Welding procedure Research of ASTM B708 R05252 tantalum sheets

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Abstract—The weldability of ASTM B708 R05252 tantalum sheet was analyzed, and the mechanical properties, bending properties and corrosion resistance of tantalum sheet were tested by GTAW butt welding. Test results show that butt joints by this welding procedure perform good mechanical property, and the metallographic microstructures &corrosion resistance of the welding joint are normal. It expresses the great significance of formulating the welding procedures of tantalum pressure vessels.

1. Weldability Research of Tantalum Pressure Vessels
A batch of pressure vessels order for waste sulfuric acid concentrator have been received by a company in Shanghai, including tantalum liner containers such as vaporizer, tantalum tube heat exchanger, tubular heater, clasp heater and condenser. ASTM B708 R05252 tantalum sheet has been widely used in design of these pressure vessels. The company intends to conduct weldability test and welding procedure qualification for the tantalum sheet before product manufacturing process.

ASTM B708 R05252 Tantalum sheet is ASTM brand material (TaW2.5 according to GB/T 3629-2017), tantalum 97.5%- Tungsten 2.5% (Ta-W2.5), belongs to tantalum-tungsten alloy. Tantalum is a refractory rare metal with characters of high density, low expansion coefficient and good high-temperature strength and machinability. Due to the corrosion resistance of tantalum and the high melting point of tungsten, the product is mainly used as heater and high-temperature operating equipment in corrosive environment.

Tantalum-tungsten alloy is good performance of corrosion resistance in most inorganic acids, and similar to glass, which has an important use in the sulfuric acid industry. In addition to hydrofluoric acid, fluorine, fuming sulfuric acid and alkali, almost all chemical media can withstand the corrosion, and its service life is dozens of times longer than stainless steel[1].

Tantalum sheet is expensive, and so its application forms are mainly composite plates and linings. In order to reduce the cost, the thickness of tantalum layer is usually designed to be as thin as possible.
Composite plates or lining welding is very difficult, because the melting point of tantalum material and steel is six to one: tantalum melting point is 2996℃, steel melting point is 1400℃. In welding process, Tantalum is often not melting, when steel underneath has become the molten iron. Therefore tantalum sheet is often designed to tantalum lining pressure vessel in actual production, and lining layers can also be connected to the steel surface with tantalum screws, or brazing. R05252 tantalum sheet has stable performance at room temperature, and when heated, it can be combined directly with non-metallic elements. When Tantalum sheet heated to 200~300℃, micro-oxidation occurs, and when heated to above 500℃, rapid oxidation occurs[2].

Tantalum tungsten alloy can be welded by inert gas shielded welding, electron beam welding, brazing, the way of self-fusion welding or sheet cutting wire as welding wire[3]. The GTAW procedure with sheet cutting wire is used on ASTM B708R05252 tantalum plate under current conditions of the company. The cooling rate of tantalum-tungsten alloy weld is low, which reduces the ductility of the weld and leads to welding cracks. Therefore, the mode of front and back argon gas protection and low thermal input is adopted, which makes the deposited metal not touch air, not easy to produce brittle compounds, and reduce the tendency of thermal cracks. There is no manufacture standard for tantalum pressure equipment in China at present, so the GTAW procedure shall be demonstrated to meet the design requirements according to the code TSG 21-2016.

2. Test Conditions of ASTM B708 R05252 Tantalum Sheets
In the waste sulfuric acid concentrator, the thickness of tantalum liner sheet T is no more than 2 mm. The thickness of tantalum sheet is chosen as 2mm in this welding test, and the welding procedure evaluation of ASTM B708 R05252 tantalum sheet is covered the thickness not more than 4mm of the weld. The test tantalum sheets are welded by GTAW with sheet cutting wire, and the mechanical property, bending property and corrosion resistance are tested and verified, as shown in Figure 1.

![Fig.1 the welding test tantalum sample](image)

2.1. Groove form
The welding groove type of tantalum sheets is mainly determined by joint position, sheet thickness, welding method and requirements of corrosion resistance. For tantalum sheets with a thickness of less than 4mm, I-shaped groove should be selected as far as possible in butt welding design to reduce consumption of welding materials and welding deformation[3]. The process of drag cover and pad protection welding with gas lens is chosen in GTAW. The sample assembled and aligned in this welding test is shown in Figure 2.

![Fig.2 assembled and aligned picture of the welding test sample](image)
2.2 Chemical composition and mechanical properties of Tantalum sheet

The chemical composition of ASTM B708 R05252 Tantalum sheet used in the test are shown in Table 1, and the mechanical properties of tantalum sheets are shown in Table 2.

H, O, N and other gas may be absorbed by ASTM B708 R05252 tantalum sheet from the environment at high temperatures and formed brittle compounds. A small amount of gas impurities can significantly affect the mechanical properties and corrosion resistance. Thus it should be of high purity of tantalum material used for welding, and the content of impurities such as H, O, N and other impurity should be strictly controlled in welding material and welding process. Before welding, welding grooves and 30 mm area on both sides of the grooves should be mechanical cleaned strictly, degreasing processed and erased scale, oil, and fibers carefully on the surface of the weld by acetone to ensure the welding material and welding groove surface clean [4].

Tab.1 chemical composition of the ASTM B708 R05252 tantalum sheet, %

| Elements | Material | Ta | Fe | Si | Ni | W | Mo | Ti | Nb | O | C | H | N |
|----------|----------|----|----|----|----|----|----|----|----|----|----|----|----|
| ASTM B708 R05252 margin | 0.00 | 0.00 | 0.00 | 2.4 | 0.01 | 0.00 | 0.15 | 0.01 | 0.00 | 0.01 | 0.01 |

Tab.2 mechanical properties of the tantalum sheet

| Items | Mechanical properties |
|-------|-----------------------|
|       | $R_{0.2}$ (MPa) | $R_m$ (MPa) | $\delta$ (%) | $A_k$ (J) |
| Properties | 228 | 407 | 40 | / |

When annealed tantalum sheet is welded, the cooling rate of tantalum-tungsten alloy is low, and tendency of heating crack is increased. Therefore, the grain size of the tantalum plate for testing shall be taken into account in welding test, and fine grains with size less than or equal to ASTM grade 5 should be adopted. The grain size of tantalum sheet in this test is grade 3.

2.3 Selection of welding materials

The shearing wire of tantalum sheet is used as welding wire in this GTAW process, and the shear wire is 2×2mm size of ASTM B708 R05252 tantalum sheet in the same furnace as the welding wire, so as to avoid impurity of the welding seam and reduce its performance. The grain size of weld and heat-affected zone can be refined by using the pulsed current inert gas welding with tungsten electrode transverse oscillation, and the grain size of weld can be changed from columnar to equiaxial, which improve the plasticity of welded joint significantly and avoid excessive

2.4 Welding process parameters

GTAW welding process parameters of ASTM B708 R05252 Tantalum sheet are shown in Table 3. It is prefer to the welding process of small tungsten electrode diameter, small heat input and multi-pass welding when GTAW welding of tantalum sheet is used. For type I joint, DCEN is preferred, and convex weld bead slightly is preferred [3].

Tab.3 technological parameters of GTAW process

| Welding process | Weld layers | Current type& polarity | Current A | Voltage V | Travel speed cm/min | Tungsten electrode diameter (mm) | Argon flow rates L/min | main burner | Additional protection |
|-----------------|-------------|------------------------|-----------|-----------|---------------------|-------------------------------|----------------------|-------------|----------------------|
| Manual GTAW     | 1           | DCEN                   | 120 ~150  | 9~12      | 24~28               | Φ1.0                         | 10 ~ 15              | 20 ~ 30     |                      |
|                 | 2           | DCEN                   | 120 ~150  | 9~12      | 24~28               | Φ1.0                         | 10 ~ 15              | 20 ~ 30     |                      |
|                 |             |                        |           |           |                     |                               |                      |             | No preheating, interlayer-temperature≤100℃ |
It is not only necessary to adopt strict technological measurements in cutting, groove processing, forming and other procedures, but also have special requirements of the welding environment, pre-welding cleaning and welding protection due to the welding characteristics of tantalum materials\cite{5}. At first, the welding site should be an independent area to keep the environment clean and prevent smoke, oil and wind. Secondly, argon gas with purity≥99.99%, special protective towing cover and double-sided ventilation protection are used to avoid oxidation. The high-temperature residence time of the welded joint should be shortened as far as possible during welding, and the welding area should be protected strictly not only in the welding process, but also in the post-welding cooling in order to ensure the corrosion resistance of the weld.

2.5 Post-weld heat treatment
In order to reduce the risk of corrosion failure in service, tantalum sheet welding samples were machined into 2 pieces for simulated post-welding heat treatment according to the manufacturing heat treatment status of all bearing parts. The welding sample was divided into two types: 1# sample maintained the as-welded state as same as shells, 2# sample underwent vacuum annealing treatment at 1200°C as same as heads, and the holding time was 1h.

3. Test results and analysis
Welding process in table 4 is adopted for tantalum sheet welding, in which 1# tantalum sheet welding sample remains in the welding state, and 2# tantalum sheet welding sample is subjected to post-welding annealing treatment. The following tests are carried out on these tantalum sheet welding samples by referring to the standard of NB/T 47014-2011.

3.1 Weld joint performance test
The results of welded joint performance test are shown in Table 4 for this test condition.

| Samples NO. | Nondestructive testing (RT+PT) | Visual inspection | Tensile strength $R_m$ MPa (The fracture is located in the heat-affected area) | side bend (4 groups) D=40mm (180°) |
|-------------|--------------------------------|------------------|---------------------------------------------------------------------------------|-------------------------------------|
| 1           | Qualified                       | Good, weld metal is silver - white | 397.6, 432.4                                                               | Intact, no cracks observed          |
| 2           | Qualified                       | Good, weld metal is silver - white | 381.3, 412.1                                                               | Intact, no cracks observed          |

The mechanical properties and bending properties of 1# and 2# samples can satisfy NB/T 47014-2011 Welding Procedure Qualification for Pressure Equipment in Table 4. The fracture strength of the tensile samples is not high, but their fracture sites are consistent in the heat-affected zone near the edge of the weld. And the structure of the heat-affected zone is the large recrystallization structure, which will lead to the crack and fracture of the tensile stress. The hardness test results in Table 5 show that there is little difference between the hardness values of 1# and 2# samples at the same point. On the whole, the hardness value of base metal < heat-affected zone < weld, but there is no big fluctuation. The microstructure hardness change of the welded joint reflects the microstructure change of each part of the joint to some extent. There is no obvious microstructure embrittlement and degradation occurred in the heat-affected area of the two samples, which is favorable to the whole microstructure and mechanical properties.
Tab.5 hardness test of the welded joint

| Samples NO. | Weld spot position | Hardness (HV0.2/30) |       |       |
|-------------|--------------------|---------------------|-------|-------|
|             |                    | base metal          | heat-affected zone | weld  |
| 1           | upper surface      | 69, 71, 68         | 78, 78, 75        | 92, 98, 91 |
|             | reverse surface    | 66, 66, 69         | 74, 80, 82        | 90, 83, 88 |
| 2           | upper surface      | 66, 62, 60         | 71, 75, 67        | 84, 89, 90 |
|             | reverse surface    | 59, 63, 64         | 66, 66, 68        | 79, 74, 85 |

3.2 Metallographic examination

The welding process can affect directly the macro and micro structures of the weld metal and the heat-affected zone, as well as the welding defects and the performance of the welded joints. The microstructure and welding defects of the welded joints can be inspected by the metallographic examination of the welded joints. After cross-section of samples polished, the weld microstructure of R05252 tantalum sheet by GTAW has no obvious defects such as cracks, incomplete welding, and non-fusion, as shown in Figure 3 (a) and (b). After eroded by aqua regia solution, metallographic structures were observed as Ta-W solid solution by magnified 200 times. The grains of weld microstructure were grew up obviously, which displayed as the cellular dendrites, and the heat affected zone on both sides of the weld displayed as recrystallization organization. There are no microscopic cracks and other defects in the weld zone and heat-affected zone, as shown in figure 3 (c) ~ (f).

Fig.3 macrostructures and metallographic structures of the welding test samples
3.3 Corrosion resistance test

Intercrystalline corrosion inspection is required to evaluate the corrosion resistance of the welded joints because tantalum sheet welds are in contact with corrosive media. According to design requirements, 1# and 2# samples were grinded and polished, and then corroded in boiling ferric sulfate solution for 120h. The intergranular corrosion test of samples was carried out by referring to GB/T 15260-2016 Standard method A ferric sulfate -50% sulfuric acid method according to the design requirements. The test results showed that no cracks were found on the outer surface of No.1 and No.2 samples after bending, and they all qualified the intergranular corrosion test, as shown in Table 6.

| Tantalum sheet      | Sample No. | Test procedure                  | Heart bending diameter | Angle of bend | Results |
|---------------------|------------|---------------------------------|------------------------|---------------|---------|
| ASTM B708 R05252    | 1          | referring to GB/T 15260-2016    | 12mm                   | 180°          | qualified |
|                     | 2          |                                 | 12mm                   | 180°          | qualified |

4. Conclusions

In this paper, the weldability of ASTM B708 R05252 tantalum sheet by GTAW method on tantalum pressure vessel is analyzed. The mechanical properties, bending performance and corrosion resistance of the weld is analyzed through welding performance testing of R05252 tantalum plate after the post weld heat treatment of as-weld and annealing states. The results show that the GTAW procedure can get the butt joint with good performance, the metallographic structures of the welded joints is normal, and the corrosion resistance performance is qualified.

In the manufacture process of ASTM B708 R05252 Tantalum liner pressure vessels with thickness of 2mm, GTAW procedure as shown in Table 3 is adopted. The outcome of vessel inspection and performance of test pieces for welding can satisfy the design requirements. The test results show that proper structure design, reasonable process parameters and effective welding protection measures are the key factors to ensure the welding quality of the thin layer tantalum liner of pressure vessel. It had the great significance of the successful welding procedure qualification to guide the welding procedures and actual manufacture of tantalum pressure vessels.

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