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## Effect of Surface State on Acid Rain Corrosion Resistance of T6 6005A Aluminum Alloy by BT-FSW Joint

## Zhiheng Qin<sup>\*</sup> and Hongji Xu

School of Materials Science and Engineering, Dalian Jiaotong University, Dalian, Liaoning Province, 116028, China

<sup>\*</sup>Corresponding author: E-mail: 494905023@qq.com

Abstract. The test sample was 5 mm thick artificial aging 6005A aluminium alloy profile, which was butted by BT-FSW. This work aimed to analyse the effect of surface state on corrosion resistance of welded joints in acid rain environment based on acetate ssalt spray test (CASS), polarization curve and EIS impedance test, combined with SEM scanning and EDS energy spectrum. The results showed that the corrosion rate of pre-welded polished joints was high at the initial stage of salt spray corrosion, and the corrosion rate of both joints were equal at the later stage of corrosion, higher than that of base metal. The best preservation of corrosion morphology was THAZ, followed by nugget zone and HAZ, and the pitting degree of base metal was the most serious. The polarization curves of the polished joints before 30 min corrosion were located at the lower right of the image, and its self-corrosion current density in the nugget zone showed the worst corrosion resistance. The corrosion resistance of the pre-welded polished joint increased after 336 hours of corrosion, the corrosion breakdown potential and self-corrosion current were not significantly different from those of the unpolished joints. The fitting curves of the two kinds of joints had similar dual capacitance arc resistance characteristics and the impedance parameters in EIS, and the electrochemical corrosion resistance of the two joints were weaker than that of the base metal.

#### **1. Instruction**

6005A is Al-Mg-Si heat-treppable aluminum alloy, which has been applied in batches in the field of rail vehicles. Its production process has realized the effect of refining the grain by increasing the content of Cr and Mn elements, effectively inhibiting the occurrence of recrystallized grains during hot processing of extruded profiles[1]. After the introduction of the FSW technology, bringing about tremendous changes, and its joint performance of aluminum alloy has been greatly improved. Bobbin Tool Friction Stir Welding (BT-FSW) is used as a plastic joining method. It is widely used in the welding of aluminum alloy thin plates, hollow siding and structural profiles, as a supplement to the traditional friction welding. Therefore, the 6005A aluminum alloy is an ideal connecting material for the side wall of rail vehicles[2]. However, its application is limited by environmental factors such as humidity, temperature, pH, atmospheric acid ion concentration and strong oxidizing gas that make the corrosion law of the joint become elusive. There have been many available reports on the corrosion resistance of aluminum alloy in China. Shenghai et al[3] studied the effect of pH on the electrochemical properties of 2024-T351 aluminum alloy. It was found that the stability of the oxide film decreased with the decrease of pH. When pH below 3, the oxide film basically lost its ability to protect the substrate. Li Tao et al[4] studied the 2A12 aluminum alloy dynamic electrochemical

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI. Published under licence by IOP Publishing Ltd 1 impedance spectroscopy (EIS) by dry and wet weekly immersion test. The scanning results showed that increasing the concentration of Cl<sup>-</sup> promoted the formation and development of pitting corrosion. The pre-welded polished treatment of traditional MIG welding is to remove the oil stain and oxide film on the surface of the sheet, avoiding the pores and inclusion defects of the joint. At present, for the 6 series aluminum alloy profiles BT-FSW joint, the different surface polishing state joints in the acid rain atmosphere corrosion behavior remains to be studied. In this paper, the corrosion behavior and mechanism were studied under environment of simulated acid rain on the artificially aged (T6) 6005A profiles' BT-FSW joints by static weight loss, salt spray test and potentiodynamic polarization curve.

## 2. Research methods

#### 2.1. Test materials

The test material is 5 mm thick 6005A aluminum alloy profiles with T6 artificial aging. The pre-welded polishing adopts the method of thousand-blade grinding. The chemical composition is shown in Table 1, and welding parameter is shown in Table 2.

			-					
Brand	Si	Fe	Cu Mn	Mg	Cr	Zn	Ti	Al
6005A-T6	0.59	0.18 <	≤0.09 ≤0.12	0.57	≤0.1	0.02	0.01	Bal.
Table 2. Process parameters of BT-FSW								
Stirrin	g needle siz	ze (mm)	Inclination	Angle	Friction	Weld	ing	Pressing
Needle	Upper	Lower	( )	( )	speed	spee	ed	amount
					(rpm)	(mm/I	<u>min)</u>	(mm)
4.3	23	20	0	0	400	300	0	0.7

Fable 1. Chem	nical compositio	n of 6005A-T6(m	ass fraction, %)

## 2.2. Salt spray test

According to the standard GB/T 10125-2012, the KLT-2008 test chamber is used for acetate ssalt spray test (CASS test) to test the corrosion resistance of the joint specimen under the simulated industrial atmospheric acid rain corrosion environment. The test chamber temperature is set to 50 °C, using continuous spray method. The concentration of 1 mol/L NaCl+0.26 mol/L CuCl<sub>2</sub> salt solution was added to the brine tank, and the pH value of the solution was adjusted to 3.0 by adding appropriate amount of glacial acetic acid. Each test piece' size was 70 mm×70 mm×5 mm and is weighed before welding. Before the corrosion test, the non-corrosive surface of the surrounding and back sides is sealed with epoxy resin. The test period contiances 24h, 48h, 72h, 96h, 120h, and 240h. Three parallel test pieces are selected for each cycle. After reaching the specified test period, the test pieces are recorded the weight after ultrasonically cleaned, epoxy resin degreased and anhydrous ethanol dehydrated. The weight loss rate and corrosion rate are calculated for drawing the curve of weight loss. Finally, the microscopic corrosion morphology of the specimen surface was observed by SUPRA 55 field emission scanning electron microscope.

## 2.3. Electrochemical test

According to the standard GB/T 24196-2009, two kinds of surface state joint potentials polarization curve (Tafel curve) and electrochemical impedance spectroscopy (EIS) were measured using CorrTest electrochemical workstation. 0.6 mol/L NaCl+0.01 mol/L NaHSO<sub>3</sub>+0.01 mol/L NaHCO<sub>3</sub>+0.01 mol/L NaNO<sub>3</sub> salt solution was used as the corrosion medium to simulate the acid rain environment with pH less than 4.0 at room temperature. The size of test sample was 10 mm×10 mm×5 mm. Before electrochemical test, polishing with metallographic sandpaper (1000 mesh) to removing the surface oxide film to be corroded to uniform roughness. The test corrosion cycle is immersion corrosion for 30 min short-term and 336 h long-term immersion corrosion, using a typical three electrode system (working electrode, SCE saturated calomel electrode and platinum plate assisted electrode),

polarization ranges -2.0V~0V, scanning speed is 1.68mV/s. When measuring EIS impedance spectrum, the scanning frequency range selects 0.01 HZ $\sim 10^5$  HZ, test potential selection the open circuit potential, the peak value of the sinusoidal disturbance signal is 5mV, and the experimental data is finally processed by Zsimpwin.

#### 3. Test results and analysis

#### 3.1. Salt spray test

After 240 hours salt spray test, the T6 state 6005A welded joints parent metal area was severely corroded, and the surface was evenly distributed densely. The corrosion production was stacked in a block shape. At this time, the corrosion pit had a tendency to develop into a large piece of peeling pit and could be observed obviously in the joint surface. Starting from the junction of THAZ and HAZ on both sides, forming a fine etch pit of the contiguous. The pit distribution had a linear distribution of the second phase, indicating that the second phase particle Mg<sub>2</sub>Si was a sensitive position of the etch[5]. The corrosion of the retreating side of the THAZ was lighter than that of the advancing side. The number and depth of the pitting pits on Nugget zone of the two kinds of joints were significantly smaller than those of parent metal and HAZ, indicating that the corrosion resistance of BT-FSW welded joints was higher than that of parent metal.

Each Surface State test piece takes 3 parallel test sample, and the average corrosion rate is calculated according to the following formula:

$$\upsilon = \frac{m_0 - m_1}{st} \tag{1}$$

$$\overline{\upsilon} = \left(\sum_{i=1}^{i=3} \upsilon_i\right) \frac{1}{3} \tag{2}$$

Where v is the corrosion rate of a single test piece,  $m_0$  is the original weight of the test piece,  $m_1$  is the weight after corrosion of the test piece, s is the area of the corrosion surface, t is the corrosion time,  $\overline{v}$  is the average corrosion rate of the piece. The results of parent metal and welded joints corrosion weight loss rate are shown in figure 1.

It can be seen from Figure 1(a) that the corrosion weight loss rate of parent metal was small and the pre-welded polished joint was the highest within 120 h of corrosion time. After corrosion for more than 120 h, the corrosion weight loss rate of parent metal increased rapidly, and that of two surface state welded joints were low and tended to be consistent. When the corrosion time reached 240 h, parent metal had the highest corrosion weight loss rate, reaching 111.84 g/m2.



Figure 1. Curves of corrosion weight loss and corrosion weight loss rate It can be clearly seen from Figure 1(b) that the corrosion rate of the two welded joints was

significantly higher than that of parent metal within 120 h at the beginning of the test. The corrosion rate of pre-welded polished joint was the highest, reaching 1.34 g/(m2 h), and the corrosion rate rised fastest in the initial 24 h. At this time, the active anions in the salt solution (such as  $Cl^{-}$ ,  $SO_{3}^{2-}$ ,  $HCO_{3}^{-}$ ,

 $NO_3^-$ , etc.) was the main corrosion driving force. Corrosion from 24 h to 240 h, corrosion rate through the process of "reduction - increase - decrease". Because of gradually accumulating corrosion production layer, the effect steric protection hindered the contact of the active anion in the corrosive medium with the aluminum alloy matrix, which caused the corrosion rate to decrease in a short period of time. After 72 h, the corrosion rate rised again. It was presumed that the surface corrosion pit expanded along the grain boundary, and the internal expansion of the corrosion production inside the corrosion pit causes the large corrosion production attached to the surface to split, creating a new channel for the corrosive medium to contact the metal matrix. After 120 h of corrosion, the weight loss rates of the two surface state welded joints tended to be the same, indicating that the coverage of the joint surface corrosion production was stable status. It effectively blocked the active anion from contacting the metal surface. After 240 h, the weight loss rate of parent metal was higher, reaching 0.466 g/(m<sup>2</sup> h). The change of corrosion rate showed that the pre-welded polished joint had poor corrosion resistance at the initial stage of the test. In the latter part of the test, the corrosion resistance of the two surface state welded joints was equivalent and better than parent metal.

In the CASS salt spray test,  $CuCl_2$  salt solution can induce corrosion quickly. Figure 2 shows the SEM results of BT-FSW joint under simulated acid rain corrosion environment. It can be seen that after 120 h of corrosion, parent metal surface was covered with reddish brown loose pits, caused by the low Cu potential in an acidic environment. The copper-rich S phase (A1<sub>2</sub>CuMg) and the  $\theta$  phase (CuA1<sub>2</sub>) were preferentially dissolved as anodes[6]. The red-brown corrosion production Cu<sub>2</sub>O corrosion production growed up and cracked due to internal stress, forming new corrosion channels to aggravate pitting corrosion. Compared with parent metal, the corrosion phenomenon of Nugget was mainly shallow exfoliation, there were micro-cracks in the pitting area. THAZ had the most complete surface morphology, shallow pitting pits, and less corrosion production attached. HAZ substrate had a large number of small diameter corrosion production, and its corrosion pit was flaky, with a tendency to develop internally[7].



(a)Parent metal  $150 \times$  (b)Nugget  $150 \times$  (c)THAZ  $150 \times$  (d)HAZ  $150 \times$ **Figure 2.** SEM images of welded joints after salt-spray corrosion



 (a) Parent metal (b) Pre-weld unpolished joints (c) Pre-welded polished connector Figure 3. EDS patterns of corrosion production

The EDS energy spectrum of corrosion production BT-FSW joint can be seen in Figure 3. Corrosion production of joints has the same composition, all of which consist of Al, C and O elements, which means that the main corrosion production of welded joints is  $Al_2(CO_3)_3$  and  $Al_2O_3$ . Because the T6 6005A aluminum alloy contains two strengthening phases of Al-Fe-Si and Mg<sub>2</sub>Si, and the corrosion production contains a small amount of Fe, Cl and Si element in addition to Cu<sub>2</sub>O. As the potential of the matrix is about -700 mV, the Mg<sub>2</sub>Si phase (-1200 mV) with lower corrosion potential is preferentially corroded as the anode end due to the action of the microcouple[8]. The corrosion

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potential of the Al-Fe-Si phase is higher (-200 mV) that the surrounding substrate will preferentially corrode to form pitting holes in the corrosion process, but it is not easy to corrode itself and trend to stay in the corrosion pit. Surrounding corrosion production is still in the state of "expansion-shedding". According to EDS spectrum analysis, after corrosion 120 h, the strengthening phase particles in the parent metal region are still retained in the corrosion pit to aggravate the corrosion, while the micro-galvanic effect is weakened with the shedding of welded joints second phase particles, which explains the reason why the macroscopic morphology of welded joints Nugget is less corrosive. At the same time, the pre-welded polished joint is similar to the composition of unpolished joints corrosion production, indicating that the corrosion resistance of the two groups is similar after the corrosion for 240h. It is consistent with the results of the weight loss rate of Figure 1.

#### 3.2. Electrochemical corrosion pitting potential

In addition to chemical corrosion, the metal surface corrodes electrolyte solution and a small amount of carbon in the metal will form micro-battery, causing electrochemical corrosion. The electrochemical tests were carried out in different areas of the welded joints in corrosion soaking time with 30 min and 336 h. The corresponding polarization curve is shown in Figure 4. The electrochemical parameters are shown in Table 3. The self-corrosion current  $I_{corr}$  and the corrosion breakdown potential  $E_{corr}$  of polarization curve can be reflecting corrosion resistance. The larger the  $I_{corr}$  in the same corrosion system, the faster the electrode reaction and the corrosion rate. The higher the potential of  $E_{corr}$ , the more difficult loss of electrons of surface oxide film, the less the corrosion tendency[9]. Therefore, the polarization curve at the top left of the image shows stronger corrosion resistance of the material.



(a) Corrosion 30min pre-welded unpolished joints (b) Corrosion 30min pre-welded polished joints



(c) Corrosion 336h pre-welded unpolished joints (d) Corrosion 336h pre-welded polished joints

Figure 4. Polarization curves of each zone of BT-FSW with different surface state

The polarization curve of the two kinds of joints has a large difference in Figure 4. When corrosion for 30 min, the polarization curve are located at the upper left of the whole image on both sides of the HAZ and weld nugget of unpolished joints, and the curve of parent metal is located at the lower right of the image. The electrochemical corrosion resistance of unpolished joints is higher than that in the parent metal region in the simulated acid rain environment. At this time, the corrosion potential of each zone of pre-welded polished is similar to that of parent metal, and polarization curve is located at the lower right corner of the image, indicating that the corrosion resistance of the polished joint is less than that of the unpolished joints. When the corrosion for 336 h, polarization curve in each area of unpolished joints has a small change compared with 30 min, while pre-welded polished joint polarization curve has a tendency to move to the left, which indicates that the corrosion resistance of

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the polished joint is weak when the corrosion time is short. However, as the corrosion time prolongs, the surface of the polished joints covered by the stable corrosion products layer. The thicker corrosion production are tightly bonded to the substrate, and the porosity of the film layer is low, so the corrosion resistance is improved to the extent similar to unpolished joints[10].

Combined with the data in Figure 4 and Table 3, it can be seen that the corrosion resistance of the BT-FSW joint is the best on both sides of HAZ, showing a lower self-corrosion density and corrosion breakdown potential. Followed by Nugget, its' self-current density and corrosion breakdown potential is similar to HAZ and exhibits a high corrosion sensitivity only when it is only corroded for 30 min. The parent metal is worst at the same corrosion time. When surface state is taken into consideration, the electrochemical parameters of unpolished joints are more stable than that of pre-welded, presumably because the pre-weld unpolished process retains the dense Al<sub>2</sub>O<sub>3</sub> film on the surface. As an solid-state welding method, the Al<sub>2</sub>O<sub>3</sub> film on the shoulder contact surface is broken into oxide particles under intense friction heat input, which participates in the plastic flow process of the joint material and finally remains in the joint structure. The oxide particles still have a certain protective effect, which improves the corrosion breakdown potential of the oxide film on the surface of the joint. In addition to the parent metal of well-preserved Al<sub>2</sub>O<sub>3</sub> film, the corrosion potential of the unpolished joints should be higher than the pre-welded polished joint of the corresponding area, which is consistent with the distribution of the two surface state welded joints polarization curve when corroded for 30 min. When the corrosion exceeding 336 h, the thickness of corrosion production on the surface of the test piece increases, which reduces the adsorption of Cl<sup>-</sup> SO<sub>3</sub><sup>-</sup> and HCO<sub>3</sub><sup>-</sup> on the surface of the body, so the corrosion potential of the pre-welded polished state joint is increased to the same level as the pre-weld unpolished joints, and the corrosion resistance is equivalent.

		I I		5		
Test sample	Corrosion	Current density/	Parent	Heat affected	Heat affected	Nugget
number time		potential metal advancing side ret		retreating side	Nugget	
Pre-weld unpolished joints	20 min	$I_{corr}/\mu A \cdot cm^{-2}$	529.16	15.11	9.54	9.01
	50 1111	E <sub>corr</sub> /mV	-1561.35	-1311.75	-1206.07	-1162.43
	336 h	$I_{corr}/\mu A \cdot cm^{-2}$	46.74	13.47	20.71	13.50
		E <sub>corr</sub> /mV	-1288.89	-1053.97	-1060.22	-1124.05
D	20 min	$I_{corr}/\mu A \cdot cm^{-2}$	531.33	33.62	111.17	492.31
polished joints	50 1111	E <sub>corr</sub> /mV	-1565.51	-1491.29	-1525.36	-1556.76
	336 h	$I_{corr}/\mu A \cdot cm^{-2}$	29.75	16.46	14.93	16.07
		E <sub>corr</sub> /mV	-1227.92	-1115.61	-1131.94	-1097.41

$-$ <b>Land</b> $3_{4}$ is the state of the stat	<b>Table 3.</b> Electrochemical	parameters of <b>BT-FSW</b>	ioints with different surface state
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#### 3.3. Electrochemical AC impedance analysis

The AC Impedance Test (EIS) is a test method for the corresponding corrosion mechanism by measuring the response signal generated by the small peak AC signal perturbation electrochemical system. Figure 5 shows the Nquist map and Bode map in open circuit potential (-0.78 V) under the simulated acid rain environment of welded joints. It can be seen from Figure 5(a) that the fitting curve and the test curve have higher coincidence in the frequency test range. The Nquist spectrum has consistent characteristics, showing two incomplete semicircular capacitive arcs. The high-frequency capacitive anti-arc on the left side represents the nature of the self-passivation film's own impedance, and the low-frequency capacitive anti-arc on the right side represents the nature of the impedance of the workpiece electrode after the passivation film is dissolved when the corrosion diffusion channel is completely opened[11]. The impedance of the passivation film is related to the arc. The larger the radius of the arc, the greater the reaction resistance, and the less the charge is transferred[12]. Judging from the radius of the arc, the low-frequency capacitive reactance of parent metal is much larger than that of pre-welded polished. The value ( $|Z|_{0.01Hz}$ ) of parent metal in low frequency impedance mode is between  $10^5 \sim 10^6 \Omega \cdot cm^2$ , which is higher than that of the two welded joints. It is

presumed that a new film layer formed on the surface of Nugget after the BT-FSW is completed. The new film layer is thinner than the parent metal passivation film, and the corrosion breakdown potential of this film is lower than that of base metal passivation film. The thicker parent metal passivation film acts as a shielding layer at the initial stage of corrosion, which well isolates the contact between the corrosion resistance of pre-welded polished joint was the worst at the beginning of corrosion, which is consistent with the results of salt spray test and Tafel curves.

The frequency-impedance of parent metal and joints is approximately a straight line with slope -1 in Figure 5(b), indicating that the resistance and capacitance are in parallel. Figure 5(c) shows that the Bode spectrum has two peaks, standing two time constants. So the EIS spectrum is fitted by the equivalent circuit diagram shown in Figure 6, and the fitting parameters of circuit component are obtained as shown in Table 4.  $R_s$  represents the resistance of the electrolyte solution between the test piece and the calomel electrode;  $R_f$  represents the impedance of the passivation film;  $R_{ct}$  represents the charge transfer resistance between the contact interface between the passivation film and the electrolyte;  $Q_c$  is common phase angle component; C represents the interface capacitance between the passivation film and the electrolyte interface[13]; n is the degree of similarity with the phase angle component  $Q_c$  and the ideal capacitance; *CHI* is the total variance of the fitted curve.

It can be seen from Table 4 that the equivalent circuit diagram fitting curve error does not exceed  $7 \times 10^{-3}$  and reflect the mechanism of the corrosion process. Since the EIS impedance spectrum is measured in the acid solution of the same corrosive medium, the value of electrolyte solution resistance  $R_s$  is less different. The  $R_f$  value and  $R_{ct}$  value of parent metal are significantly higher than the two surface state welded joints. The impedance of the parent metal passivation film is large when the corrosion reaction occurs, and so as the charge transfer resistance of the electrolyte and the workpiece surface, indicating the corrosion resistance of parent metal is the best. The corrosion resistance of the specimen: parent metal>unpolished joints>polishing joint is from strong to weak when the corrosion medium is exposed for a short time. The EIS fitting results show that the polishing process will reduce the corrosion resistance of the joints. But when the corrosion time is prolonged, the surface of the joints will form a protective layer of stable corrosion production, which hinders the contact channel of corrosive ions, The corrosion resistance of the joints is not obviously reduced by grinding process.







Figure 6. Equivalent circuit diagram of impedance spectrum of welded Joint Table 4. EIS fitting data of welded joints in different surface state

Test sample	$R_s$ ( $\Omega \cdot \mathrm{cm}^2$ )	$Q_c$ ( $\Omega^{-}$ cm <sup>-2</sup> s <sup>n</sup> )	п	$R_f$ ( $\Omega \cdot \mathrm{cm}^2$ )	C (µF)	$R_{ct}$ ( $\Omega \cdot \mathrm{cm}^2$ )	CHI
Unpolished joints	5.609	1.513×10 <sup>-5</sup>	0.894	$2.178 \times 10^4$	122.4	$1.02 \times 10^{5}$	6.59×10 <sup>-3</sup>
Polished joints	5.508	1.216×10 <sup>-5</sup>	0.911	$2.906 \times 10^4$	109.2	$0.87 \times 10^{5}$	6.19×10 <sup>-3</sup>
Parent metal	6.951	$0.655 \times 10^{-5}$	0.907	$1.851 \times 10^5$	22.59	$3.26 \times 10^{5}$	2.16×10 <sup>-3</sup>

#### 4. Conclusion

(1) The rate change of the salt spray test accords with the law of "lower-rise-lower". Before 120 h, the weight loss rate of parent metal is the minimum, the rate of pre-welded polished joints is the highest and the most corroded. After 240 h, the corrosion rate of parent metal rises rapidly. At this time, the weight loss rate of the two welded joints is similar, and better than that of parent metal.

- (2) The surface of salt spray test joint has shallow pitting corrosion, mainly shallow exfoliation. The active anion in acidic medium and the high-potential second phase particles not falling off in the corrosion pit are the main corrosion driving forces of pitting corrosion.
- (3) Comparing the different dynamic potential polarization curves: short-term corrosion for 30 min, the self-corrosion current of unpolished joints is lower, the corrosion breakdown potential of the passivation film is higher, the corrosion resistance is much higher than that of parent metal. The pre-welded polished joints show poor corrosion resistance. After corroded for 336 h, the pre-welded polished has limited influence on electrochemical corrosion resistance
- (4) The electrochemical characteristics of the EIS spectra of the two kinds of joints are basically the same, and the Nquist spectra reflect the corrosion behavior of the passivation film on the surface of the two joints is also consistent. At short time corrosion, the oxide film's resistance and charge transfer resistance on the surface of parent metal are much higher than the new film formed on the surface of the two welded joints. Meanwhile, the pre-welded polished process reduces the corrosion resistance of the joint to some extent. As the corrosion time prolongs, the pre-weld surface state has little effect on the corrosion resistance of the joint. Therefore, the process of production can be considered to cancel the pre-welded polished process to reduce the production cost.

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