Micro-image analysis in the diffusion-bonded zone of Fe₃Al/Q235 carbon steel dissimilar materials

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Abstract. The chemical composition of the second phase precipitation in the vacuum diffusion-bonded zone of Fe₃Al intermetallic compound and Q235 carbon steel was analysed by means of electron probe micro-analyser (EPMA). The relative content of the second phase precipitation and grain size was evaluated through a micro-image analyser. The percentage of Fe and Al content in the diffusion zone was measured by EPMA. The results indicated that the relative content of the second phase precipitation rich in carbon and chromium at the Fe₃Al/Q235 interface was much higher. With the transition from Fe₃Al intermetallic compound to Q235 carbon steel across Fe₃Al/Q235 interface, the grain diameter decreased from 250 μ m to 112 μ m, Al atom content decreased from 27% to 15%, while Fe atom content increased from 76% to 96%.

Keywords. Fe₃Al/Q235 diffusion-bonded zone; vacuum diffusion bonding.

1. Introduction

Fe₃Al is a newly developed intermetallic, which has unique electronic and magnetic properties as well as excellent resistance to corrosion and oxidization by the addition of alloying elements such as Cr, B, Ce and Zr (Mckameey *et al* 1991; Fair and Wood 1994). But the additional alloying elements can easily cause the second phase precipitation in Fe₃Al intermetallic compound. The coarse grain formed in Fe₃Al during heating can affect its property (Yin *et al* 1996; Kim and Cantor 1994). Especially the microstructure in the weld zone of Fe₃Al intermetallic compound changed considerably when it was welded with dissimilar materials. Traditionally, optical microscopy is used to make a qualitative assessment of the microstructure.

The relative content of second phase precipitation in the Fe₃Al/Q235 diffusion-bonded zone was evaluated through XQF-2000 image analyser and the grain diameter was computed. Chemical composition of the second phase precipitates and Fe, Al content in the diffusion zone from Fe₃Al intermetallic compound to Q235 carbon steel across Fe₃Al/Q235 interface were analysed by means of EPMA. These help to evaluate correctly the relation between microstructure and performance in Fe₃Al/Q235 diffusion-bonded zone, to optimize weld technology and to promote further the property and quality of the diffusion-bonded zone.

2. Experimental

Materials used were Fe₃Al intermetallic compound and Q235 carbon steel. Fe₃Al was as cast plate and its chemical and thermo-physical characteristics are listed in table 1. The surfaces of Fe₃Al and Q235 base metals were polished by sand paper, then they were cleaned with alcohol propanone, washed and dried. The test plates were superposed and inserted in the vacuum chamber of the diffusion bonding equipment. In diffusion bonding, materials were heated by resistance heating under a vacuum of 5×10^{-6} Torr, and at a pressure of 9.8 MPa. The peak temperature was 1080°C with a holding time of 60 min.

A series of specimens were cut from the location of the diffusion-bonded zone. They were then etched with HCl and $HNO_3 + CH_3COOH$ solution. Fe₃Al was etched with a solution containing 36.5% HCl while Q235 with a solution containing 3% HNO₃ and 97% CH₃COOH because the resistance to corrosion between Fe₃Al intermetallic compound and Q235 carbon steel differ considerably.

The second phase precipitation feature in the Fe₃Al/Q235 diffusion-bonded zone was observed by means of JXA-840 SEM. The percentage of the second phase precipitation was measured by electron probe micro-analyser (EPMA) and the relative content of the second phase precipitation and grain diameter in the Fe₃Al/Q235 diffusion-bonded zone were evaluated by means of XQF-2000 computer micro-image analyser.

The degree of grain size (G) is defined by GB6394-86 (Chinese national standard) and it is measured by the cutting-line method (calculating the number of cuttings

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on the grid for a given length). The formula for the degree of grain size is

$$G = -3.2877 + 6.6439 \log (m \times N/L),$$

where L is the length (mm) of the grid lattice, m the magnification and N the number of cuttings on the gauge line (Cui 1988).

3. Results and discussion

3.1 Evaluation of second phase precipitation

The microstructure of the Fe₃Al/Q235 carbon steel diffusion-bonded zone is shown in figure 1(a). It consists of three regions, the region near to Fe₃Al, the Fe₃Al/Q235 interface and the region near to Q235 carbon steel. The width of the whole diffusion zone was about 0.5 mm. On the Fe₃Al side, columnar grain was coarse. At the interface, the grain was isometric and fine. On the Q235 carbon steel, the microstructure composed of blocks of ferrite and pearlite. There were Fe₃Al, FeAl and **a**-Fe (Al) solid solution phases in Fe₃Al/Q235 diffusionbonded zone as obtained from EPMA and X-ray analysis (Wang *et al* 2001). Second phase precipitates were distributed discontinuously in the Fe₃Al/Q235 diffusion-bonded zone (figure 1(b)). Its relative content is given in table 2. From table 2, it can be seen that the number of second phase precipitates at the Fe₃Al/Q235 interface was more than that in any other region. Both Fe and Al contents in second phase precipitates were less than those in Fe₃Al base metal, but the contents of C and Cr were more than those in Fe₃Al base metal (see table 3). It was possible to form carbon and chromium compounds.

The cooling rate was very high and solute cannot diffuse rapidly during welding, which cause C and Cr element to segregate partially in the grain after crystallization. In addition, the second phase precipitation on the grain boundary was caused by the difference in lattice distortion energy between grain and grain boundary. The greater the difference in radius between the solute and the solvent, the greater is the distortion energy and more is the solubility of the solute atom on the grain boundary. The chromium atom is much bigger than the ferrite atom and the lattice distortion energy in the grain is much more than that on the grain boundary. So Cr atom segregates to the grain boundary, which is a stable state. The less the solid solubility, the more the tendency to segregate to the grain boundary.

Table 1. Chemical compositions and thermo-physical data of Fe₃Al intermetallic compound.

| Chemical composition (wt.%) | | | | | | | | |
|------------------------------|---------------------------------|--------------------------|-----------------------|--|------------------------------------|--------------------------------|-----------------|----------------|
| Fe | | Al | Cr | Nb | Zr | В | С | e |
| 76.5 | | 16.2 | 0.38 | 1.93 | 0.28 | 0.01 | 0 ⋅1 | 15 |
| Thermo-physical performances | | | | | | | | |
| Crystal structure | T _c ordering (°C) | Young's modulus (GPa) | Melting point (°C) | Coefficient of therm expansion (10 ⁻⁶ ·K ⁻ | al Density ($g \cdot m^{-3}$) | s _b (MPa) | d (%) | R _c |
| DO ₃ | 540 | 140 | 1540 | 11.5 | 6.72 | 455 | 2 | 29 |



Figure 1. Microstructure feature in $Fe_3Al/Q235$ diffusion-bonded zone: (a) Microstructure feature in diffusion-bonded zone and (b) second phase precipitation feature.

3.2 Grain characteristics in diffusion-bonded zone

The grain of Fe_3Al intermetallics was coarse because grain grows rapidly during heating. Grains combine and grow and the grain direction will change because of the

Table 2. The second phase precipitation content in $Fe_3Al/Q235$ diffusion-bonded zone.

| Position | Fe ₃ Al base | Near to Fe ₃ Al | Fe ₃ Al/Q235 interface | Near to Q235 |
|-------------|-------------------------|-------------------------------|--------------------------------------|-----------------|
| Content (%) | 13.5 | 12.3 | 25.3 | 6 |

Table 3. EPMA analysis in $Fe_3Al/Q235$ diffusion-bonded zone (%).

| Position | Fe | Al | С | Cr | Mn | Si |
|--------------------------------|----------------------------------|----------------------------------|--------------------------------|------------------------------|------------------------------|------------------------------|
| Base metal | 82.6 82.7 81.9 82.0 | 16·6 16·3 17·2 16·9 | 0.14 0.13 0.13 0.13 | 1.02 0.99 1.01 0.94 | 0.15 0.15 0.13 0.18 | 0·18 0·22 0·20 0·20 |
| Second phase precipitate | 74·66 77·90 77·04 78·77 | 14·31 15·90 15·45 13·10 | $0.65 \\ 0.61 \\ 0.50 \\ 0.22$ | 1.18 1.18 1.32 1.26 | 0·21 0·23 0·23 0·20 | 0.07 0.10 0.10 0.06 |

Table 4. The degree of grain size, the grain diameter and the hardness in $Fe_3Al/Q235$ diffusion-bonded zone.

| Position | Fe ₃ Al base | Fe ₃ Al/Q235 | Nearby |
|--|-------------------------------|------------------------------------|-----------------------------|
| | metal | interface | Q235 |
| Degree of grain size | 1.02 | 2·05 | 3·30 |
| Grain diameter (µm) | 250 | 173 | 112 |
| Macro-hardness Micro-hardness (VPH) | R _C 20·5 412 | <i>R</i> _в 87 331 | R _в 66 159 |



Figure 2. The concentration of Fe and Al across the interface from Fe_3Al to Q235.

diffusion of elements in Fe₃Al and Q235 carbon steel. In order to study the effect of grain feature in diffusionbonded zone on the property, the degree of grain size was evaluated by means of XQF-2000 and grain diameter was computed according to formula

$$d^2 = 1/2^{G+3}$$

where d is the grain diameter, G the degree of grain size.

At the same time, the hardness of Fe_3Al and Q235 base metal and $Fe_3Al/Q235$ interface was measured and the result is shown in table 4.

From table 4, it can be found that the grains in the Fe₃Al/Q235 diffusion-bonded zone are much finer than that in Fe₃Al intermetallic compound because the heating rate is slow and atom diffuses mainly during diffusion bonding. The grain diameter decreases from 250 μ m to 112 μ m. The macro-hardness and micro-hardness decrease. This is explained by the diffusion of Fe and Al atoms. The concentration of Fe and Al across the interface from Fe₃Al to Q235 is shown in figure 2.

The Fe, Al atom content curve (figure 2) shows that with the transition from Fe₃Al to Q235 carbon steel across Fe₃Al/Q235 interface, Al atom content decreases from 27% to 1% and Fe atom content increases from 13% to 96%. There is no obvious brittle phase according to equilibrium phase diagram, which is favourable to improve the synthetical property in diffusion-bonded zone (Li Yajiang *et al* 2001). Thus the property of Fe₃Al/ Q235 diffusion-bonded zone can meet the requirement for use.

4. Conclusions

(I) There is a second phase precipitation in the Fe₃Al/Q235 diffusion-bonded zone. The Al, Fe atom content in second phase is less, while C and Cr atom content is much more than that in Fe₃Al base metal. The relative content of second phase precipitation is more at the Fe₃Al/Q235 interface because of the large difference in distortion energy between both sides of the Fe₃Al/Q235 interface.

(II) From Fe₃Al to Q235 carbon steel across Fe₃Al/Q235 interface, the grain diameter decreases from 250 μ m to 112 μ m. Fe and Al atom diffuse across the Fe₃Al/Q235 interface. Al atom content decreases from 27% to 1%, while Fe atom content increases from 13% to 96%. This causes a decrease in micro-hardness of diffusion-bonded zone.

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