Improved mechanical properties in dissimilar Ti-AISI 304L joints

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The effect of low temperature post weld heat treatment on the tensile strength and bend test properties of dissimilar friction welds between titanium and AIS1304L stainless steel joints is investigated. Post weld heat treatment at temperatures less than 873 K has no effect on joint tensile strength properties, but markedly improves bend test properties. The highest bend angle is produced using a post-weld heat treatment at 773 K for 1 h (the Larson-Miller parameter corresponding to this treatment is 15.5×10^3 Kh⁻¹). Low temperature heat treatment improves bend ductility, because stress relaxation occurs with minimal increase in the transition region width at the bondline region. Dissimilar joint bend testing properties decrease markedly when the width of the transition region exceeds $1-2 \mu m$. An explanation for the detrimental effect of thick transition regions at the joint interface region on the mechanical properties of dissimilar joints is proposed. It is suggested that the development of significant triaxial stress due to the constraint imposed by large, needle-shaped intermetallic particles promotes premature joint failure in joints containing thick transition regions.

1. Introduction

In prior research, one of the present authors investigated the influence of joining parameters on the mechanical properties of friction welds between titanium and AISI 304L stainless steel [1]. The highest attainable tensile strength was 460 MPa and failure occurred in the titanium substrate during tensile testing. However, the bend test properties of joints between AISI 304L and titanium substrates were very poor. The influence of post-weld heat treatment (PWHT) on the mechanical and metallurgical properties of dissimilar joints was investigated. A low temperature post-weld heat treatment markedly improved bend ductility [2]. However, a more detailed investigation concerning the effects of low temperature post-weld heat treatment on joint mechanical properties was required. The present paper examines the influence of post-weld heat treatment at temperatures ranging from 623 to 1173 K for times ranging from 10 s to 2 h on the mechanical properties of dissimilar AISI 304L-titanium friction welds.

2. Experimental procedure

The chemical compositions of the 16 mm diameter commercially pure titanium and solution treated AISI

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304L stainless steel bars are shown in Table I. A brake type friction welding device was employed throughout, and joining was accomplished using the following parameter settings: frictional pressure, 200 MPa; friction period, 3 s; upsetting pressure, 300 MPa; upsetting period, 10 s; and rotational speed, 1560 r.p.m. In all cases, the contacting surfaces were polished and degreased using acetone prior to joining.

Post-weld heat treatment was carried out in a vacuum furnace at 623-1173K for holding periods ranging from 10 s to 2 h. The heating rate to the PWHT temperature was $2 K s^{-1}$.

The tensile test specimen dimensions were 10 mm diameter $\times 60$ mm long (Fig. 1). The bend test specimens were also 10 mm diameter and during threepoint bending, the radius of bending was 20 mm (twice the sample diameter). The maximum bend angle attained during joint bend testing was taken as the indicator of bend testing properties. All mechanical testing was carried out at room temperature.

Joint interface regions were examined using a combination of optical, scanning electron and transmission electron microscopy. Scanning electron microscopy was carried out using an Hitachi S-2150 device equipped with a Horiba EMAX-2770 energy

Figure 1 Test specimen dimensions: (a) joint tensile test specimen, and (b) joint bend test specimen.

dispersive X-ray analyser (EDX) attachment. Transmission electron microscopy (TEM) was carried out using a 200 kV Joel JEM-2010 microscope containing a Noran UTW-EDX chemical analysis attachment (Series 11, ultrathin window type). During chemical analysis, the mean spot diameter was 2 nm.

Prior to optical microscopy, the specimens were etched in a mixture of perchloric acid and glacial acetic acid. The weld zone microstructure was examined at the joint centreline region and $2 \mu m$ on either side of the fracture surfaces of broken test specimens. The TEM specimens were removed transverse to the joint axis at a point midway between the centreline and the periphery of the friction welded component. All TEM preparation procedures comprised cutting 1 mm thick test specimens, wet polishing them to 0.2 mm thickness, followed by dimpling and final thinning using a dual ion milling device and argon gas.

3. Results

3.1. Joint tensile properties

Fig. 2 shows the effect of the PWHT temperature on the tensile strength of dissimilar friction welds (for holding times ranging from 10 s to 1 h). When the holding time was very short (10 s), post-weld heat treatment at temperatures up to 873 K had a negligible effect on joint tensile strength properties and all test samples failed in the titanium substrate. However, post-weld heat treatment at temperatures exceeding 973 K for 10 s decreased joint tensile strength and promoted failure at the joint interface.

Figure 2 Effect of heating time and PWHT temperature on joint tensile strength. Fracture position: $(\Box, \Diamond, \triangle)$ titanium substrate, $(•, A)$ interface. AW, as-welded.

Post-weld heat treatment for 1 h at temperatures ranging from 773 to 1073 K significantly decreased joint tensile strength (below that of as-welded joints). Also, post-weld heat treatment at 973 and 1073 K shifted the locus of failure during tensile testing from the titanium substrate to the joint interface region. It will be shown later that the decrease in tensile strength is associated with recrystallization, with the removal of the work hardened region immediately adjacent to the bondline in the titanium substrate, and with increase in the width of the transition region at the joint interface.

3.2. Bend testing

Fig. 3 shows the effect of post-weld heat treatment on the bend testing properties of dissimilar AISI 304L-titanium joints. Using a 10 s holding time, the bend angle increased gradually with increasing PWHT temperature and peaked at 873 K. The bend angle exceeded 30° in joints post-heat treated at 873 K and was three times higher than in the as-welded joints.

Post-weld heat treatment at 773 K for 1 h produced bend angles exceeding 50° . For example, one joint sample post-weld heat treatment at 773 K for 1 h had a bend angle of 180°. However, post-weld heat treatment for 1 h at temperatures above 773 K markedly

Figure 3 Effect of heating time and PWHT temperature on joint bend test properties: (\circ) 10 s, (\triangle) 3.6 ks. AW, as-welded.

Figure 4 Effect of heating time at (\circ) 773, (\triangle) 873 and (\Box) 973 K on joint bend test properties. AW, as-welded.

decreased the bend angle values (see Fig. 3). Fig. 4 indicates the effect of holding time on the bend angles produced during post-weld heat treatment at 773, 873 and 973 K, respectively. Post-weld heat treatments at 773 K for 1 h and 873 K for 30 min produced the highest bend angles. It is apparent from Fig. 4 that post-weld heat treatment at 973 K had a detrimental effect on bend test properties (for all holding times). The optimum post-weld heat treatment conditions (773 K for 1 h and 30 min at 873 K) can be compared using the well known Larson-Miller parameter, $P = T$ [20 + log t], where T is the temperature in degrees Kelvin and t is the holding time (h). The highest bend angles occurred when the Larson-Miller parameter ranged from 15.5×10^3 to 17.5×10^3 $(Kh^{-1}).$

Figure 5 The residual stress distribution in the .radial direction, 0.125 mm from the bondline region in (\bullet) AISI 304L-(\circ) titanium friction welds (after Kim *et aI.* [3] ; The joint periphery occurs at $r = 6.5$ mm).

It is clear from Figs. 2 and 4 that post-weld heat treatment for 10 s at temperatures up to 873 K had no effect on joint tensile strength properties, but had a beneficial effect on bend test results. The different outputs produced during tensile and bend testing depend on the manner in which the joint interface region is strained during mechanical testing. Tensile testing is not particularly effective when the mechanical properties of dissimilar joints is evaluated. During tensile testing, joint interface failure will be determined by the mechanical properties of the dissimilar substrates and by the mechanical properties of material at the bondline. Also, rigid restraint will produce triaxial stress at the bondline region, i.e. the apparent strength at the joint interface will be increased because of the difference in mechanical properties of the interface material and the adjacent metal substrates. In contrast, bend testing is more effective when assessing joint interface properties and the output results markedly depend on the residual stress produced by the friction welding operation. Kim et *al.* [3] have modelled residual stress generation during AISI 304L-titanium bonding and indicated that localized tensile residual stress is produced at the joint periphery in the titanium substrate (see Fig. 5). The presence of tensile residual stress in titanium material at the periphery of the component will have an extremely detrimental effect on the bend test properties of as-welded dissimilar joints. It follows that one of the beneficial effects of low temperature post-weld heat treatment is that it relaxes the tensile residual stress produced by the friction joining operation.

3.3. Metallurgical examination of the joint interface region

Fig. 6 shows optical micrographs of the joint interface region in dissimilar AISI 304L-titanium friction welds. The as-welded joint contained a $100 \mu m$ wide unetched region immediately adjacent to the bondline region in the titanium substrate (see Fig. 6a). This region was formed as a result of plastic deformation

Figure 6 Optical microstructures of AISI 304L-titanium friction welded joints.

during the joining operation [2]. Post-weld heat treatment allowed recrystallization of the AISI 304L and titanium substrates (see Fig. 6b-e) and higher PWHT temperatures increased the width of the intermediate layer (transition region) formed at the joint interface.

Fig. 7 shows the hardness distribution at the bondline in as-welded and heat treated dissimilar joints. The hardness of the titanium substrate immediately adjacent to the joint interface was higher than in the bulk material. Post-weld heat treatment markedly

decreased the hardness of this region (its hardness decreased from H_v 250 to 170). The relation between the Larson-Miller parameter and hardness in the titanium and AISI 304L substrates at a distance 50 μ m from the joint interface is shown in Fig. 8. The hardness of the titanium substrate decreases markedly when the Larson-Miller parameter exceeds $13 \times$ 10^3 K h⁻¹ (this corresponds with post-weld heat treatments of 653 K for 1 h or 703 K for 6 min, for example). The joints hardness decreased during post-weld

Figure 8 Relation between joint hardness and the Larson-Miller parameter: H_v titanium: 160-180; H_v aluminium: 200-220.

heat treatment due to recrystallization and removal of the locally hardened region immediately adjacent to the joint interface in the titanium substrate.

Figs. 9 and 10 show SEM and TEM micrographs of the as-welded joint interface region and the iron, chromium, nickel and titanium concentration changes at the joint interface. Although a number of individual

Figure 7 Hardness of (a) titanium-(b) AISI 304L friction welds. *Figure 9* SEM micrographs and Fe, Cr, Ni and Ti concentration changes in an as-welded AISI 304L-titanium joint.

crystals were observed at the joint interface (in Fig. 10), electron diffraction analysis could not be performed since the grains were too small. Consequently, the phases present at the joint interface were deduced using a combination of the chemical analysis results and the Fe-Cr-Ti ternary equilibrium phase diagram. Using this approach, regions A and B comprise stainless steel containing χ phase, region C contains λ and χ phases, region D contains FeTi, λ and β Ti phases, and region E contains β Ti and FeTi phases. Fig. 10 confirms that the total width of regions B, C and D is \sim 300 nm.

Figs. 11 and 12 show SEM and TEM micrographs of a test sample post-weld heat treated at 773 K for i h. Regions A and F in Fig. 12 are the stainless steel and titanium substrates, respectively. Region B comprises stainless steel and χ phase, region C contains λ and FeTi phases, and regions D and E contain β Ti, aTi and FeTi phases. The total width of regions B and C is \sim 450 nm.

It is apparent from Figs. 10 and 12 that the titanium and iron concentrations are non-uniformly distributed at the joint interface (about the point where the iron and titanium concentrations are 50 at %). A similar feature has been observed in joints between pure titanium pure iron diffusion couples and, although the diffusion rates of iron and titanium are uncertain during post-weld heat treatment at 773 K, it is suggested that the transition region grows from the titanium substrate into the stainless steel.

Figure 10 (a) TEM micrograph showing the bondline region in an as-welded AISI 304L-titanium joint: and (b) the concentration changes across the region. (\bullet) Ti, (\circ) Fe, (\bullet) Ni, (\triangle) Cr.

4. Discussion

The transition region is formed at the joint interface between the dissimilar substrates. In the present study, the transition region in AISI 304L stainless steel-titanium joints contains a mixture of intermetallic phases χ , λ and FeTi and regions of interdiffusion (solid solution). It is well known that the mechanical properties of dissimilar joints markedly depend on the dimensions of the transition region and that when some critical thickness is exceeded, the joint mechanical properties are very poor. In the present study, optical microscopy confirmed that the width of the transition layer was $5-6 \mu m$ in joints post-weld heat treated at 773 K for 1 h. However, EDX analysis during SEM and TEM microscopy on the same test sample confirmed that the transition layer width was $2 \mu m$ or less. It is apparent, therefore, that the method of estimating the transition layer thickness must be carefully considered when any measured value is compared with results published in the literature.

Figure 11 SEM micrographs an Fe, Cr, Ni and Ti concentration changes in an AISI 304L-titanium joint post-weld heat treated at 773 K for 1 h.

It has been suggested that intermetallic formation in the transition region of dissimilar friction and pressure welds has no effect on joint integrity, when the thickness of the layer is $\langle 1 \mu m \vert 4 \vert$. In the present study, TEM microscopy confirmed that the thickness of the transition region in the as-welded AISI 304L-titanium joint was much less than $1 \mu m$ (see Figs 9 and 10). It follows that intermetallic phase formation cannot account for the poor bend test properties of as-welded joints. This suggests that the poor as-welded bend test properties of dissimilar joints result from a combination of strain hardening and tensile residual stress generated during the friction welding operation.

The optimum bend test properties occurred in joints post-weld heat treated at 773 K for 1 h (see Fig. 3), since low temperature post-weld heat treatment allowed recrystallization and relief of the tensile residual stress generated during the friction welding operation. This readily explains the effectiveness of the Larson-Miller parameter in determining the optimum post-weld heat treatment condition (this corresponds with a parameter value of 15.5×10^3 K h⁻¹).

The transition region width was $1-2 \mu m$ in joints post-weld heat treated at 773 K for 1 h (see Fig. 12). Also, longer holding times at 773 K or post-weld heat treatment at much higher temperatures will markedly decrease bend test results and increase the thickness of the transition region (see Fig. 4 and $\lceil 2 \rceil$). The much poorer bend test properties produced using higher post-weld heat treatment temperatures can be readily

Figure 12 (a) TEM micrograph showing the bondline region in an AISI 304L-titanium joint post-weld heat treated at 773 K for 1 h: and (b) the concentration of changes across the region. (\bullet) Ti, (\circ) Fe, (\blacktriangle) Ni, (\triangle) Cr, \ominus (- - -) lamellar black region.

understood if the critical width of the transition region in AISI 304L-titanium dissimilar friction welded joints is taken as $1-2 \mu m$.

Kharchenko [5] explained the beneficial effect of thin transition regions by suggesting that such regions deform easily and have less effect on the deformation characteristics of the softer, adjacent substrate materials. However, Kharchenko $[5]$ did not explain why joints become brittle when the transition region exceeds some critical dimension. With this in mind, an explanation for the detrimental influence of intermetallic phases and of transition layer dimensions on dissimilar joint mechanical properties will be presented.

4.1. Thin transition layers

Interdiffusion is restricted when thin transition regions are formed during dissimilar welding. In thin transition regions, it is suggested that the influence of intermetallic particles on bondline mechanical properties is similar to that during precipitation hardening, i.e. the transition region acts as a ductile material reinforced with rigid particles. If the particle geometry permits unrestricted plastic flow, the material at the bondline will be strengthened, but plastic deformation can occur without crack formation. When the transition region width increases in size and the amount of intermetallic formation increases and the particles grow in size, the resultant increase of material strength at the bondline may have a beneficial effect on the joints mechanical properties. There is experimental evidence to support this contention. For example, it has already been shown that increasing the transition layer thickness from 0.2 to 1 μ m markedly improved the joint shear strength of A1-Steel friction welds [4].

4.2. Thick transition layers

Small elongated crystals are formed at the joint interface region (see Figs 10 and 12) and the transition zone comprises a mix of intermetallic particles (χ, λ) and FeTi) and interdiffusion (solid solution) regions. When the width of the transition region increases, the amount and the size of intermetallic particles formed at the joint interface both increase. Thick transition regions can therefore be considered as composite materials. The influence of second phase particles on the deformation characteristics of a composite material containing short fibres (0.5 μ m thick \times 50 μ m long SiC whiskers in an aluminium alloy matrix) has been recently examined by Christman *et al.* [6]. Large tensile triaxial stresses are generated at the ends of short fibres and compressive stresses are generated in matrix material immediately adjacent to the fibre peripheries. In addition, when two high aspect ratio particles are close to each other, the region between them is subjected to triaxial stress and increasing the applied stress during mechanical testing will lead to void or crack formation [6, 7]. It is apparent from Figs 10 and 12 that elongated intermetallic-rich regions are formed at the joint interface region during the friction welding operation and during subsequent post-weld heat treatment. The formation of high aspect ratio, intermetallic particles in the transition region has an important effect on the joints mechanical properties. For example, when the matrix contains spherical particles, the absence of stress concentrations markedly reduces the matrix plastic strains in the vicinity of the reinforcing particles [6]. With this in mind, it is suggested that the detrimental effect of intermetallic particles in the thick transition regions depends on the generation of triaxial stress due to the constraint imposed by the reinforcing (intermetallic) material. Triaxial stress will be promoted by the formation of needle-shaped intermetallic particles and by clustering of the intermetallic particles. When a dissimilar joint containing a thick transition region at the joint interface is loaded, void formation and cracking will occur at the bondline and the bend test (and sometimes, the tensile strength) properties will be significantly impaired.

The critical transition region thickness of $1-2 \mu m$ in AISI 304L-titanium friction welded joints is similar to that found in dissimilar niobium-Armco iron joints [8] and during pressure welding of dissimilar materials [9]. However, it is worth noting that the critical transition region thickness in other dissimilar joining situations can exceed $1-2 \mu m$. For example, the critical transition region width in a pure aluminium-pure titanium joint is around 10 μ m [10]. Consequently, the factors that determine transition region width and intermetallic formation during friction welding are extremely important. For example, it has been suggested that some balance between the rotational velocity and the axial deformation during friction joining will minimize intermetallic phase formation during dissimilar welding [9]. Also, it has been suggested that maintaining the temperature of the contact zone below 633–723 K during aluminium–steel friction welding (by increasing the frictional pressure) will avoid intermetallic formation [11].

The mechanical and thermophysical properties of the dissimilar substrates will also have a major influence on the dimensions of the transition region and on intermetallic formation. For example, during friction welding, Kim *et al.* [3] confirmed that the temperatures attained by each substrate markedly depend on the thermophysical properties of the two substrates and on the joining parameters selected. For example, during A1SI 304L-titanium and aluminium-titanium friction welding, the temperatures attained by the stainless steel and aluminium substrates (at a distance of 3.25 mm from the bondline) were \sim 373 and 473 K higher than in the titanium substrate. Consequently, the flow stress-temperature relations for each substrate will have an important influence on the width of the transition region produced during dissimilar friction welding. In particular, when one substrate has a much higher flow istrength at high temperature than the other, the flux from one substrate will be substantially decreased and a very thin transition region will be formed. For example, Aritoshi *et al.* [12] compared the friction welding characteristics of oxygen free copper (OFC)-aluminium and Cu -70 wt % Waluminium joints. CuAl and $CuAl₂$ intermetallics were detected in the thick interdiffused region observed in the OFC copper-aluminium joints. In contrast, CuA1 and $CuAl₂$ intermetallics were only detected in an extremely thin transition region at the joint interface in the $Cu-70$ wt %W-aluminium joints. However, when the high temperature flow strength of the Cu-W composite material was decreased (by decreasing the tungsten content in copper to 30 wt %), the width of the interdiffused region produced during friction welding markedly increased. Aritoshi *et al.* [12] associated the higher tensile strength of $Cu-70$ wt %W-aluminium joints with the formation of a thin transition region at the joint interface.

It is worth noting that it might not be possible to form a transition region at the joint interface, when the thermophysical and high temperature mechanical properties of both substrates are widely different. This situation has been confirmed during stainless steel-copper friction welding [4].

5. Conclusions

The effect of low temperature post-weld heat treatment on the tensile strength and bend test properties of dissimilar friction welds between titanium and AISI 304L stainless steel was investigated. It has been confirmed that

1. Post-weld heat treatment at temperatures less than 873 K for 10 s had no effect on joint tensile strength properties. However, the joints tensile strength decreased when the PWHT temperature was 973 K or greater, and all test samples failed at the bondline region. In contrast, the bend test properties of dissimilar AISI 304L-titanium joints were markedly improved by low temperature post-weld heat treatment, and the highest bend angles were produced following post-weld heat treatment at 773 K for 1 h.

The Larson-Miller parameter corresponding to this treatment was 15.5×10^3 K h⁻¹. Low temperature heat treatment improved bend test properties because stress relaxation occurred with minima in the width of the transition region formed at the bondline region.

2. Joint bend test properties decreased markedly when the width of the transition region exceeded $1-2 \mu m$. An explanation for the detrimental effect of thick transition regions on the mechanical properties of dissimilar joints is proposed. It is suggested that the development of significant triaxial stress due to the constraint imposed by large, needleshaped intermetallic particles promotes premature joint failure in joints containing thick transition regions.

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