbides in the center of the weld were completely dissolved, and only a small amount of insoluble MC carbides existed. Relevant literature [17, 18] shows that Laves phase still exists at the dendrite in the center of the weld when the solid solution temperature is lower than 1050 °C, so there is still Laves phase in the weld when the solid solution temperature is lower than 1050 °C.

Figure 7 shows the results of the second phase line scan of the plasma welded joint of Inconel 625 alloy. It can be seen from the figure that the second phase of the welded joint appears to be an aggregation of elements, with the elemental content of Nb and Mo increasing and the elemental content of Ni and Cr decreasing, combined with the previous EDS spectrum analysis to determine the second phase of the laves phase.
Figure 3. SEM images of the whole base material and grain boundary at different solid solution temperatures: (a) and (b) 950 °C, (c) and (d) 1050 °C, (e) and (f) 1150 °C.

Figure 4. EDS results of precipitated phase at grain boundary and in grain in figure 3(b): (a) The chain carbide at point a, (b) The spherical carbide at point b.

Figure 5. SEM figure of weld center at different solid solution temperatures: (a) and (b) 950 °C, (c) and (d) 1050 °C, (e) and (f) 1150 °C.
Figure 8 shows the equilibrium phase diagram of Inconel625 alloy [6] and TTT (temperature-time-transition) curve [14]. It can be seen from figure 4(a), that there are three types of carbides in Inconel625 alloy: MC, M6C and M23C6. When the temperature is 760 °C ~ 950 °C, these three types of carbides will be precipitated in the base material of Inconel625 alloy [4, 19]. Combined with figure 3, it can be seen that after solid solution treatment, Inconel625 alloy will precipitate large Nb-riched MC type carbides (Nb, Ti)C [20] and small Cr-rich M23C6 type carbides Cr23C6.

The dissolution of carbides in alloys is mainly related to element diffusion, and the element diffusion rate is determined by solid solution temperature. As the solid solution temperature increases, the atomic diffusion energy increases and the element diffusion resistance decreases. As the solid solution temperature increases, the carbides in the alloy dissolve more fully [21]. The second phase carbides existing at grain boundaries have a pinning effect on grain boundaries. Chain carbides at grain boundaries can improve the diffusion activation...
energy of grain boundaries and effectively inhibit the growth of austenite in base material [17, 22]. Therefore, the austenite grains of base material will grow significantly when chain carbides dissolve at grain boundaries.

3.3. Influence of solid solution treatment temperature on mechanical properties of joint

3.3.1. Influence of solid solution temperature on tensile properties of joints

Figure 9 shows the location of the fracture and the tensile properties of the joint, figure 10 shows the stress-strain curve for Inconel 625 plasma welded joints, table 4 shows the tensile fracture properties of the joints under different solid solution treatments. It can be seen from figure 9 that the tensile sample breaks at the base metal side when the solid solution temperature is 1100 °C, and the other samples are broken at the weld. The tensile specimens have necking phenomenon at the fracture, and the fracture direction is approximately 45°, with typical ductile fracture characteristics. When the solid solution temperature is lower than 1050 °C, the second harmful Laves phase still exists in the dendrite at the weld. Laves phase is a brittle phase, which can hardly bear plastic deformation, and will become a crack initiation source to promote the fracture of materials in the tensile process [1, 4]. When the solid solution temperature reaches 1100 °C, the Laves phase melts completely at the weld and the sample breaks at the base metal. However, when the solid solution temperature is 1150 °C, the cellular crystal at the weld abnormally grows into coarse cellular crystal, and the weld becomes the weak area of the joint.

When the solid solution temperature is 950 °C, the tensile strength and elongation of the joint are similar to those before solid solution treatment, and the microstructure of the weld is still fine dendritic crystal and there are Laves phase and carbides between the dendritic crystal [12]. When the solid solution temperature is 1000 °C.
and 1050 °C, the tensile strength of the joint decreases but not significantly, while the elongation of the joint increases slightly. This is because with the increase of solid solution temperature, the fine dendritic crystal and Laves phase at the weld gradually melt, leading to the decrease of the tensile strength and the increase of the elongation of the joint \[6, 11\]. When the solid solution temperature was further increased to 1100 °C, the Laves phase at the weld was completely melted, and the welded joint was fractured at the base metal side. The tensile strength of the joint decreased and the elongation increased obviously. When the solid solution temperature increased to 1150 °C, the abnormal growth of cellular crystals further reduced the tensile strength and elongation of the joint.

Figure 11 shows the SEM of tensile fracture of Inconel625 plasma welded joint under different solid solution temperatures. The fracture of the joint at different solution treatment temperatures consisted of a large number of tough nests and tear ridges, but at 1100 °C and 1150 °C the tough nests were smaller and the tear ridges were not prominent, indicating that the mechanical properties of the joint were better at this temperature \[11\].

Table 4. Tensile fracture properties of joints at different solid solution treatment temperatures.

| Solid solution temperature | Stretch image | Fracture location | Joint efficiency % |
|----------------------------|---------------|-------------------|--------------------|
| Welding                    |               | Weld seam         | 97.44              |
| 950                        | 950°C         | Weld seam         | 97.72              |
| 1100                       | 1000°C        | Weld seam         | 96.93              |
| 1050                       | 1050°C        | Weld seam         | 96.51              |
| 1100                       | 1100°C        | Base material     | 94.58              |
| 1150                       | 1150°C        | Weld seam         | 91.84              |

When the solid solution temperature is lower than 1050 °C, the fracture characteristics of welded joint are similar, including a large number of dimples. The dimples are relatively uniform in size, and the shape is regular with certain directionalness. The fracture mode of the joint is ductile fracture. When the solid solution temperature was 1050 °C, the tear ridges at the fracture became denser and more prominent with the increase of solid solution temperature, indicating that the toughness of the joint would increase with the increase of solid solution temperature. When the solid solution temperature gradually increases to 1100 °C and 1150 °C, the fracture morphology of the sample changes significantly. At this time, there are many large and deep dimples in the fracture, and many small dimples are distributed around the dimples, indicating obvious ductile fracture characteristics, and the ductility of the fracture is better than that of the tensile fracture at low solid solution temperature. The appearance of large and deep dimples at the fracture is due to the separation of the material and the formation of cavities, which gradually grow larger under the action of slip and connect with other cavities to form large and deep dimples \[17\]. However, the number of large and deep dimples in the fracture at 1150 °C is reduced compared with that at 1100°C, indicating that the elongation of the sample at 1150 °C is lower than that at 1100 °C.
3.3.2. Impact of solid solution treatment on joint impact toughness

Figure 12 shows the macro impact sample and impact performance of Inconel625 joint. It can be seen from figure 12 that the impact energy of the weld decreases when the solid solution temperature is 950 °C compared with that of the original joint. At this time, there is massive carbide precipitation at the weld, which leads to the reduction of the impact energy of the weld. When the solid solution temperature increases, the fine dendritic crystal and Laves phase at the weld gradually melt, and the impact energy at the weld gradually increases. At 1100 °C, the Laves phase at the weld melts completely, dendritic crystal change into cellular crystals, and the weld impact energy increases obviously. When the solid solution temperature is increased to 1150 °C, the cell grain of welding seam grows abnormally, and the impact energy decreases compared with 1100 °C.

Figure 13 shows the SEM of impact fracture of each part of Inconel625 joint at different solid solution temperatures. From left to right, the base metal, heat affected zone and weld core zone are in sequence. When the solid solution temperature was increased, the hard and brittle precipitation phase in the joint decreased, the
impact performance increased, a large number of tough nests appeared on the fracture, and the joint showed the characteristics of ductile fracture [23]. When the solid solution temperature is 950 °C, the fracture microstructure of the impact sample at the weld is mainly a large number of dimples, and there are a large number of tear ridges around the dimples, which show better impact performance compared with the base metal and the heat affected zone. When the solid solution temperature is higher than 1100 °C, there are a large number of evenly distributed and deep dimples in the weld, indicating that the impact toughness of the weld is good. However, compared with 1100 °C, the dimple of impact fracture at 1150 °C is relatively small and the impact toughness is relatively poor. At different solid solution temperature, the weld is ductile fracture.

3.3.3. Distribution of joint micro-hardness after solid solution treatment
Figure 14 shows the distribution of joint hardness. With the increase of solid solution temperature, the joint hardness gradually decreases, and the hardness at the joint weld is gradually close to that at the base metal. At 1150 °C, there is little difference between the hardness of joint weld and base metal. The joint hardness decreases with the increase of solid solution temperature mainly because of the phenomenon of second phase melting and grain growth. Grain grew up without a significant before 1100 °C, but the second phase has already begun to melt, to 1100 °C when the second phase to melt completely, seam dendritic crystal into cellular crystal, both interaction makes joint hardness drops, continue to rise after solid solution temperature to 1150 °C, welds already completely melt, the second phase is 1100 °C had no obvious effect on joint hardness, But at this time, the cellular crystal at the weld is further longer and the joint hardness is further reduced.
4. Conclusion

(1) Solid solution treatment can effectively improve the performance of Inconel625 plasma welded joint and reduce the content of the second phase in the joint. When the solid solution temperature is too high, the grains grow abnormally and the mechanical properties decrease. With the increase of solid solution temperature, the mechanical properties of the joint increase first and then decrease, and the content of second phase and grain size are the main factors affecting the joint properties.

(2) With the increase of solid solution treatment temperature, the austenite of the base material of Inconel625 plasma welding joint gradually grows up, and dendritic crystals at the weld gradually dissolve into cellular crystals, and all of them dissolve into cellular crystals at 1100 °C. The cellular crystals further grow up with the increase of solid solution temperature.

(3) When the solid solution temperature is 950 °C, M23C6 carbide is precipitated at the weld, and chain carbide is precipitated at the grain boundary of the base material. When the temperature rises to 1050 °C, carbide and Laves phase begin to melt. When the temperature reaches 1150 °C, Laves phase is completely melted at the weld and only a small amount of unmelted MC type carbide is found. Only a few lumps of insoluble carbide remain in the base metal.

(4) The optimal solid solution temperature of Inconel625 plasma welded joint is 1100 °C. The tensile fracture of the joint was at the base metal, and the strength was 742.8 MPa, which reached 97% before solid solution treatment. The elongation was 43.1%, significantly higher than 31.9% before solid solution treatment. The impact energy at different positions was significantly higher than that before solid solution treatment. Both tensile and impact fracture modes are ductile fracture. The joint hardness is lower than that before solid solution treatment.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
Declaration of interest statement

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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